



The Chemours Company  
1007 Market Street  
PO Box 2047  
Wilmington, DE 19899

302-773-1000 t  
chemours.com

March 5, 2020

**2016-2017 Residential Drinking Water Well Surveying and PFAS Sampling Program Status Update, Chemours Chambers Works Complex, Deepwater, New Jersey (submitted September 2017) – Redacted/Removed Information**

Notice – The tables, figures and appendices listed below have been removed from the version of this report posted on this website because these tables, figures and appendices contain private, personal information such as names and/or addresses. To ensure residential confidentiality, the drinking-water well and treatment system results, if applicable, are now identified by a Regulatory Location ID, instead of the sample ID, which is the drinking-water well address or an abbreviated version of the address. All drinking-water wells and treatment system results, if applicable, can be found in Table 1 ([hyperlink](#)) and Figure 1 ([hyperlink](#)) listed by the Regulatory Location ID. Treatment system results may also be found in the quarterly Operation, Maintenance and Monitoring report ([hyperlink](#)).

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The Chemours Company  
1007 Market Street  
PO Box 2047  
Wilmington, DE 19899

302-773-1000 t  
chemours.com

September 8, 2017

Sameh Abdellatif  
Hazardous Waste Programs Branch  
Clean Air Sustainability Division  
U.S. Environmental Protection Agency, Region 2  
290 Broadway, 22nd Floor  
New York, NY 10007-1866  
Phone: (212) 637-4103

**2016-2017 Residential Drinking Water Well Surveying and PFAS Sampling Program  
Status Update, Chemours Chambers Works Complex  
Deepwater, New Jersey**

Dear Mr. Abdellatif:

Attached please find the 2016-2017 Residential Drinking Water Well Surveying and PFAS Sampling Program Status Update for the Chemours Chambers Works Complex in Deepwater, New Jersey. This report was prepared as per the agreement reached between the Chemours Company (Chemours), the New Jersey Department of Environmental Protection (NJDEP), and Environmental Protection Agency (EPA) Region 2 at the July 5, 2017 project status update meeting.

Chemours is submitting three hard copies of the report and one electronic version on a CD to both NJDEP and EPA.

If you have any questions, please email me at [Andrew.S.Hartten@chemours.com](mailto:Andrew.S.Hartten@chemours.com) or call me at 302-773-1289.

Sincerely,

A handwritten signature in black ink, appearing to read "Andrew S. Hartten", written in a cursive style.

Andrew S. Hartten  
Project Director, Chambers Works  
Chemours Corporate Remediation Group

cc: Helen Dudar, Case Manager, NJDEP (three hard copies and one CD)  
Chambers Works File

Chemours Chambers Works  
2016-2017 Residential Drinking Water Well Surveying and PFAS Sampling  
Program Status Update

CERTIFICATION I

"I certify under penalty of law that the information provided is true, accurate and complete. I am aware that there are significant civil penalties for knowingly submitting false, inaccurate or incomplete information and that I am committing a crime of the fourth degree if I make a written false statement which I do not believe to be true. I am also aware that if I knowingly direct or authorize the violation of any statute, I am personally liable for the penalties."



Andrew S. Harrten  
Project Director, Chambers Works  
Chemours Corporate Remediation Group

Sept. 8, 2017

WITNESSED THIS 8 DAY OF September, 2017



Notary Public

# **2016-2017 Residential Drinking Water Well Surveying and PFAS Sampling Program Status Update**

Chemours Chambers Works Complex  
Deepwater, New Jersey

Submitted on behalf of:  
The Chemours Company

Submitted by:  
AECOM  
Sabre Building  
Suite 300  
4051 Ogletown Road  
Newark, DE 19713

Project Number: 60533851  
Date: September 2017

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## Acronym List

Acronym	Explanation
µg/L	Microgram per Liter
APFO	Ammonium Perfluorooctanoate
CBI	Confidential Business Information
CSM	Conceptual Site Model
DuPont	E.I. du Pont de Nemours and Company
DVM	Data Validation Module
EPA	U.S. Environmental Protection Agency
GAC	Granular Activated Carbon
HA	Health Advisory
LCS	Laboratory Control Spike
LCSD	Laboratory Control Spike Duplicates
MS	Matrix Spike
MSD	Matrix Spike Duplicate
ND	Not Detected
NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid
ng/L	Nanograms per Liter
NJAW	New Jersey American Water
NJDEP	New Jersey Department of Environmental Protection
NJGWIIA	New Jersey Groundwater Class IIA Standards
NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid
OM&M	Operations Maintenance and Monitoring
PDF	Portable Document Format
PFAS	Poly- and Perfluoroalkyl Substances
PFCs	Perfluorinated Compounds
PFNA	Perfluorononanoic Acid
PFOA	Perfluorooctanoic Acid
PFOS	Perfluorooctane Sulfonate
PHA	Provisional Health Advisory
ppt	Parts per Trillion
PTEE	Polytetrafluoroethylene
PWS	Public Water Supply
QA/QC	Quality assurance/quality control
QAPP	Quality Assurance Project Plan
RPD	Relative Percent Differences
SOP	Standard Operating Procedure

## 1.0 Introduction

This report summarizes the status and findings of the ongoing 2016-2017 Residential Drinking Water Well Surveying and Per- and Polyfluoroalkyl Substances (PFAS) Sampling Program (2016-2017 program) conducted in the vicinity of the Chemours Company [Chemours; formerly E.I. du Pont de Nemours and Company (DuPont)] Chambers Works Complex (Chambers Works) located in Deepwater, New Jersey (see Figure 1). The purpose of this investigation is to evaluate the presence and distribution of 14 PFAS in residential drinking water wells in the vicinity of Chambers Works. This program is a follow-up program to the Residential Drinking Water Well Surveying and Sampling Program that was conducted by DuPont within a 2-mile radius of Chambers Works in 2009 (the 2009 program). The purpose of the 2009 investigation was to evaluate the presence and distribution of perfluorooctanoic acid (PFOA), one PFAS compound, in residential drinking water wells within two miles of Chambers Works. This report also includes a brief review of the 2009 program and findings. In addition, this report provides an update on the status of all drinking water wells that were qualified for treatment under either program, the type of treatment provided to reduce human exposure to PFAS from the drinking water, if accepted, and a summary of the ongoing quarterly granular activated carbon (GAC) treatment system Operations, Maintenance and Monitoring (OM&M) program.

### 1.1 Background

PFAS are a diverse group of compounds that are resistant to heat, water, and oil. PFAS have been used in both industrial applications and consumer products such as carpeting, upholstery, apparel, and fire-fighting foams. At Chambers Works, PFAS and precursors to PFAS (i.e., fluorotelomer alcohols) have been used in the production of fluoroelastomers, fluorotelomers, and have also been unintentionally created within manufacturing processes and waste streams. As described in the *Conceptual Site Model (CSM) for Poly- and Perfluoroalkyl Substances (PFAS) for the Chambers Works Complex* (PFAS CSM report; AECOM, 2017), 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 used during the manufacturing processes. The use of PFOA has been reduced at Chambers Works, and implementation of reduction programs have resulted in an overall 99% reduction in PFOA emissions compared to emissions from the year 2000. However, historical PFAS-containing air emissions may have migrated to off-site soils where PFAS may have leached into groundwater by way of precipitation. Groundwater is the drinking water source for many residents in the vicinity of Chambers Works.

In addition to the known on-site PFAS sources that may have migrated to off-site groundwater, other potential sources for PFAS in residential drinking water wells were identified in the Chambers Works PFAS CSM report. These include the use and disposal of consumer and industrial products that contain PFAS, such as stain-resistant treatments on upholstery and carpets, cleaning products, fire extinguishers, and many other common items that frequently are present in homes and businesses and which could also contribute to PFAS in residential drinking water wells. In addition, there may be potential contributions from nearby industries, airports, fire training area activities, and fire-fighting activities related to automobile accidents on the three major highways that cross the area.



## 1.2 Purpose and Objectives

The purpose of the 2009 and ongoing 2016-2017 programs is to evaluate the presence and distribution of PFAS in residential drinking water wells in the vicinity of Chambers Works.

The objectives of these investigations are as follows:

- Survey residences in the vicinity of Chambers Works and identify wells used as drinking water sources.
- Offer sampling to homeowners and renters who use these drinking water wells.
- Analyze the samples for PFOA (the 2009 program) or the 14 PFAS (the 2016-2017 program).
- Compare the PFAS results against applicable screening criteria.
- Offer treatment to owners of drinking water wells that exceed the applicable screening criteria.
- Install GAC treatment systems or connect the resident to the public water supply (PWS), if practical, or provide permanent bottled water, as appropriate.
- Provide GAC treatment system OM&M on a quarterly basis for GAC treatment systems installed under the programs.
- Evaluate the results to determine the following:
  - If trends are evident in the PFOA data where the data are comparable between the two programs
  - If evidence of the potential for PFAS sources unrelated to Chambers Works is observed
  - If expansion of the 2016-2017 program survey area is warranted

The objectives of this report are as follows:

- Summarize the 2009 program and provide information on the 2016-2017 program status.
- Present the evaluation of the PFAS results.
- Propose a path forward.

## 1.3 Report Structure

The remainder of this report is organized as follows:

- Section 2 provides a summary of the program conducted in 2009 and information on the implementation of the ongoing 2016-2017 program.
- Section 3 describes the sampling methodology, analytical methods, and data quality evaluation, including third-party data validation of the ongoing 2016-2017 program data.
- Section 4 provides the results of the 2009 program and the ongoing 2016-2017 program.

- Section 5 provides information on the drinking water wells qualified for treatment under both programs, the treatment provided, and an update on the ongoing quarterly GAC treatment system OM&M program.
- Section 6 provides the evaluation of the data and the observations made.
- Section 7 presents the proposed path forward.
- Section 8 provides the references cited in this report.

## **2.0 Residential Drinking Water Well Surveying and Sampling Program Implementation**

### **2.1 2009 Residential Drinking Water Well Surveying and Sampling Program**

In 2009, DuPont agreed to implement a voluntary program to sample residential drinking water wells within a 2-mile radius of Chambers Works (see Figure 2). The purpose of this program was to evaluate the distribution of PFOA in the off-site residential drinking water wells within the survey area. The surveying was conducted by developing a mailing list that included approximately 225 names and addresses, performing mass mailings, and establishing a call center to manage sampling requests. Residential drinking water well owners who called the call center were given the opportunity to have their drinking water wells sampled and analyzed for PFOA.

The program included sampling 110 residential drinking water wells (see Table 1; see Section 4 for a discussion of the results). Of the 110 drinking water wells sampled, only one drinking water well contained a PFOA concentration greater than U.S. Environmental Protection Agency's (EPA's) 2009 Provisional Health Advisory (PHA) level of 0.4 micrograms per liter ( $\mu\text{g/L}$ ), the screening criterion available at that time. A GAC treatment system was installed in June 2009 to reduce human exposure to PFOA from the drinking water, and quarterly OM&M has been and continues to be conducted to ensure the effectiveness of the GAC treatment system (see Section 5 for details of the ongoing quarterly OM&M program).

Reports documenting the progress of the 2009 program were submitted to the EPA Region 2 and the New Jersey Department of Environmental Protection (NJDEP) on approximately a monthly basis. The last report, which was submitted in July 2009, is provided here for reference as Appendix A<sup>1</sup>.

### **2.2 Ongoing 2016-2017 Residential Drinking Water Well Surveying and Sampling Program**

#### **2.2.1 2-Mile Radius Program**

In June, 2016, Chemours voluntarily began a follow-up survey and PFAS sampling program within the 2-mile radius of Chambers Works. For the 2016-2017 program, the same mailing list was used as in the 2009 program. Letters were mailed to residents requesting them to call a Chemours representative and answer a set of questions to determine if the well was qualified for sampling. Three letters were mailed to residents, approximately three weeks apart, giving the resident several opportunities to respond to the offer of sampling and analysis of 14 PFAS (see Table 2 for the complete analyte list and Appendix B for example documentation sent to residents) including PFOA, which was the PFAS analyzed for in 2009. Once a resident contacted the Chemours representative, their name was removed from the mailing list. During this timeframe, updated spreadsheets and maps were provided to EPA and NJDEP.

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<sup>1</sup> The Confidential Business Information (CBI) files included with that report are not included here. In addition, the laboratory reports are also not included here.

In addition, Chemours met with EPA and NJDEP on an approximately biweekly basis to provide status updates on the progress of the outreach program, including the sampling completed or scheduled, and to summarize the available final results. Any letters received back as “return to sender” due to a problem with the name and/or address were followed up by AECOM in an attempt to resolve the issue, and these situations were discussed with EPA and NJDEP during the biweekly conference calls. As the program progressed, these updates also included portable document formats (PDFs) of result letters mailed to residents, the status of the offers of treatment made by Chemours, and GAC treatment system installation or public water connection progress, again shared via updated spreadsheets and maps. Result letters to residents were also provided to appropriate County Departments of Health and Municipal Clerks. In addition, in November 2016, EPA completed additional outreach to residents who were still non-responsive to Chemours’ outreach efforts (see Appendix B).

By the end of the second quarter of 2017, the sampling of drinking water wells for those residents that accepted the offer was completed. As of July 31, 2017 and as shown in Appendix C:

- 195 residents were contacted via mailings or contacted AECOM on their own.
- 52 residents who were contacted via mailings are non-responsive to the offer to sample (see green rows in Appendix C).
- 55 residents contacted via mailings had names and/or addresses that were still undeliverable for various reasons after AECOM follow-up (see pink rows in Appendix C).
- 90 drinking water wells were sampled<sup>2</sup> (see blue rows in Appendix C and note that one resident has three wells).

PFAS results for the 90 drinking water wells sampled are presented and discussed in Section 4. Based on agreement between Chemours, EPA and NJDEP, results for the three PFAS for which there are applicable screening criteria, Perfluorononanoic Acid (PFNA), PFOA, and Perfluorooctane Sulfonate (PFOS), were compared to the applicable criteria shown below:

- The New Jersey Groundwater Class IIA (NJGWIIA) groundwater quality criterion for PFNA of 0.01 µg/L
- EPA’s 2016 Lifetime Health Advisory (HA) for PFOA and PFOS of 0.07 µg/L. If both PFOA and PFOS are detected in a drinking water well, the individual PFOA and PFOS concentrations and the sum of PFOA and PFOS concentrations were compared to the HA of 0.07 µg/L
- The NJDEP preliminary health-based guidance for PFOA in drinking water value of 0.04 µg/L (comparison to this criterion was based on a separate agreement between Chemours and NJDEP)

Based on the screening of PFNA, PFOA and PFOS, 43 drinking water wells identified during the 2-mile radius surveying and sampling exceeded the applicable screening criteria and were qualified for treatment to reduce exposure to PFAS from the drinking water. Section 5 provides further discussion of qualified drinking water wells, the status of treatment offered for each well, and the ongoing quarterly OM&M program for GAC treatment system installations. Based on the results within the 2-mile radius survey area

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<sup>2</sup> Copies of the result letters are provided under separate cover as these letters include CBI.

that showed exceedances of criteria near the 2-mile radius boundary in select areas, the survey area was expanded to 2.5-mile radius in a northeast to southeast direction, as described below.

## 2.2.2 Expanded Areas Programs

Figure 3 shows the expanded surveying and sampling area in the northeast through southeast direction to a 2.5 mile radius (identified as Expanded Area 1 in the figure). Starting in February 2017, cold calls by a team of two Chemours representatives were used to visit residences and drop off a letter offering sampling of drinking water wells and the EPA outreach document developed in November 2016 (see Appendix B for example documentation). However, on February 20, 2017, as the Chemours representatives were finishing this effort, a resident was encountered who was threatening to the Chemours representatives and the cold calls were stopped immediately. At the same time, the expanded area results available showed exceedances of screening criteria near the 2.5-mile radius boundary in select locations; therefore, the survey area was again expanded to the Layton's Lake community and to approximately a 2.75 mile distance from the site along Pennsville Pedricktown Rd., as shown in Figure 3 and identified as Expanded Area 2.

On May 3, 2017, the first of three mailings, approximately three weeks apart, were sent to all residences in the 2- to 2.5-mile radius expanded area who were non-responsive to the offer of sampling or who had not had a cold call contact (see Appendix B for example documentation). Letters were also sent to the residences in Layton's Lake expanded area and to the residences in the further expansion along Pennsville Pedricktown Rd. Appendix D<sup>3</sup> provides information on the residences within the expanded areas. Residences were identified in the expanded areas based on field reconnaissance and tax record searches. Again, letters received as "return to sender" were followed up by AECOM and discussed with EPA and NJDEP at the biweekly conference calls.

As of August 29, 2017 within the expanded areas (outside of the 2-mile radius):

- 101 residents were contacted.
- 64 drinking water wells were sampled and results are final for 62 wells<sup>4</sup> (see blue rows in Appendix D).
- 34 residents within the expanded area have not responded to Chemours' offer of drinking water well sampling (see green rows in Appendix D).
- Three letters were "return to sender," and AECOM has not been able to resolve the address issues (see pink rows in Appendix D).

PFAS results for the drinking water wells sampled are discussed in Section 4. Results for PFNA, PFOA, and PFOS were compared to the applicable criteria listed in Section 2.2.1. Of the 62 wells results screened against the criteria, 21 are qualified for treatment to reduce exposure to PFAS from the drinking water. Section 5 provides further discussion of qualified drinking water wells, the status of treatment offered for each drinking water well, and the ongoing quarterly OM&M program.

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<sup>3</sup> CBI has been removed from this appendix. This appendix with CBI is provided under separate cover.

<sup>4</sup> Copies of the result letters are provided under separate cover as these letters contain CBI.

### 3.0 Sampling Methodology, Analytical Methods, and Data Quality

A draft Quality Assurance Project Plan (QAPP) for the Chemours 2016-2017 program, was submitted to EPA and NJDEP on April 19, 2016. Comments on the draft QAPP were received by Chemours on April 29, 2016 and were addressed in the QAPP dated June 2, 2016. On June 3, 2016, the final QAPP titled *Quality Assurance Project Plan For the PFAS Drinking Water Sampling Program, Chemours Chambers Works, Deepwater, New Jersey* was submitted to Mr. Sin Kie Tjho, the EPA Region 2 Project Manager at that time. The QAPP was also copied to Ms. Linda Range, the NJDEP case manager at that time. The QAPP was approved by Mr. Tjho on June 9, 2016. The QAPP is provided here as Appendix E for reference.

The QAPP was prepared using the Uniform Federal Policy for Quality Assurance Project Plans as requested by EPA and was 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.

#### 3.1 Sampling Methodology

Drinking water was sampled from taps at residential locations according to the procedure found in QAPP Worksheet #21, taking care to avoid possible sources of PFAS contamination.

Information related to collection of each drinking water sample was recorded on a data collection sheet (see Appendix E of the QAPP) in a sampling book with carbon copy sheets.

Quality control samples were collected as described in QAPP Worksheet #20, including collection of field duplicate samples at better than a frequency of 1 in 20. Extra sample volume from designated sample locations was planned at a frequency of 1 in 20 to allow analysis of laboratory QC samples as matrix spikes (MS) and laboratory replicates, as an assessment of precision and accuracy. Where extra volume was not submitted to the laboratory for a given lot of samples, precision and accuracy were measured by laboratory analysis of laboratory control spikes (LCS) and LCS duplicates (LCSD).

Field blank samples were collected by performing a bottle-to-bottle transfer of laboratory blank water during each day of sampling.

#### 3.2 Analytical Methods

Drinking water samples were submitted to TestAmerica Sacramento, West Sacramento, California for PFAS analysis using liquid chromatography tandem mass spectrometry. The laboratory Standard Operating Procedure (SOP) used for analysis can be found in Appendix A of the QAPP.

The target list reported can be found in Table 2 of this report.

The target list was reported to a reporting limit of 2 nanograms per liter (ng/L) in water except that N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA) and N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA) were reported to 20 ng/L.

Laboratory reports provided by TestAmerica can be found in Appendix F.

### 3.3 Data Quality

Data for the 2016 and 2017 residential samples have been 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 (EIM DVM) and manual reviewer evaluations. The data are evaluated against the following data usability checks:

- Field and laboratory blank contamination
- EPA hold time criteria
- Missing QC samples
- MS/Matrix Spike Duplicate (MSD) recoveries and the relative percent differences (RPDs) between these spikes
- LCS/ LCSD recoveries and the RPDs 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.

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.
U	Not Detected.

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

Overall, the analytical data are acceptable for use without qualification; however, some analytical results have been qualified in the database. The individual DVM narrative report for each project (2016 and 2017), found in Appendix G, lists which samples were qualified, the specific reasons for the qualification, and the potential bias in reported results.

### 3.4 Third-Party Data Validation

Approximately ten percent of the data points generated during the 2016 and 2017 residential sampling in three laboratory lots are currently being validated by a third-party reviewer, Environmental Standards, Inc., Valley Forge, Pennsylvania for compliance with the laboratory SOP and data usability. Validation is taking place concurrent with data reporting in order to expedite reporting of results. The National Functional Guidelines will be used as a guide for report formatting and application of qualifiers. 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 (see Appendix J of the QAPP).

The validation reports will be shared with EPA when available and upon request.



## 4.0 Drinking Water Well Results

PFOA results from 2009 and PFOA, PFOS and PFNA results from 2016-2017 are provided in Figure 4<sup>5</sup>. Data shown in Figure 4 are organized by the number on the letters mailed to residents where appropriate. The figure also includes a table that links the letter number and the street address of the drinking water well. Samples collected for which there was no letter mailed (some residents obtained the Chemours representatives phone number from other residents who did receive letters) are organized by street address. In the table within Figure 4, these locations are listed at the top of the table as “no letter number.” Table 4 provides the PFOA, PFOS and PFNA results that are posted in Figure 4 that are organized by the location identification, which is a modified version of the street address. Appendix H provides all PFAS data for the 2009 and 2016-2017 programs, including GAC treatment system data.

The 2009 PFOA results shown in Figure 4 were screened against EPA 2009 PHA of 0.4 µg/L. The 2016-2017 PFNA, PFOA, and PFOS results were screened against the criteria listed in Section 2.2.1. In Figure 4, circles are used to identify locations sampled in 2009 with the follow color coding:

- Pink circles – PFOA is greater than or equal to 0.4 µg/L.
- Yellow circles – PFOA is quantifiable and less than 0.4 µg/L.
- White circles – PFOA is non-detect or not quantifiable at the concentration posted.

Locations sampled during 2016-2017 are indicated by squares or triangles with the following color coding:

- Pink squares – PFNA is greater than 0.01 µg/L and/or PFOA, PFOS, or PFOA plus PFOS is greater than or equal to 0.07 µg/L.
- Pink triangles – PFOA is greater than 0.04 µg/L but less than 0.07 µg/L.
- Yellow squares – PFNA, PFOA, and PFOS are less than the screening criteria listed in Section 2.2.1.
- White squares – PFNA, PFOA, and PFOS are less than the reporting limit of 0.002 µg/L.

All locations in pink indicate drinking water wells that exceed screening criteria and are qualified for an offer of treatment by Chemours. Section 5 provides information on the process by which owners of qualified drinking water wells were offered treatment to reduce exposure to PFAS from the drinking water, the treatment status of each qualified drinking water well, and the quarterly OM&M program.

Figure 4 shows that there are pink locations throughout and near the boundary of the surveying and sampling area. Section 6 presents the evaluation of the data shown in Figure 4, and Section 7 provides the proposed path forward.

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<sup>5</sup> Note that the locations of residents that have not responded to the offer of sampling have been removed in Figure 4. These locations were shown in draft versions of the 2-mile radius map and the expanded area map figures that were previously provided to EPA and NJDEP.

## 5.0 Drinking Water Wells Qualified for Treatment

Based on the data collected during the 2009 program, one residential drinking water well qualified for treatment because the PFOA concentration measured was greater than the EPA's 2009 Provisional Health Advisory of 0.4 µg/L. A GAC treatment system was installed for that drinking water well in June 2009, and a quarterly OM&M program was developed and implemented for that treatment system.

Based on the data collected for the 2016-2017 program as of August 28, 2017, 65 drinking water wells are qualified for treatment because these wells have concentrations of PFNA, PFOA and/or PFOS above the screening criteria identified in Section 2.2.1. Once a drinking well was identified as qualified for treatment, the well owner was contacted, and an offer of bottled water was made. Bottled water was provided to renters of properties with permission from the well owner in situations where the property was a rental property. Bottled water delivery continued until completion of the GAC treatment system installation or the PWS connection. Which treatment option that was offered depended upon the location of the residence with respect to PWS water lines. PWS connection was offered wherever possible. However, in some cases, residents did not want connection to the PWS, so installation of a GAC treatment system was offered. All costs associated with treatment (bottled water, GAC installation or PWS connection) were paid for by Chemours. Owners accepting PWS connection were responsible for all costs subsequent to the PWS connection being completed. Not all residents accepted the offer of bottled water and a few residents declined the offer of treatment all together (see Section 5.4 below). In addition, there was one resident who accepted bottled water as an interim measure until such time as the new well he will be drilling is installed (see Section 5.3 for additional details).

Table 5 provides a list of the qualified wells and the treatment status. Figure 5 shows the location of the 66 drinking water wells identified during the 2009 and the 2016-2017 programs qualified for treatment and provides the following:

- The locations for which GAC treatment systems were offered and installed, or installation is underway
- The locations for which PWS connections were offered and connections were completed, or are underway
- The location where bottled water is being delivered as a temporary interim measure
- The locations where offers of treatment were declined

Additional information on each of these treatment options are provided below<sup>6</sup>.

### 5.1 GAC Treatment System Installation and the Operations, Maintenance and Monitoring Program

Of the 66 drinking water wells qualified for treatment, 56 well owners were offered installation of a residential GAC treatment system (see Figure 5). Iron issues were encountered at several drinking water wells subsequent to the GAC treatment system installation, and bottle delivery resumed at these locations until the iron issues were

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<sup>6</sup> Documentation for GAC installations, PWS connections, declines, and bottled water are provided under separate cover as this information contains CBI.

resolved, at which time bottled water delivery ended. Following installation of a GAC treatment system, the drinking water well was incorporated in the OM&M program in the quarter following the installation.

Prior to the third quarter of 2016, there was only one GAC treatment system installed, which was installed under the 2009 program. Between June 2009 and the third quarter of 2016, samples were collected from after the first carbon bed (BED1) and after the second carbon bed (BED2) on a quarterly basis, although for a time period, only BED2 (which is representative of the treated water being consumed by the residents) was sampled. A prior to treatment sample was also collected in the third quarter of each year. Samples collected were analyzed for PFOA. A carbon change out was performed in July 2015 because the system had been in operation for five years at that point.

Only two GAC systems required sampling in the fourth quarter of 2016, the system installed in 2009 and the one system installed in the third quarter of 2016. BED1 and BED2 samples were collected and were analyzed for PFNA, PFOA, and PFOS. The GAC system OM&M program currently underway was fully developed during the fourth quarter of 2016 and was implemented during the first quarter of 2017.

Under the current OM&M program, quarterly monitoring consists of two samples: BED1 and BED2. BED1 is collected from the sample port after the first carbon bed. BED2 is collected from a sampling port after the second carbon bed and is representative of the treated water used in the residence. BED1-D, a field duplicate sample may be collected from the GAC system at the same time as the BED1 sample for quality control. The BED2 sample will only be analyzed if the PFOA, PFOS or PFNA results for the BED1 sample are greater than or equal to 0.0020 µg/L. When the PFOA or PFOS, or PFOA plus PFOS concentration in the BED2 sample reaches 0.015 µg/L, or the PFNA concentration in BED2 is greater than or equal to 0.002 µg/L, the well owner will be contacted and arrangements will be made to replace the carbon in BED1 of the system. Carbon beds will be replaced every five years if the change out criteria are not reached within five years. In the third quarter of each year, a sample will also be collected from a sampling port prior to the treatment system, which will be sampled for PFOA, PFOS and PFNA.

First quarter 2017 OM&M program included sampling of 28 GAC systems. The *Chemours Chambers Works Survey and Sampling Program Residential GAC System Results – 1Q17* was submitted to EPA and NJDEP on May 8, 2017. The second quarter 2017 OM&M program included sampling of 36 GAC systems. One GAC system that should have been sampled during 1Q17 and 2Q17 was not sampled as iron issues were still being resolved and the system was in bypass mode during that timeframe (the resident received bottled water during this time). The report for the second quarter 2017 sampling was submitted to EPA and NJDEP on August 25, 2017.

Between issuing the first quarter report and the second quarter report, the decision was made that moving forward, Table 1 in these OM&M reports will be submitted with Confidential Business Information (CBI; names, addresses, phone numbers, etc.) removed and a version of the table with the CBI included will be submitted under separate cover to EPA and NJDEP. In addition, the PDF of the result letters mailed to GAC system owners will no longer be included in the report but will also be submitted under separate cover. Therefore, a revised first quarter 2017 report will also be submitted as soon as possible with CBI under separate cover. The second quarter 2017 OM&M report was issued with CBI provided under separate cover.

## 5.2 Public Water Supply Connection

Of the 66 drinking water wells qualified for treatment, seven were located in the vicinity of a PWS water line [in this case New Jersey American Water (NJAW)] and connection to the PWS was offered. Locations of PWS connections are provided in Figure 5. One resident who was offered connection to NJAW made arrangements for have the residence connected to NJAW because he was in the process of selling the residence and Chemours has reimbursed the resident for his costs for connection.

## 5.3 Bottled Water

One resident with a qualified drinking water well who was offered GAC treatment informed the Chemours representative that within the next year, he was planning on demolishing the residence and then rebuilding a new residence, including installing a new drinking water well. Therefore, the resident accepted an offer of bottled water until such time as the new well is installed and sampled. The new well will be sampled for the 14 PFAS listed in Table 2. If the well is qualified for treatment based on the screening criteria for PFNA, PFOA and or PFOS, a GAC treatment system will be installed.

## 5.4 Treatment Offer Declines

Two residents declined the offers of treatment from Chemours by signing the decline paperwork provided with the results and offer letter sent when the data were final. The locations of these residences where owners declined treatment are shown in Figure 5.

## 6.0 Data Evaluation

Figure 4 shows exceedances of the applicable screening criteria throughout and near the boundary of the surveying and sampling area. The data shown in Figure 4 were evaluated to determine if qualitative trends could be observed in the PFOA concentrations between the 2009 and the 2016-2017 results, to determine if the concentrations of PFOA, PFNA, and PFOS observed indicate the possible presence of PFAS sources unrelated to Chambers Works, and to determine if expansion of the survey and sampling area is warranted.

### 6.1 Comparison of 2009 and 2016-2017 PFOA Results

Table 6 provides PFOA results for locations sampled in 2009 and in 2016-2017. PFOA data from 2009 and 2016-2017 were compared relative to the order of magnitude (increase, decrease, same) between the data sets; however, different methods and laboratory SOPs were used to analyze the samples<sup>7</sup>.

The following observations were made:

- 68 drinking water wells have data from 2009 and 2016-2017 which can be compared.
- Seven drinking water well results from 2016-2017 show one order of magnitude increase compared to the 2009 results.
- Four drinking water well results from 2016-2017 one order of magnitude decrease compared to the 2009 results.
- Wells with PFOA concentrations identified as increasing or decreasing are located throughout the surveying and sampling area and are not clustered together.
- 57 of the 68 drinking water wells' results from 2016-2017 are the same order of magnitude when compared to the 2009 results.

These data show that in general, the concentrations appear to be similar between 2009 and 2016-2017. However, there are locations where increases or decreases are observed.

### 6.2 Dot Map Evaluation

Dot maps showing PFOA, PFNA, and PFOS results were prepared as a means of evaluating the data (see Figures 6, 7, and 8, respectively). PFOA and PFNA are Chambers Works-related PFAS, whereas PFOS is not a Chambers Works-related PFAS. As indicated in the PFAS CSM report for Chambers Works in Figure 21 and Table 14 (provided here as Figure 9 and Table 7), there are many potential PFAS sources that may be found in the vicinity of Chambers Works, in addition to the PFAS

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<sup>7</sup> In 2009, PFOA was analyzed using MPI Research SOP 01M-008-Q46, Method of Analysis for the Determination of Ammonium Perfluorooctanoate (APFO) in Water. This SOP describes an LCMSMS method using external standard quantitation. In 2016-2017, the EPA 537 target list was analyzed using TestAmerica Sacramento's SOP WS-LC-0025 Rev. 1.9, Perfluorinated Compounds (PFCs) in Water, Soils, Sediments and Tissue [Method 537 Modified]. The TestAmerica Sacramento SOP describes an LCMSMS method using isotope dilution quantitation.

that is site-related. Some examples of PFAS associated products and materials that may be used or disposed of off-site by either residential property owners or by outside businesses include the following (Guo, 2009):

- Pre-treated carpeting
- Carpet care liquid treated carpeting
- Treated apparel
- Treated upholstery
- Treated home textiles
- Treated non-woven medical garments
- Industrial floor wax and wax removers
- Stone, tile, and wood sealants
- Membranes for apparel
- Food contact paper
- Dental floss/tape
- Thread sealant tape
- Polytetrafluoroethylene (PTFE) cookware

The off-site use or disposal of PFAS associated products/materials, or the washing of PFAS associated products/materials and subsequent disposal of gray water (either via discharge to ground surface or via septic system), could create a PFAS source that could migrate to off-site environmental media. A study of domestic drinking water wells (Schaidler, 2016; Silentspring.org article) found that residential septic systems were the main source of contaminants to groundwater; contaminants detected in this study included PFOS and PFOA. Although specific information on local off-site sources and migration pathways are not known in detail for the area surrounding the Chambers Works site, it is important to acknowledge that these likely do exist and may be contributing to the concentrations of PFAS measured in off-site environmental media.

For the PFOA, PFNA and PFOS dot maps, the concentration ranges are in parts per trillion (ppt) and were selected to reflect the ranges of data observed and are different in each dot map. Evaluation of the three dot maps is complicated because very limited information are available on well construction and depths, the data are clustered in neighborhoods and along roads, and there are large areas for which there are no houses and therefore, no data. However, several qualitative observations can be made in evaluating and comparing the dot maps:

- All three dot maps show areas where higher concentrations and non-detects are adjacent to each other as seen in Cedar Crest Manor, just south of the intersection between the New Jersey Turnpike and Rt. 40. At this cluster of houses, the houses with the highest concentration of PFOA also have the highest concentrations of PFNA and PFOS.
- All three dot maps also show locations where the higher concentrations of PFOA, PFNA and PFOS are not co-located. For example, the cluster of houses at the southern end of South Golfwood Ave. shows that the house with the highest PFOA concentration in that cluster is not the same house as the one with the

highest PFOS concentration, and those houses do not have the highest PFNA concentration in the cluster.

- Three of the wells in the data set have results that are very different from the rest of the data sets and suggest another PFAS source:
  - The well with the PFNA concentration of 140 ppt [all other PFNA data range from not detected (ND) to 36 ppt],
  - The two wells with PFOS concentrations of 1,300 ppt and 320 ppt (all other PFOS data range from ND to 55 ppt).
- In the PFOA dot map, the concentrations of PFOA appear to be to be relatively consistent across the area, with the exception of a cluster of higher PFOA results in Cedar Crest Manor.
- For the PFNA dot map:
  - There are clusters of higher PFNA in the vicinity of the New Jersey Turnpike and Rt. 40 in Cedar Crest Manor, on Sportsmans Rd., and near Layton's Lake.
  - There also appears to be an area of wells with no detections of PFNA near the 2-mile radius boundary.
- The PFOS dot map also appears to shows relatively consistent concentrations throughout the area and also shows a cluster of higher results within Cedar Crest Manor as is observed in the PFOA and PFNA dot maps and may suggest another PFAS source.
- The PFOS dot map also appears to show isolated higher concentrations sporadically across the area. However, within the Layton's Lake development just north of the New Jersey Turnpike, south of Layton's Lake, there is a cluster of houses where the PFOA and PFOS concentrations are both higher; however, this area contains the majority of the highest PFOS concentrations measured. These higher PFOS concentrations suggest another PFAS source.

To further evaluate the data and identify wells which suggest PFAS sources other than Chemours, a figure displaying a pie chart for PFOA, PFOS, and PFNA at each sample location was developed (see Figure 10). In this figure, the size of the pie chart represents the total concentration of PFOA plus PFOS plus PFNA (which is also shown in black in each chart) and the three colors show the percent of each in the sample.

The pie charts shown in Figure 10 emphasize some of the observations made from the dot maps, including the following:

- The distinctly different pattern of PFOS concentrations approximately equal to or greater than PFOA in the area between Layton's Lake and the New Jersey Turnpike compared to the other pie charts.
- The two locations with the highest concentrations of PFNA (140 ppt) and PFOS (1,300 ppt) appear to be outliers in the data set.

In addition, the pie charts emphasize that PFOA, in general, is present in higher concentrations than PFNA or PFOS and that even as the total amount of these three PFAS increases, the relative proportions appear to remain relatively constant.

The observations made on the dot maps and the pie chart figure suggest the existence of another source or sources of PFAS to the drinking water in addition to PFAS from the site.

### **6.3 Summary of Observations**

The post map provided in Figure 4 shows that there are drinking water wells within and at the boundary of the survey area that have concentrations of PFOA, PFNA and PFOS that exceed the applicable criteria. However, PFOS in drinking water is unrelated to the site. The distribution of PFOS across the survey area and the variability in the concentrations measured support that there may be a source or sources of PFOS (and perhaps other PFAS) that are impacting drinking water in addition to the PFAS migrating from the site in historical air emissions.



## 7.0 Proposed Path Forward

Based on the data evaluation observations presented in Section 6, the following path forward will be discussed during September 19, 2017 meeting between Chemours, EPA, and NJDEP:

- Additional expansion of the surveying and sampling area in increments to be determined
- Establishment of representative background concentrations of PFOA, PFNA, and PFOS
- Additional investigation into potential PFAS sources unrelated to the site in the vicinity of the site

## 8.0 References

- AECOM, 2017. *Conceptual Site Model (CSM) for Poly- and Perfluoroalkyl Substances (PFAS) for the Chambers Works Complex, Deepwater, New Jersey.*
- EPA. 2009. *EPA Guidance for Labelling Externally Validated Laboratory Analytical Data for Superfund Use (EPA-540-R-08-005).*
- Guo, Liu, Krebs, Roache. 2009. *Perfluorocarboxylic Acid Content in 116 Articles of Commerce. National Risk Management Research Laboratory, Office of Research and Development. U.S. Environmental Protection Agency. EPA/600/R-09/033. March 2009*
- Schaider et al., 2016. *Septic systems as sources of organic wastewater compounds in domestic drinking water wells in a shallow sand and gravel aquifer. Science of the Total Environment 547 (2016) 470-481*
- SilentSpring.org article. *Tests find new contaminants in Cape Cod's drinking water supply; septic systems are likely the main source of the pollution.*  
<http://www.silent.spring.org/research-update/tests-find-new-contaminants-cape-cod%E2%80%99s-drinking-water-supply-septic-systems-are>

## Tables

**Table 2**  
**PFAS Analyte List - 2016-2017 Residential Drinking Water Well Samples**  
**2016-2017 Residential Drinking Water Well Surveying and PFAS Sampling Program**  
**Chemours Chambers Works Complex**  
**Deepwater, New Jersey**

Analyte Name	Abbreviation	CAS No.
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	PFDoA	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	PFTA	376-06-7
Perfluorotridecanoic acid	PFTTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnA	2058-94-8
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	—
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	—


**Table 7**  
**Off-Site PFAS Pathways**  
**2016-2017 Residential Drinking Water Surveying and PFAS Sampling Program**  
**Chemours Chambers Works Complex**  
**Deepwater, New Jersey**

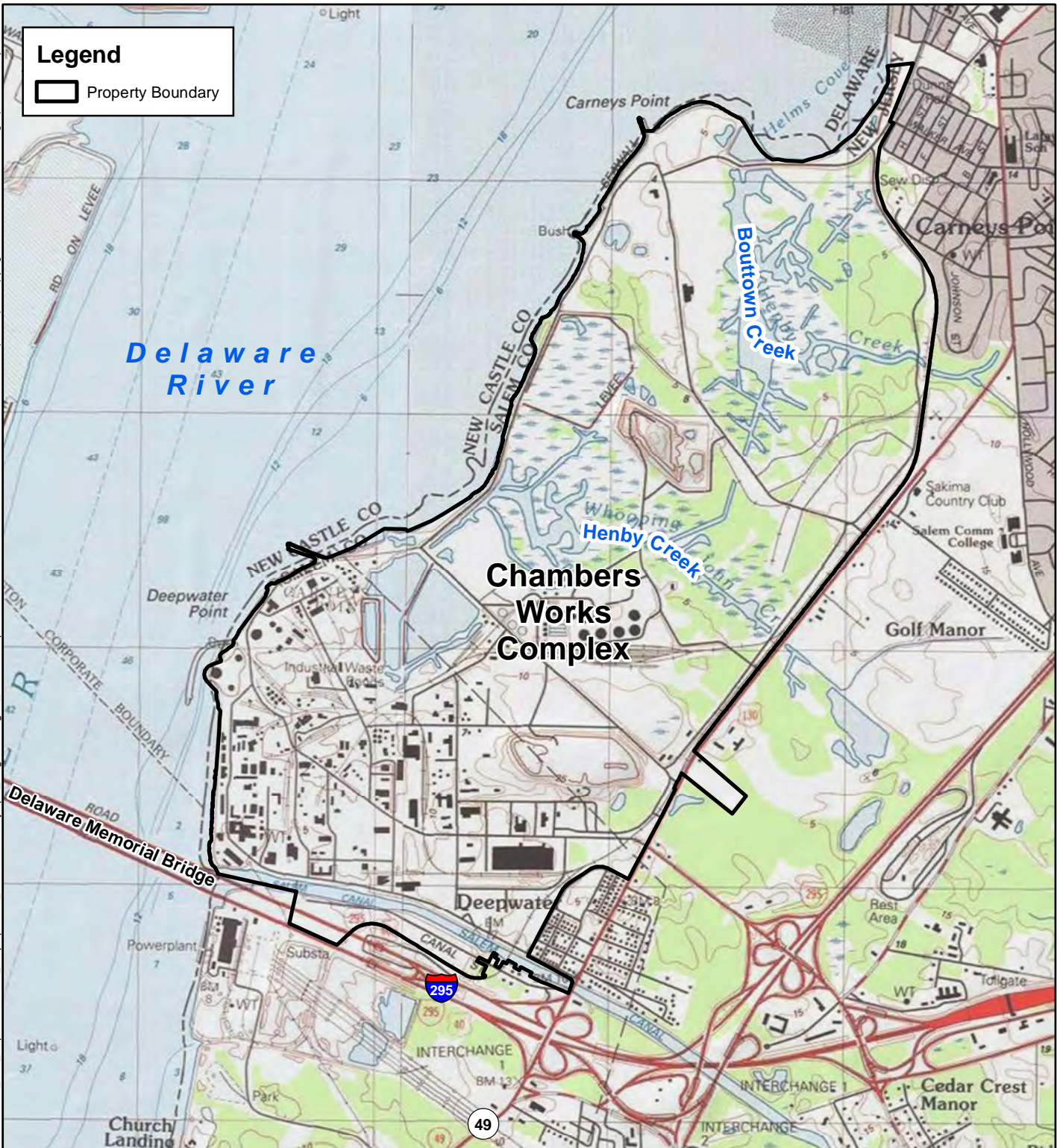
<b>Index</b>	<b>Impacted Media Acting as a Source</b>	<b>Release Mechanism/Pathway</b>	<b>Receptor Media Potentially Impacted</b>
1	Landfill Leachate, Airport Tarmacs, Paved Surfaces, Fields	Air Emissions	Air, Soil, Surface Water, Sediment, and Groundwater
2	PFAS-Containing Groundwater	Discharge	Surface Water and Sediment
3	PFAS-Containing Surface Water	Infiltrate and Recharge	Groundwater
4	PFAS-Containing Groundwater	Well Pumping	Potable, Industrial, or Irrigation Water
5	PFAS-Containing Gray Water	Discharge to Septic Systems (Tanks, Leachate Fields)	Groundwater

Impacted Media Acting as a Source - Source of initial media impact is not defined but could include the following: Fire Stations and Fire Training Areas, Landfills, Treatment Plants, and Industry and Residential properties that have historically used, stored, or disposed of PFAS containing products.

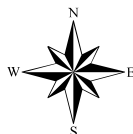
## Figures

**Legend**

 Property Boundary



AECOM  
 Sabre Building, Suite 300  
 4051 Ogletown Road  
 Newark, DE 19713



0 1,000 2,000

Feet

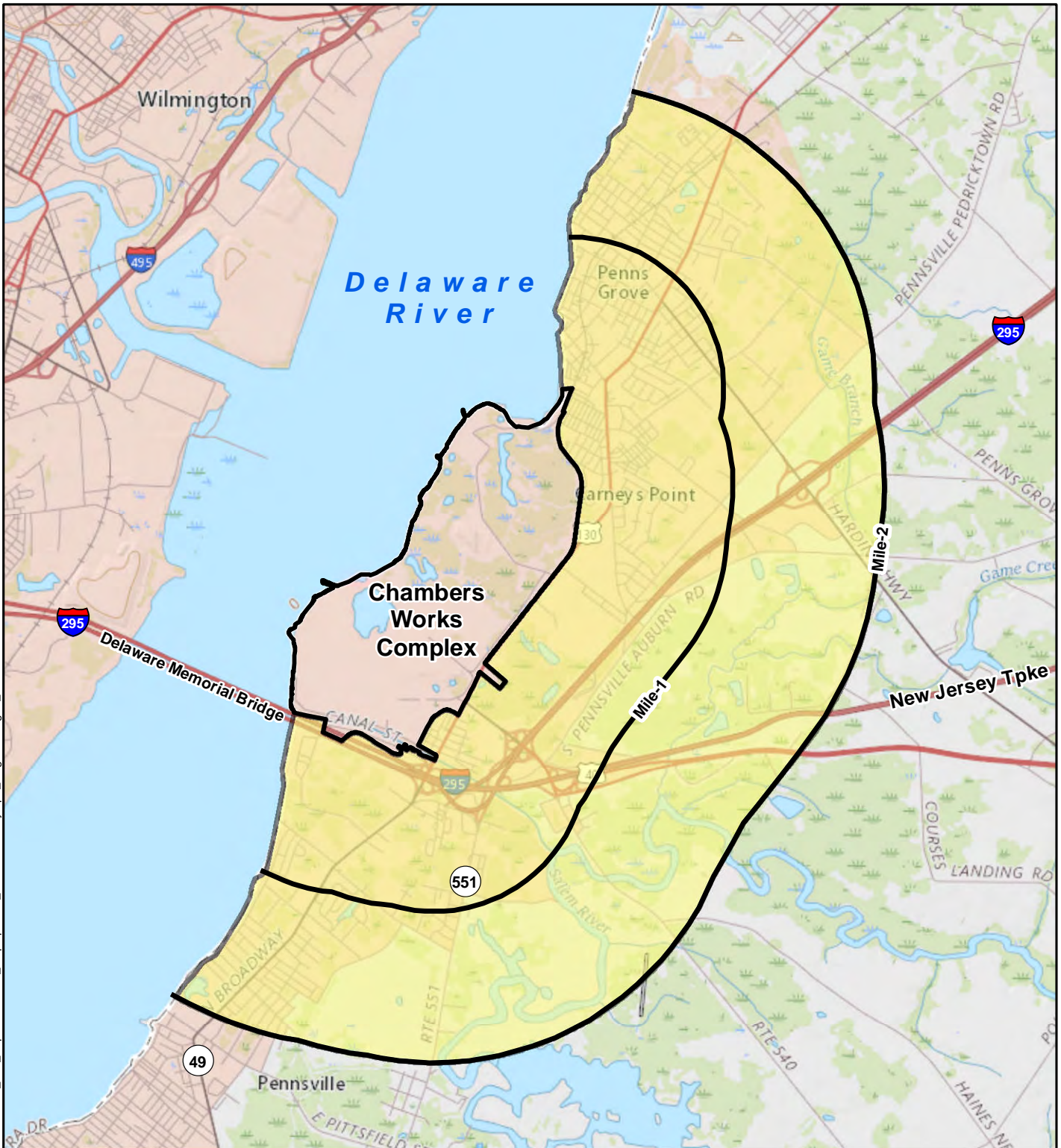
1 inch = 2,000 feet

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### SITE LOCATION MAP

2016-2017 RESIDENTIAL DRINKING WATER  
 WELL SURVEYING AND SAMPLING PROGRAM  
 CHEMOURS CHAMBERS WORKS COMPLEX  
 DEEPWATER, NEW JERSEY

TASK NUMBER:	17006	PROJECT NUMBER:	60533851
DESIGNED BY:	K. L. DAVIS	DATE:	9/7/2017
DRAWN BY:	C. DUFFY	FIGURE NUMBER:	1
DATA QUALITY CHECK BY:	S. MURPHY		



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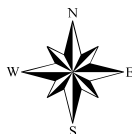
**Legend**

- Property Boundary
- Approximate 2-Mile Radius Area

USGS TNM Topo Base Map Credits:  
 USGS The National Map - National Boundaries Dataset, 3DEP Elevation Program, Geographic Names Information System, National Hydrography Dataset, National Land Cover Database, National Structures Dataset, and National Transportation Dataset; U.S. Census Bureau - TIGER/Line and USFS Road Data



AECOM  
 Sabre Building, Suite 300  
 4051 Ogletown Road  
 Newark, DE 19713



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Feet

1 inch = 5,000 feet

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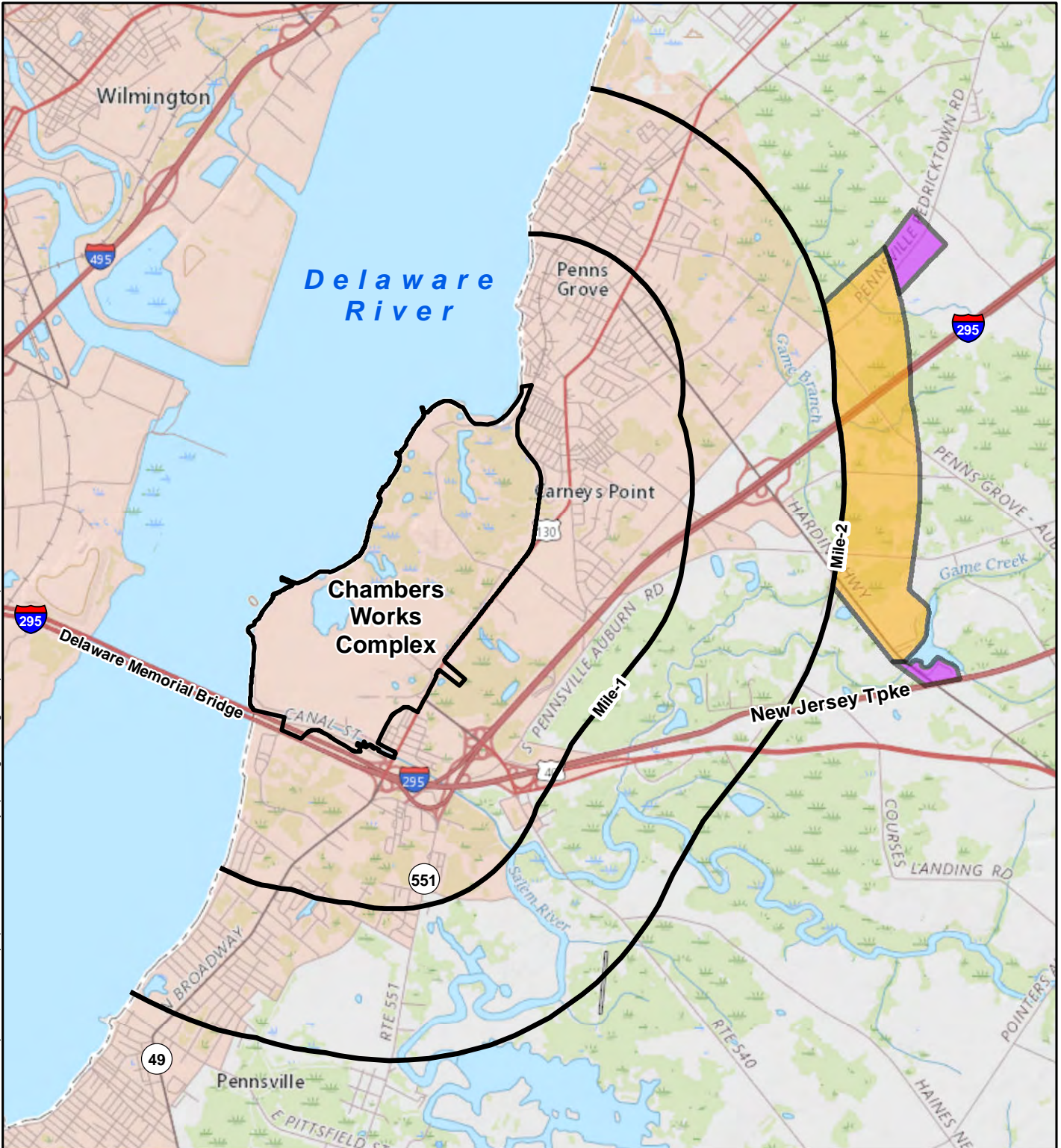
**2-MILE RADIUS MAP**

2016-2017 RESIDENTIAL DRINKING WATER  
 WELL SURVEYING AND SAMPLING PROGRAM  
 CHEMOURS CHAMBERS WORKS COMPLEX  
 DEEPWATER, NEW JERSEY

TASK NUMBER:	17006	PROJECT NUMBER:	60533851
DESIGNED BY:	K. L. DAVIS	DATE:	9/7/2017
DRAWN BY:	C. DUFFY	FIGURE NUMBER:	2
DATA QUALITY CHECK BY:	S. MURPHY		



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**Legend**

- Property Boundary
- Expanded Area 1 (Approximate)
- Expanded Area 2 (Approximate)

USGS TNM Topo Base Map Credits:  
 USGS The National Map - National Boundaries Dataset, 3DEP Elevation Program, Geographic Names Information System, National Hydrography Dataset, National Land Cover Database, National Structures Dataset, and National Transportation Dataset; U.S. Census Bureau - TIGER/Line and USFS Road Data



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 4051 Ogletown Road  
 Newark, DE 19713



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Feet

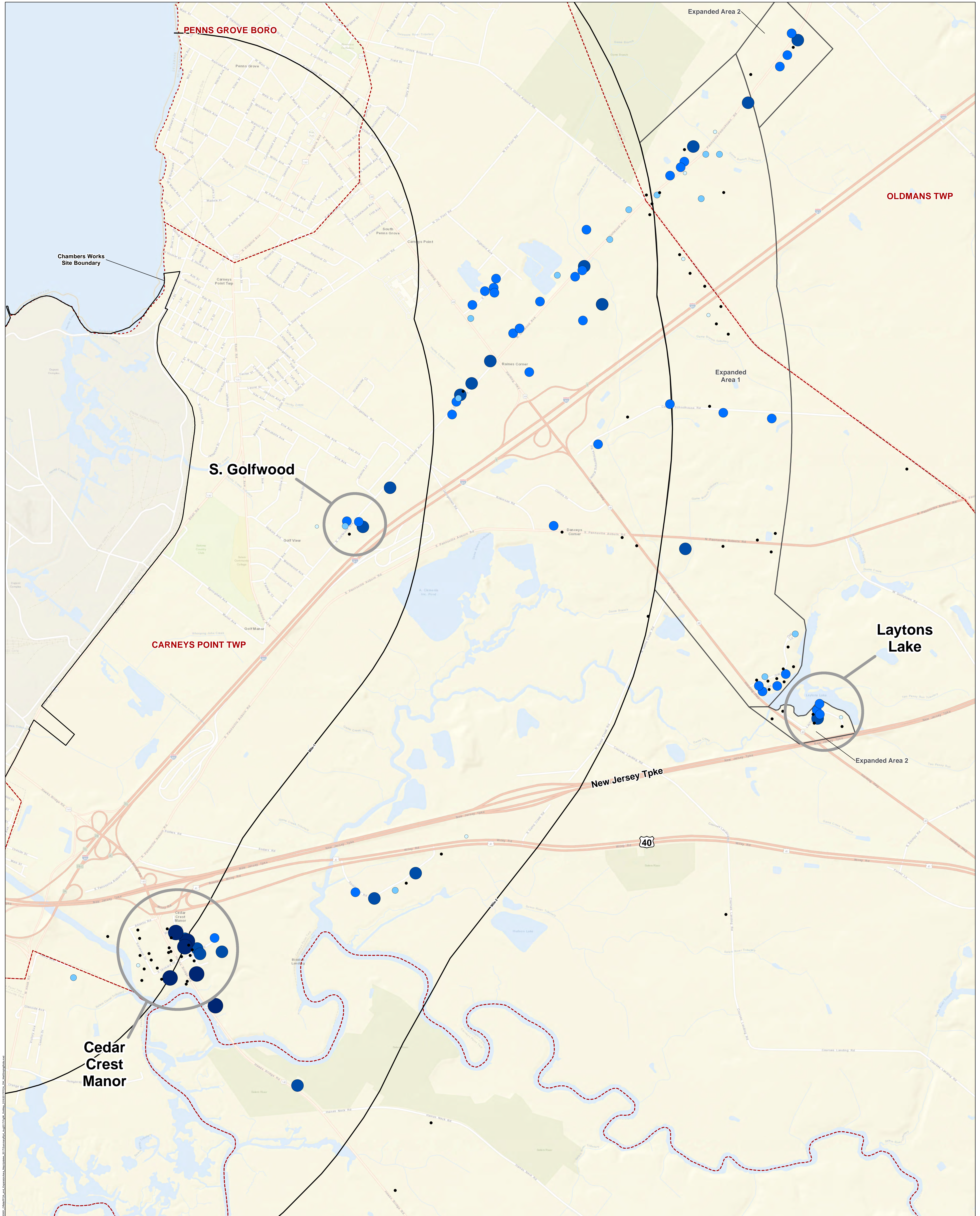
1 inch = 5,000 feet

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**EXPANDED AREAS MAP**

2016-2017 RESIDENTIAL DRINKING WATER  
 WELL SURVEYING AND SAMPLING PROGRAM  
 CHEMOURS CHAMBERS WORKS COMPLEX  
 DEEPWATER, NEW JERSEY

TASK NUMBER:	17006	PROJECT NUMBER:	60533851
DESIGNED BY:	K. L. DAVIS	DATE:	9/7/2017
DRAWN BY:	C. DUFFY	FIGURE NUMBER:	3
DATA QUALITY CHECK BY:	S. MURPHY		



**LEGEND**

- Mile Radius Interval
- Municipal Boundary
- Property Boundary
- Expanded Areas 1 and 2 (Approximate)

**Perfluorooctanoic Acid (PFOA)**

- No Detection
- 2 - 9.9 ppt
- 10 - 49 ppt
- 50 - 99 ppt
- 100 - 180 ppt
- 240 - 500 ppt

World Street Map Sources: Esri, HERE, DeLorme, USGS, Intermap, increment P Corp., NRCAN, Esri Japan, METI, Esri China (Hong Kong), Swis (Tailand), TomTom, Magnum, OpenStreetMap contributors, and the GIS User Community

Scale: 1 inch = 600 feet

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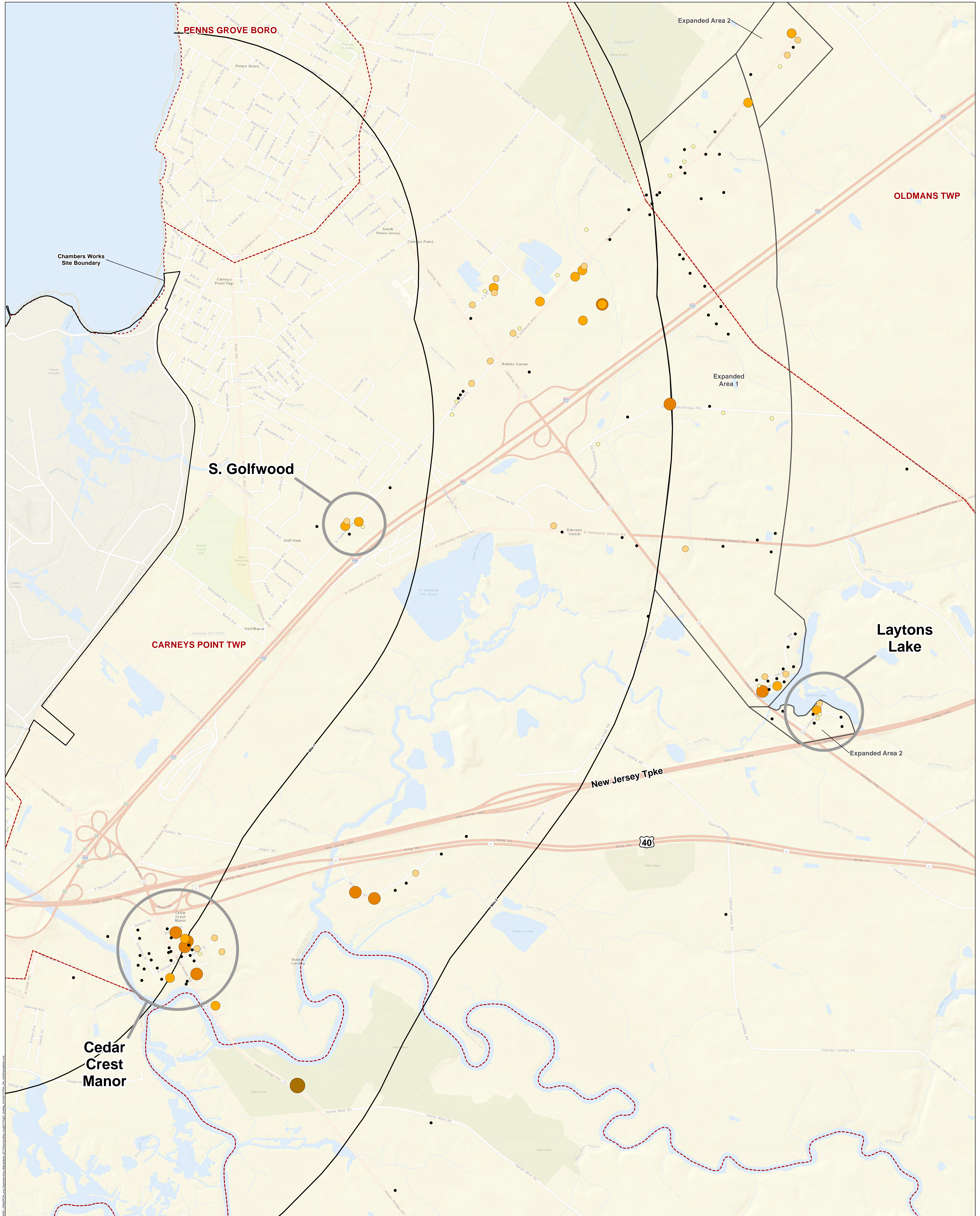
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PROJECT NUMBER: 60533851

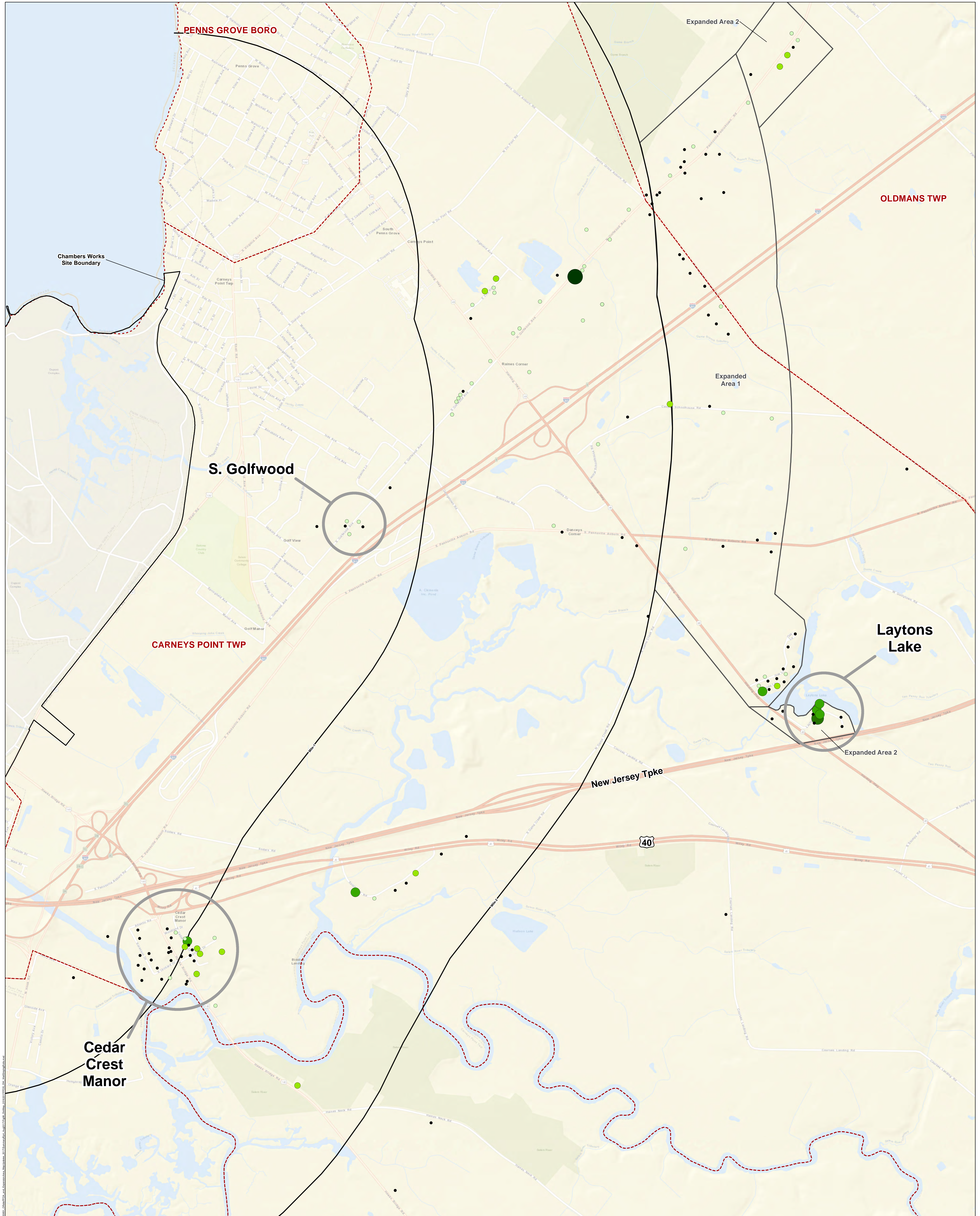
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**AECOM**

2016-2017 RESIDENTIAL DRINKING WATER SURVEYING AND SAMPLING PROGRAM  
CHEMOURS CHAMBERS WORKS COMPLEX  
DEERFIELD, NEW JERSEY



<b>LEGEND</b> Mile Radius Interval Property Boundary Municipal Boundary Expanded Areas 1 and 2 (Approximate)	<b>Perfluoronanoic Acid (PFNA)</b> • No Detection • 2 - 4.9 ppt		• 5 - 9.9 ppt • 10 - 19 ppt • 20 - 36 ppt • 140 ppt		World Street Map Sources: Esri, HERE, DeLorme, USGS, Intermap, Incorporeo P Corp., NRCAN, Esri Japan, METI, Esri China (Hong Kong), Swis (Thailand), TomTom, Mapbox, © OpenStreetMap contributors, and the GIS User Community  MAP FORMATTED FOR 11" (94" X 44") SIZE SHEET TEXT SCALE NOT VALID FOR DIFFERENT PAGE SIZE	TASK NUMBER: 17006 DESIGNED BY: K. L. DAVIS DRAWN BY: C. DUFFY DATA QUALITY CHECK BY: B. CUENTO	 AECOM 601 Corporate Road Newark, DE 19713	DOT MAP OF 2016-2017 PFNA DRINKING WATER WELL SAMPLING RESULTS 2016-2017 RESIDENTIAL DRINKING WATER WELL SURVEYING AND SAMPLING PROGRAM CHEMOURS CHARGES WORKS COMPLEX DEERWATER, NEW JERSEY	PROJECT NUMBER: 60533951 DATE: 9/7/2017 SHEET NUMBER: 7



**LEGEND**

- Mile Radius Interval
- Property Boundary
- Municipal Boundary
- Expanded Areas 1 and 2 (Approximate)

**Perfluorooctansulfonate (PFOS)**

- No Detection
- 2 - 9.9 ppt
- 10 - 19 ppt
- 20 - 55 ppt
- 320 ppt
- 1,300 ppt

World Street Map Sources: Esri, HERE, DeLorme, USGS, Intermap, Incorporeo P Corp., NRCAN, Esri Japan, METI, Esri China (Hong Kong), Swis (Thailand), TomTom, Mapbox, and OpenStreetMap contributors, and the GIS User Community

Scale: 1 inch = 600 feet

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 DESIGNED BY: K. L. DAVIS  
 DRAWN BY: C. DUFFY  
 DATA QUALITY CHECK BY: B. CUENTO

**AECOM**

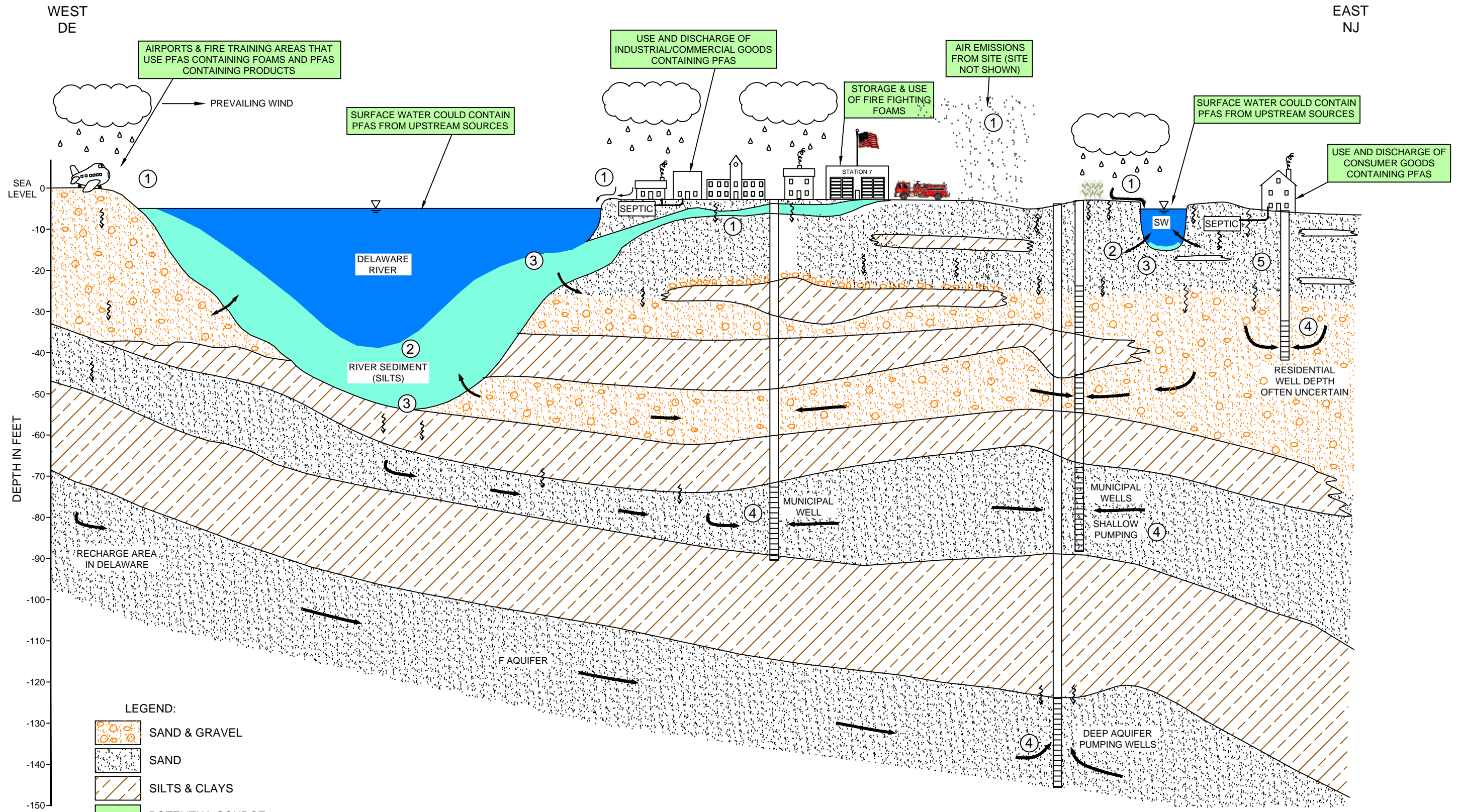
AECOM  
 601 Corporate Road  
 Newark, DE 19713

**DOT MAP OF 2016-2017 PFOS DRINKING WATER WELL SAMPLING RESULTS**

2016-2017 RESIDENTIAL DRINKING WATER WELL SURVEYING AND SAMPLING PROGRAM  
 CHEMOURS CHAMBERS WORKS COMPLEX  
 DEERWATER, NEW JERSEY

PROJECT NUMBER: 60533951  
 DATE: 9/7/2017  
 SHEET NUMBER: 8

V:\Projects\Dupont\Chambers\_Works\CAD\_1\60483012 - PFOA Report\112017\PFAS Conceptual Site Model Cross-Section2.dwg, 9/7/2017 4:17 PM, Littel, David E., PDF995.pc3, User2767, 1'-0" = 1'-0"



- LEGEND:**
- SAND & GRAVEL
  - SAND
  - SILTS & CLAYS
  - POTENTIAL SOURCE
  - WATER ELEVATION
  - DIRECTION OF GROUNDWATER FLOW
  - DOWNWARD GROUNDWATER MIGRATION

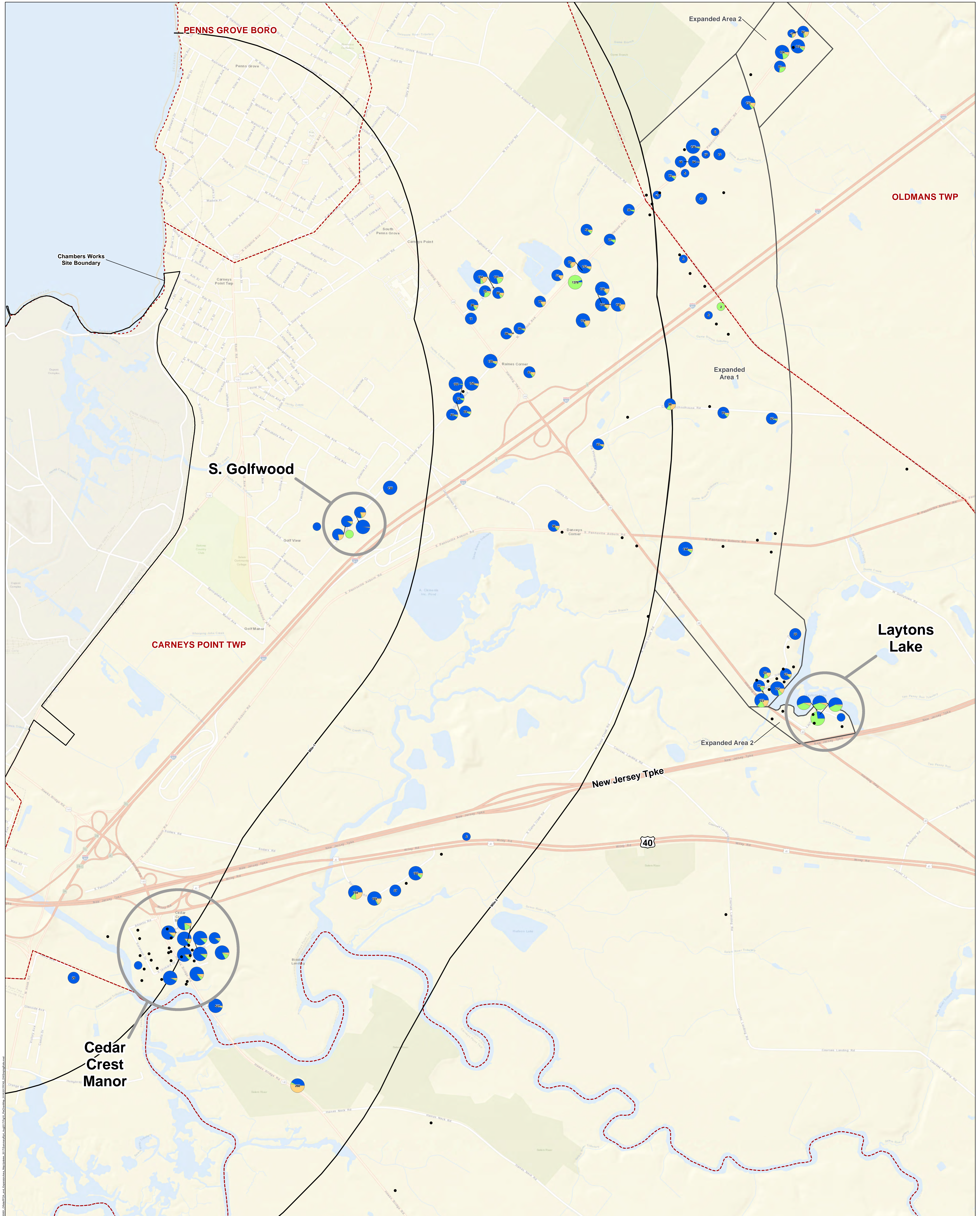
**NOTE:**  
DRAWING IS A SCHEMATIC CROSS-SECTION, BUT REPRESENTS CONDITIONS ACROSS THE SITE AND IS NOT TO SCALE.

DESIGNED BY:  
K. WEST  
DRAWN BY:  
D. LITTEL  
DATA QUALITY CHKD:  
T. MCGEE  
APPROVED BY:

**AECOM**  
Sabre Building, Suite 300  
4051 Ogletown Road  
Newark, Delaware 19713  
Phone: 302-781-5900

**CONCEPTUAL CROSS-SECTION LOCAL OFF-SITE**  
  
2016-2017 RESIDENTIAL DRINKING WATER WELL SURVEYING AND SAMPLING PROGRAM  
CHEMOURS CHAMBERS WORKS COMPLEX DEEPWATER, NEW JERSEY

PROJECT NO.  
60533851  
DATE  
4/6/17  
FIGURE No.  
9



PENNS GROVE BORO

Expanded Area 2

OLDMANS TWP

Chambers Works Site Boundary

S. Golfwood

Expanded Area 1

CARNEYS POINT TWP

Laytons Lake

New Jersey Tpke

Expanded Area 2

Cedar Crest Manor

<b>LEGEND</b> — Mile Radius Interval    - - - Municipal Boundary □ Property Boundary    □ Expanded Areas 1 and 2 (Approximate)		• No Detection of PFOA, PFOS or PFNA • Label indicates the sum (ppt) of PFOA, PFOS, and PFNA.	 PFOA %    PFOS %    PFNA % 0.01 - 10 ppt    >10 - 100 ppt    >100 ppt	PFOA = Perfluorooctanoic acid PFOS = Perfluorooctanesulfonic acid PFNA = Perfluorononanoic acid	World Street Map Sources: Esri, HERE, DeLorme, USGS, Intermap, increment P Corp., NRCAN, Esri/Japan, METI, Esri/China (Hong Kong), Esri (Thailand), TomTom, Mapbox, © OpenStreetMap contributors, and the GIS User Community	 0 325 650 1,300 Feet 1 inch = 650 feet MAP FORMATTED FOR 11" (94" X 44") SIZE SHEET. TEXT SCALE NOT VALID FOR DIFFERENT PAGE SIZE.	TASK NUMBER: 17006 DESIGNED BY: K. L. DAVIS DRAWN BY: C. DUFFY DATA QUALITY CHECK BY: B. CUENTO	 AECOM Sabre Building, Suite 300 4501 Corporate Road Newark, DE 19713	PROJECT NUMBER: 62633951 DATE: 9/7/2017 SHEET NUMBER: 10
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PIE CHART MAP OF 2016-2017 PFOA, PFOS, AND PFNA DRINKING WATER WELL SAMPLING RESULTS

## Appendices

## **Appendix B**

### **2016-2017 Surveying and Sampling Program Documentation for Residents**





The Chemours Company  
1007 Market Street  
PO Box 2047  
Wilmington, DE 19899

302-773-1000 t  
chemours.com

Letter No.:1

June 13, 2016

[REDACTED]  
or Current Resident

[REDACTED]  
Mannington, NJ 08079

### **Chemours Private Drinking Water Well Sampling Program (2016)**

In 2009, E. I. du Pont de Nemours and Company (DuPont) worked in collaboration with the New Jersey Department of Environmental Protection (NJDEP) to conduct a Perfluorooctanoic Acid (PFOA) Private Drinking Water Well Survey and Sampling Program within a two-mile radius surrounding the Chambers Works facility, located in Deepwater, New Jersey. DuPont sampled private drinking water wells to determine if PFOA concentrations in those wells measured 0.40 parts per billion (ppb), or 0.40 micrograms per liter ( $\mu\text{g/L}$ ), or greater, which is the provisional health advisory established by the U. S. Environmental Protection Agency (EPA) Office of Water on January 8, 2009. EPA developed a drinking water health advisory for PFOA of 0.07 ppb in May 2016, as explained at <https://www.epa.gov/ground-water-and-drinking-water/drinking-water-health-advisories-pfoa-and-pfos>. In addition, New Jersey Department of Environmental Protection (NJDEP) has developed an interim specific groundwater quality criterion for perfluorononanoic acid (PFNA) of 0.01 ppb.

On July 1, 2015, The Chemours Company (Chemours) became an independent publically traded company through the spin-off of DuPont Performance Chemicals. Chemours is working in collaboration with U.S. EPA Region 2 and NJDEP to conduct a drinking water well sampling program.

Chemours requests your permission to sample your drinking water well at this time. The water from your well will be analyzed for PFOA, PFNA, and other perfluorinated compounds, and the results will be provided at no cost to you. Your participation in this sampling event is greatly appreciated. We encourage you to schedule your drinking water well for sampling as soon as possible by contacting Ms. Linda Wallace of Chemours at **856-540-2830**. The sampling will be scheduled at your convenience and requires a technician to come to your house for less than 10 minutes to collect a small container of water.

For your information, additional facts regarding perfluorinated compounds can be found at <https://www.epa.gov/assessing-and-managing-chemicals-under-tsca/perfluorooctanoic-acid-pfoa-perfluorooctyl-sulfonate>.

If you have any questions regarding this sampling program, please feel free to contact Ms. Pat Seppi of U.S. EPA Region 2 at **646-369-0068**.

Sincerely,

The Chemours Company

A handwritten signature in black ink, appearing to read "Andrew S. Hartten".

Andrew S. Hartten

Principal Remediation Project Manager  
Corporate Remediation Group

cc: Sin-Kie Tjho, EPA Region 2  
Linda Range, NJDEP



# U.S. Environmental Protection Agency Recommends Water Testing for People Within a Two-mile Radius of the Chambers Works Facility in Deepwater, NJ

Community Fact Sheet

November 2016

## COMMUNITY HIGHLIGHTS

### **PERFLUOROALKYL SUBSTANCES**

Perfluorooctanoic acid (PFOA), perfluorooctane sulfonate (PFOS) and perfluorononanoic acid (PFNA) are fluorinated organic chemicals that are part of a larger group of chemicals referred to as perfluoroalkyl substances (PFAS).

### **CHEMOURS CONTINUES TO SAMPLE PRIVATE DRINKING WATER WELLS**

Chemours has agreed to sample private drinking water wells located within a two-mile radius of the Chambers Works facility at no cost to residents. ***EPA strongly recommends*** that residents have their private well water sampled and tested for PFAS.

**EPA HEALTH ADVISORY LEVEL:** 70 parts per trillion (ppt) of PFOA/PFOS

**NJDEP HEALTH BASED DRINKING WATER GUIDANCE:** 40 ppt PFOA

**NJDEP INTERIM GROUNDWATER QUALITY STANDARD:** 10 ppt PFNA

**For more information regarding the EPA Health Advisory please visit the following website:**

[https://www.epa.gov/sites/production/files/2016-06/documents/drinkingwaterhealthadvisories\\_pfoa\\_pfos\\_updated\\_5.31.16.pdf](https://www.epa.gov/sites/production/files/2016-06/documents/drinkingwaterhealthadvisories_pfoa_pfos_updated_5.31.16.pdf)

**To request sampling or inquire about the Chemours sampling program please contact the following:**

#### **EPA Community Involvement Coordinator**

Pat Seppi  
646-369-0068 - cell  
212-637-3679 - office  
[Seppi.Pat@epa.gov](mailto:Seppi.Pat@epa.gov)

OR

#### **EPA Remedial Project Manager**

Theresa Hwilka  
(212) 637-4409  
[Hwilka.Theresa@epa.gov](mailto:Hwilka.Theresa@epa.gov)

The U.S. Environmental Protection Agency (EPA) recommends that people living within a 2-mile radius of the Chambers Works Facility get their drinking water well tested for a group of chemicals called perfluoroalkyl substances (PFAS).

### **What are Perfluoroalkyl substances?**

Perfluorooctanoic acid (PFOA), perfluorooctane sulfonate (PFOS) and perfluorononanoic acid (PFNA) are fluorinated organic chemicals that are part of a larger group of chemicals referred to as perfluoroalkyl substances (PFAS). PFOA and PFOS have been the most extensively produced and studied of these chemicals. They have been used to make carpets, clothing, fabrics for furniture, paper packaging for food and other materials (e.g., cookware) that are resistant to water, grease or stains. They are also used for firefighting at air-fields and in a number of industrial processes.

While consumer products and food are a large source of exposure to these chemicals for most people, drinking water can be an additional source in the small percentage of communities where these chemicals have contaminated water supplies. Such contamination is typically localized and associated with a specific facility, for example, an industrial facility where these chemicals were produced or used to manufacture other products or an airfield at which they were used for firefighting.

### **Private drinking water well sampling**

In 2009, E. I. du Pont de Nemours and Company (DuPont) worked with the New Jersey Department of Environmental Protection (NJDEP) to conduct a PFOA Private Drinking Water Well Survey and Sampling Program within a two-mile radius surrounding the Chambers Works facility, located in Deepwater, New Jersey. DuPont sampled private drinking water wells to determine if PFOA concentrations in those wells exceeded PFOA health advisory levels at that time.

On July 1, 2015, The Chemours Company (Chemours) became an independent company through the spin-off of DuPont Performance Chemicals. After the new PFOA/PFOS health advisory levels were published by EPA in May 2016, Chemours did a second round of residential well sampling in June 2016.

Sampling results for the second round of sampling indicate that of the 76 samples collected thus far, 31 samples had PFAS results exceeding the current EPA health advisory levels and/or NJDEP guidance level or standard. Chemours is working with EPA Region 2 and NJDEP to continue conducting the Private Drinking Water Well Survey and Sampling Program.

Previous attempts to contact residents and sample private wells have been made by Chemours; however, approximately 60% of the residents within the two-mile radius have not yet responded.

**EPA strongly recommends** that people located within a two-mile radius of the facility (see map below) who have a private drinking water well get that well tested free of charge. To get your well tested, contact Pat Seppi, EPA Community Involvement Coordinator or Theresa Hwilka, EPA Project Manager via the information provided in the “Community Highlights” box of this fact sheet.

If your well water is found to exceed the EPA health advisory levels or NJDEP standards for PFAS, bottled water will be provided until point-of-entry treatment systems (known as POETS) can be installed. POETS are small treatment systems that will remove contaminants from well water using granulated activated carbon. The treatment systems must be periodically sampled and replaced to ensure they are working properly. The sampling, bottled water and installation and upkeep of the treatment systems will be provided at **NO COST** to eligible residents.

### Health Advisories

EPA develops health advisories to provide information on contaminants that can cause human health effects and are known or anticipated to occur in drinking water. To provide people, including the most sensitive populations, with a margin of protection from a life-time of exposure to PFOA and PFOS from drinking water, EPA established the health advisory levels at 70 parts per trillion (ppt) in May 2016. When both PFOA and PFOS are found in drinking water, the combined concentrations of PFOA and PFOS should be compared with the 70 ppt health advisory level.

NJDEP has developed a guideline for chronic (lifetime) exposures to PFOA of 40 ppt. NJDEP has also established an interim specific groundwater quality standard for perfluorononanoic acid (PFNA) of 10 ppt.

### CHEMOURS COMPANY PRIVATE DRINKING WATER WELL SURVEY AND SAMPLING PROGRAM TWO-MILE RADIUS





The Chemours Company  
1007 Market Street  
PO Box 2047  
Wilmington, DE 19899

302-773-1000 t  
chemours.com

May 3, 2017

### **Chemours Private Drinking Water Well Sampling Program (2016-2017)**

In 2009, E. I. du Pont de Nemours and Company (DuPont) worked in collaboration with the New Jersey Department of Environmental Protection (NJDEP) to conduct a Perfluorooctanoic Acid (PFOA) Private Drinking Water Well Survey and Sampling Program within a two-mile radius surrounding the Chambers Works facility, New Jersey. DuPont sampled private drinking water wells to determine if PFOA concentrations in those wells measured 0.40 parts per billion (ppb), or 0.40 micrograms per liter ( $\mu\text{g/L}$ ), or greater, which is the provisional health advisory established by the U. S. Environmental Protection Agency (EPA) Office of Water on January 8, 2009. EPA developed a drinking water health advisory for PFOA of 0.07 ppb in May 2016, as explained at <https://www.epa.gov/ground-water-and-drinking-water/drinking-water-health-advisories-pfoa-and-pfos>. In addition, NJDEP is establishing a maximum contaminant level (MCL) for perfluorononanoic acid (PFNA) of 0.013 ppb.

On July 1, 2015, The Chemours Company (Chemours) became an independent publically traded company through the spin-off of DuPont Performance Chemicals. Chemours is currently working in collaboration with U.S. EPA Region 2 and NJDEP to conduct another drinking water well sampling program in approximately the same area.

Results for the current sampling activities in your area have shown the presence of PFOA, PFOS and PFNA in drinking water. Chemours requests your permission to sample your drinking water well at this time and is encouraging your participation in the sampling program. If you accept the sampling offer, the water from your well will be analyzed for PFOA, PFNA, and other perfluorinated compounds, and the results will be provided at no cost to you. Your participation in this sampling event is greatly appreciated. We encourage you to schedule your drinking water well for sampling as soon as possible by contacting Ms. Shannon Murphy of Chemours at **856-981-1510**. The sampling will be scheduled at your convenience and requires a technician to come to your house for less than 10 minutes to collect a small container of water.

For your information, additional facts regarding perfluorinated compounds can be found at <https://www.epa.gov/assessing-and-managing-chemicals-under-tsca/perfluorooctanoic-acid-pfoa-perfluorooctyl-sulfonate>.

If you have any questions regarding this sampling program, please feel free to contact Ms. Pat Seppi of U.S. EPA Region 2 at **646-369-0068**.

Sincerely,

The Chemours Company

A handwritten signature in black ink, appearing to read "Andrew S. Hartten".

Andrew S. Hartten

Principal Remediation Project Manager  
Corporate Remediation Group

cc: Sam Abdellatif, EPA Region 2  
Linda Range, NJDEP



The Chemours Company  
1007 Market Street  
PO Box 2047  
Wilmington, DE 19899

302-773-1000 t  
chemours.com

May 3, 2017

### **Chemours Private Drinking Water Well Sampling Program (2016-2017)**

In 2009, E. I. du Pont de Nemours and Company (DuPont) worked in collaboration with the New Jersey Department of Environmental Protection (NJDEP) to conduct a Perfluorooctanoic Acid (PFOA) Private Drinking Water Well Survey and Sampling Program within a two-mile radius surrounding the Chambers Works facility, New Jersey. DuPont sampled private drinking water wells to determine if PFOA concentrations in those wells measured 0.40 parts per billion (ppb), or 0.40 micrograms per liter ( $\mu\text{g/L}$ ), or greater, which is the provisional health advisory established by the U. S. Environmental Protection Agency (EPA) Office of Water on January 8, 2009. EPA developed a drinking water health advisory for PFOA of 0.07 ppb in May 2016, as explained at <https://www.epa.gov/ground-water-and-drinking-water/drinking-water-health-advisories-pfoa-and-pfos>. In addition, NJDEP is establishing a maximum contaminant level (MCL) for perfluorononanoic acid (PFNA) of 0.013 ppb.

On July 1, 2015, The Chemours Company (Chemours) became an independent publically traded company through the spin-off of DuPont Performance Chemicals. Chemours is currently working in collaboration with U.S. EPA Region 2 and NJDEP to conduct another drinking water well sampling program in approximately the same area.

Results for the current sampling activities in your area have shown the presence of PFOA, PFOS and PFNA in drinking water. Chemours requests your permission to sample your drinking water well at this time and is encouraging your participation in the sampling program. If you accept the sampling offer, the water from your well will be analyzed for PFOA, PFNA, and other perfluorinated compounds, and the results will be provided at no cost to you. Your participation in this sampling event is greatly appreciated. We encourage you to schedule your drinking water well for sampling as soon as possible by contacting Ms. Shannon Murphy of Chemours at **856-981-1510**. The sampling will be scheduled at your convenience and requires a technician to come to your house for less than 10 minutes to collect a small container of water.

For your information, additional facts regarding perfluorinated compounds can be found at <https://www.epa.gov/assessing-and-managing-chemicals-under-tsca/perfluorooctanoic-acid-pfoa-perfluorooctyl-sulfonate>.

If you have any questions regarding this sampling program, please feel free to contact Ms. Pat Seppi of U.S. EPA Region 2 at **646-369-0068**.

Sincerely,

The Chemours Company

A handwritten signature in black ink, appearing to read "Andrew S. Hartten".

Andrew S. Hartten

Principal Remediation Project Manager  
Corporate Remediation Group

cc: Sam Abdellatif, EPA Region 2  
Linda Range, NJDEP

## **Appendix E**

### **Quality Assurance Project Plan for the Chemours 2016 PFAS Residential Drinking Water Well Surveying and Sampling Program, Chambers Works, Deepwater, New Jersey**



The Chemours Company  
1007 Market Street  
PO Box 2047  
Wilmington, DE 19899

302-773-1000 t  
chemours.com

June 2, 2016

Mr. Sin-Kie Tjho  
U.S. EPA Region 2 – 22<sup>nd</sup> Floor  
290 Broadway  
New York, NY 10007-1866

**Submission of the Quality Assurance Project Plan for the Chemours 2016 PFAS Residential Drinking Water Well Surveying and Sampling Program, Chambers Works, Deepwater, New Jersey**

Dear Mr. Sin Kie Tjho:

Attached please find the Quality Assurance Project Plan (QAPP) for use with the Chemours 2016 PFAS Residential Drinking Water Well Surveying and Sampling Program, which is described below. The attached QAPP was prepared for the sampling and analysis of the drinking water well samples that will be collected under the Chemours 2016 Residential Drinking Water Well Surveying and Resampling Program. This QAPP was prepared using the Uniform Federal Policy for Quality Assurance Project Plans as requested by EPA. A DRAFT QAPP was submitted to EPA on April 19, 2016. Comments received by Chemours on April 29, 2016 and on June 1, 2016 have been addressed.

In 2009, E. I. du Pont de Nemours and Company (DuPont) worked in collaboration with the New Jersey Department of Environmental Protection (NJDEP) to conduct a Perfluorooctanoic Acid (PFOA) Private Drinking Water Well Survey and Sampling Program within a two-mile radius surrounding the Chambers Works facility, located in Deepwater, New Jersey. DuPont collected 113 samples from 111 private drinking water wells to determine if PFOA concentrations in those wells measured 0.40 parts per billion (ppb), or 0.40 micrograms per liter ( $\mu\text{g/L}$ ), or greater, which is the provisional health advisory established by the U. S. Environmental Protection Agency (EPA) Office of Water on January 8, 2009. Based on the results of the 2009 sampling, one residential granular activated carbon (GAC) treatment system was installed.

On May 19, 2016, EPA developed a drinking water health advisory for PFOA, and perfluorooctanesulfonic acid (PFOS) or PFOA plus PFOS of 0.07 ppb. In addition, NJDEP recently developed an interim specific groundwater quality criterion for perfluorononanoic acid (PFNA) of 0.01 ppb. The Chemours 2016 PFAS Residential Drinking Water Well Surveying and Resampling Program will begin within three days of EPA's approval of the attached QAPP. Drinking water wells sampled under this program will be qualified for treatment if measured concentrations of PFOA or PFNA are detected above the respective criteria listed above.

Under this new program, free sampling and analysis will be offered to all residents who were contacted during the 2009 surveying and sampling event, regardless of whether they responded to the program in 2009 or not. All wells currently used as drinking water sources will be qualified for sampling or resampling, as appropriate. A spreadsheet was developed for use in this surveying and sampling program and includes contact information and previous sampling results. A call center has been established to receive calls from residents interested in the program and will use the spreadsheet to document calls into the call center and related information. Table 1 provides a list of the drinking water wells/residences that will be contacted and contact information for the residents. The locations of these residences and results for those wells sampled in 2009 are provided in Figure 1 and are identified by letter number as indicated in Table 1.

Certified letters will be sent to the addresses used in the 2009 surveying and sampling program, but the letters will be addressed to the owner identified in 2009 or the "current resident" to encourage residents to respond even if the property has changed ownership since 2009. The call center will document information from the residents who call, and will schedule the sampling or resampling activities at the resident's convenience<sup>1</sup>. All residents who do not respond to the letter requesting their participation in this program within three weeks of the mailing of the first certified letter will be identified as non-response in the database. A second certified letter requesting their participation will be then mailed. If no response is received within three weeks from the mailing of the second letter, the residents contact information will be provided to EPA for appropriate follow up by EPA. Certified letters that are returned to Chemours, or its designated representative, will be documented in the database, and a second certified letter will be mailed three weeks after the first to provide a second opportunity for the resident or current owner to decide to participate. If no response is given within three weeks of the mailing of the second certified letter, the residents contact information will be provided to EPA for appropriate follow up by EPA.

Drinking water well samples will be analyzed for perfluoroalkyl substances (PFASs) by TestAmerica (West Sacramento, California) using EPA Method 537 Modified, as indicated in the attached QAPP. If a drinking water well is identified in which the measured concentration of PFOA is greater than EPA's Health Advisory of 0.07 ppb and/or if the measured concentration of perfluorononanoic acid (PFNA) is greater than NJDEP interim specific groundwater quality criterion of 0.01 ppb, an offer for treatment will be made. Treatment may include installation of a residential GAC treatment system or another EPA-approved alternative, typically, but not limited to connection to a GAC treated public water supply. Treatment provided to the resident/property owner and operation and maintenance of the system, including quarterly sampling, will be provided by Chemours at no cost to the resident/owner.

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<sup>1</sup> If the resident is not the owner of the property, the owner of the property will need to be contacted and give consent for sampling to take place. The owner will also receive results from any sampling completed. If the drinking water well qualifies for treatment, the owner's consent would be required prior to installing or offering treatment.



Mr. Sin-Kie Tjho  
June 2, 2016  
Page 3

As indicated above, the Chemours 2016 PFAS Residential Drinking Water Well Surveying and Resampling Program will begin within three days of EPA's approval of the attached QAPP. Please contact me at 302-773-1289 if you have any questions.

Sincerely,  
The Chemours Company

A handwritten signature in black ink, appearing to read "Andrew S. Hartten".

Andrew S. Hartten

Principal Remediation Project Manager  
Chemours Corporate Remediation Group

cc: Linda Range (NJDEP) -- electronic



Environment

Submitted on behalf of  
The Chemours Company

Submitted by  
AECOM  
Sabre Building  
Suite 300  
4051 Ogletown Road  
Newark, DE 19713

# Quality Assurance Project Plan

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

Project #: 60392286

Submitted: May 2016

Revised: June 2016

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Appendix B PFCs Sampling Checklist

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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 within approximately two miles 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 (EPA) 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. Chemours<sup>1</sup> Project Number: 509026
  - d. AECOM Project Number: 60392286
2. Lead Organization: Chemours Corporate Remediation Group (CRG)
3. Lead Contractor: AECOM
4. Approvals:

_____ Andrew Hartten, Chemours Project Director	_____ Date
_____ Mark Houlday, AECOM Project Manager	_____ Date
_____ Katherine Davis, AECOM Project Technical Adviser	_____ Date
_____ Michael Aucoin, AECOM Project Chemist	_____ Date
_____ Lance Holman, AECOM Quality Assurance (QA) Officer	_____ Date
_____ Sin Kie Tjho, EPA Region 2	_____ Date

<sup>1</sup> 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.

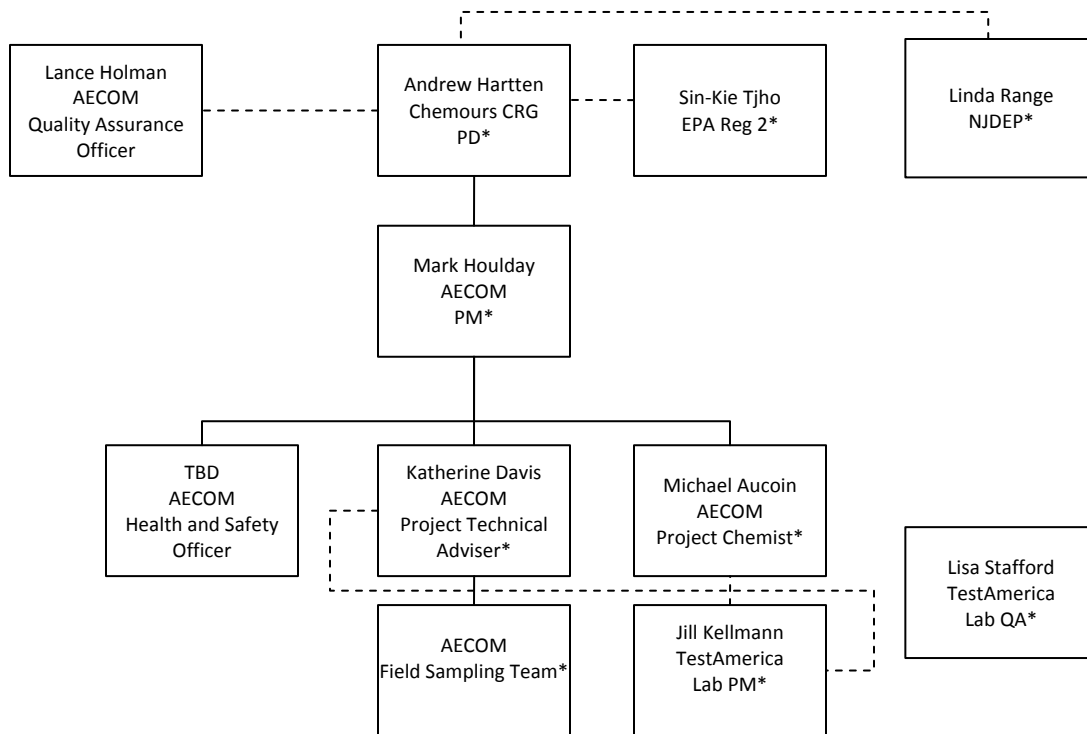


**QAPP Worksheet #3 & 5: Project Organization and QAPP Distribution  
(UFP-QAPP Manual Section 2.3 and 2.4)  
(EPA 2106-G-05 Section 2.2.3 and 2.2.4)**

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
Mark Houlday	Project Manager	BS, PG	PFASs Sampling Checklist	
Katherine Davis	Project Technical Adviser	PhD	PFASs Sampling Checklist	
Lance Holman	QA Officer	BS	PFASs Sampling Checklist	
Michael Aucoin	Project Chemist	BS	PFASs Sampling Checklist	

ORGANIZATION: TestAmerica Sacramento

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Lisa Stafford	QA Manager	BS	40 hour TestAmerica QA Manager	
Jill Kellmann	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 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**

Mark Houlday, 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 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**

AECOM will provide a health and safety officer for the project. The health and safety officer will be responsible for developing, reviewing, and approving of 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:

- Developing and 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**

Michael Aucoin, 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 coordinate internal and third party review of data generated by the laboratory.

### **Laboratory Personnel**

TestAmerica Sacramento, located in West Sacramento, California, is the laboratory that will perform sample analysis. 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 laboratory project manager, Jill Kellmann. 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 laboratory has 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.

<b>Communication Driver</b>	<b>Organization</b>	<b>Name</b>	<b>Contact Information</b>	<b>Procedure (timing, pathway, documentation, etc.)</b>
Regulatory agency interface	Chemours	Andrew Hartten	<a href="mailto:Andrew.S.Hartten@AECOM.com">Andrew.S.Hartten@AECOM.com</a>  302-773-1289	As needed, phone calls and email
Call Center referrals to EPA (Pat Seppi, 646-369-0068)	AECOM	Linda Wallace	<a href="mailto:Linda.Wallace@AECOM.com">Linda.Wallace@AECOM.com</a> 856-540-2830	As needed, Referrals documented in the call center master spreadsheet <sup>2</sup> and in the summary spreadsheet <sup>3</sup> included in Monthly Status Reports

<sup>2</sup> The call center master spreadsheet includes (but is not limited to) letter numbers, names, contact information, GPS coordinates, sample ID, date sampled and results for samples collected in 2009 and contact information for non-responsive residents from the 2009 surveying. It also includes 2016 letter numbers; questions to be asked to the residents to confirm/correct names, addresses and provide data concerning renters versus property owners; qualification for sampling in 2016, including phone numbers, any comments/questions; and any information on scheduling and sampling that is completed during the 2016 surveying and sampling. This spreadsheet will also include information on letters returned to Chemours and those residents that are non-responsive to the request for sampling. Columns of information may be added to the call center master spreadsheet as needed, based on conditions encountered during the call center operation.

<sup>3</sup> The summary spreadsheets included in the monthly status reports to EPA and NJDEP will include information such as (but not limited to) residents contact information, sample information, status of non-responsive residents and follow-up contact attempts, and comments. Columns of information may be added to the summary spreadsheets as needed based on conditions encountered during the call center operation and the surveying and sampling.

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Qualification Determination	AECOM	Linda Wallace/Kathy Davis	<p><a href="mailto:Linda.Wallace@AECOM.com">Linda.Wallace@AECOM.com</a> 856-540-2830</p> <p><a href="mailto:Katherine.I.davis@AECOM.com">Katherine.I.davis@AECOM.com</a> 302-781-5890</p>	Communication between Linda Wallace and Kathy Davis via phone calls, emails and call center spreadsheet review and documented in the call center master spreadsheet and in the summary spreadsheet included in Monthly Status Reports
Sample Scheduling	AECOM	Linda Wallace/Field Samplers (TBD)	<p><a href="mailto:Linda.Wallace@AECOM.com">Linda.Wallace@AECOM.com</a> 856-540-2830</p> <p>Field Samplers (TBD)</p>	Communication between Linda Wallace and Field Samplers via phone calls, emails and spreadsheet review and documented in the call center master spreadsheet and in the summary spreadsheet included in Monthly Status Reports
Sample Collection	AECOM	Field Samplers (TBD)	Field Samplers (TBD)	Communication between Kathy Davis and Field Samplers via phone calls, emails and spreadsheet review and documented in the call center master spreadsheet and in the summary spreadsheet included in Monthly Status Reports.
Stop work due to safety issues	AECOM	Field Samplers/Mark Houlday	<p><a href="mailto:Mark.Houlday@AECOM.com">Mark.Houlday@AECOM.com</a> 302-547-6569</p>	As needed, phone calls between Mark Houlday and Field Staff and documented in emails to EPA and NJDEP.

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Sample Submission/ receipt	AECOM/Test America	Field Samplers (TBD)/Mike Aucoin/Jill Kellmann (or designee)	Field Samplers (TBD)/ <a href="mailto:Michael.Aucoin@AECOM.com">Michael.Aucoin@AECOM.com</a> 302-781-5873 /TestAmerica <a href="mailto:Jill.Kellmann@testamericainc.com">Jill.Kellmann@testamericainc.com</a> 919-373-5600	Communication via emails, phone calls between Field Samplers, Mike Aucoin and TestAmerica
Laboratory quality control variances	AECOM/Test America	Mike Aucoin/Jill Kellmann (or designee)	<a href="mailto:Michael.Aucoin@AECOM.com">Michael.Aucoin@AECOM.com</a> 302-781-5873 /TestAmerica <a href="mailto:Jill.Kellmann@testamericainc.com">Jill.Kellmann@testamericainc.com</a> 919-373-5600	Communication via emails, phone calls between Mike Aucoin and TestAmerica
Analytical corrective actions	AECOM/Test America	Mike Aucoin/Jill Kellmann (or designee)	<a href="mailto:Michael.Aucoin@AECOM.com">Michael.Aucoin@AECOM.com</a> 302-781-5873 /TestAmerica <a href="mailto:Jill.Kellmann@testamericainc.com">Jill.Kellmann@testamericainc.com</a> 919-373-5600	Communication via emails, phone calls between Mike Aucoin and TestAmerica
Data verification issues, e.g., incomplete records	AECOM/Test America	Mike Aucoin/Jill Kellmann (or designee)	<a href="mailto:Michael.Aucoin@AECOM.com">Michael.Aucoin@AECOM.com</a> 302-781-5873 /TestAmerica <a href="mailto:Jill.Kellmann@testamericainc.com">Jill.Kellmann@testamericainc.com</a> 919-373-5600	Communication via emails, phone calls between Mike Aucoin and TestAmerica
Data validation issues, e.g., non-compliance with procedures	AECOM/Test America	Mike Aucoin/Jill Kellmann (or designee)	<a href="mailto:Michael.Aucoin@AECOM.com">Michael.Aucoin@AECOM.com</a> 302-781-5873 /TestAmerica <a href="mailto:Jill.Kellmann@testamericainc.com">Jill.Kellmann@testamericainc.com</a> 919-373-5600	Communication via emails, phone calls between Mike Aucoin and TestAmerica
Data review corrective actions	AECOM/Test America	Mike Aucoin/Jill Kellmann (or designee)	<a href="mailto:Michael.Aucoin@AECOM.com">Michael.Aucoin@AECOM.com</a> 302-781-5873 /TestAmerica <a href="mailto:Jill.Kellmann@testamericainc.com">Jill.Kellmann@testamericainc.com</a> 919-373-5600	Communication via emails, phone calls between Mike Aucoin and TestAmerica
Result letter finalization - preparation	AECOM	Mike Aucoin/Kathy Davis	<a href="mailto:Michael.Aucoin@AECOM.com">Michael.Aucoin@AECOM.com</a> 302-781-5873 / <a href="mailto:Katherine.l.davis@AECOM.com">Katherine.l.davis@AECOM.com</a> 302-781-5890	Communication via emails, phone calls between Mike Aucoin and Kathy Davis
Result letter finalization - transmittal	AECOM/TBD	Kathy Davis/TBD	<a href="mailto:Katherine.l.davis@AECOM.com">Katherine.l.davis@AECOM.com</a> 302-781-5890/TBD	Result letter mailed to resident and copied to EPA 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:

<b>Name</b>	<b>Organization</b>	<b>Title/Role</b>	<b>Email/Phone</b>
Andrew Hartten	Chemours	Project Director	<a href="mailto:Andrew.S.Hartten@chemours.com">Andrew.S.Hartten@chemours.com</a> 302-773-1289
Mark Houlday	AECOM	Project Manager	<a href="mailto:Mark.Houlday@AECOM.com">Mark.Houlday@AECOM.com</a> 302-547-6569
Katherine Davis	AECOM	Project Technical Adviser	<a href="mailto:Katherine.l.davis@AECOM.com">Katherine.l.davis@AECOM.com</a> 302-781-5890
Michael Aucoin	AECOM	Project Chemist	<a href="mailto:Michael.Aucoin@AECOM.com">Michael.Aucoin@AECOM.com</a> 302-781-5873

Notes/Comments:

Consensus decisions made:

Action Items:

<b>Action</b>	<b>Responsible Party</b>	<b>Due Date</b>
Prepare the QAPP	Michael Aucoin, AECOM	April 15, 2016



**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). Please note that the CSM as presented below is incomplete and is not intended to reflect the entire site. The information gathered during completion of the PFAS drinking water sampling program will fill in data gaps and allow for the CSM to be revised as new data become available.

PFASs, specifically PFOA, were determined to potentially be associated with five processes at the Chambers Works site. As indicated by DuPont and NJDEP models, the primary transport pathway from these on-site sources to off-site environmental media is via migration of historical air emissions. As of 2014, the Chambers Works site had achieved a greater than 89% reduction in air emission across the site. The air transport model has been confirmed through various data collections that have been conducted since 2008 including the following:

- The on-site groundwater monitoring program conducted since early 2008 under NJPDES-DGW Permit No. NJ0083429.
- A voluntary sampling event of 20 new and existing off-site groundwater monitoring wells within a two mile radius of the site in 2009-2010.

A voluntary program to evaluate private drinking water wells within a two-mile radius of the Chambers Works site, which included collection of 113 samples from 111 private drinking water wells in 2009, provided additional data that support the air transport model.

This monitoring has demonstrated declining concentrations with distance from the manufacturing areas. Up to approximately 100 micrograms per liter ( $\mu\text{g/L}$ ) of PFOA were measured in shallow groundwater adjacent to the fluorotelomer process area on-site, approximately 1  $\mu\text{g/L}$  in shallow groundwater near the site boundary, and, most importantly, levels of PFOA in groundwater at or near the two mile radius which were at or below the limit of detection (LOD, range of 0.0026 to 0.0043  $\mu\text{g/L}$ ). It should also be noted that on-site groundwater does not flow toward off-site private water wells or public water wells as it is contained by the actively pumping groundwater interceptor well system on-site at Chambers Works.

**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 EPA'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.
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 and by laboratory analysis of an expanded target list of compounds. In addition, if PFOA concentrations in those wells measure 0.40 parts per billion (ppb) (or 0.40 µg/L) or greater, which is the provisional health advisory established by the U. S. Environmental Protection Agency (EPA) Office of Water on January 8, 2009, installation of residential granular activated carbon (GAC) treatment (or an alternative treatment approved by EPA and NJDEP) will be offered at no cost to the property owner. On May 19, 2016, EPA developed a drinking water health advisory for PFOA, and perfluorooctanesulfonic acid (PFOS) or PFOA plus PFOS of 0.07 ppb. GAC treatment (or an alternative treatment approved by EPA and NJDEP) will be offered at no cost to property owners where PFOA, PFOS, or PFOA plus PFOS concentrations in their drinking water wells are above the new criteria. In addition, GAC treatment (or an alternative treatment approved by EPA and NJDEP) will be offered at no cost to property owners where perfluorononanoic acid (PFNA) concentrations in their drinking water wells are above the New Jersey Department of Environmental Protection (NJDEP) interim specific groundwater quality criterion for PFNA of 0.01 ppb (0.01 µg/L).
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, if qualified. A call center will be established and is expected to provide supplemental information. Analytical results will supplement understanding of any PFAS contaminant migration outside of the Chambers Works.
4. Define the Boundaries of the Study – Chemours has agreed to perform sampling and resampling of residential drinking water sources within approximately two miles of the Chambers Works. Drinking water samples will be analyzed by method 537 Modified to reach a reporting limit of 2 ng/L, as communicated to Chemours by EPA Region 2 and if achievable for each of the following target compounds:

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	PFTA	Perfluorotetradecanoic acid
72629-94-8	PFTTrDA	Perfluorotridecanoic acid
2058-94-8	PFUnA	Perfluoroundecanoic acid
—	NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid
—	NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid

5. Develop the Analytic Approach – 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.
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/537 Modified  
 Concentration Level: Low

<b>Data Quality Indicator (DQI)</b>	<b>QC Sample or Measurement Performance Activity</b>	<b>Measurement Performance Criteria</b>
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	Analyte-specific (see Table 1)
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Samples	Analyte-specific (see Table 1)
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	Analyte-specific (see Table 1)
Analytical Accuracy/Bias (matrix interference)	Isotope Dilution Analytes	Analyte-specific (see Table 2)
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations $\geq$ 1/2 RL
Sensitivity	RL verification sample (spiked at RL)	Recovery within $\pm$ 50% of RL
Completeness	Percentage of valid data compared to total data collected	100%*

\*100% of the residential well locations identified under this program will be contacted and will be sampled if the residence responds and 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.

<b>Data type</b>	<b>Source</b>	<b>Data uses relative to current project</b>	<b>Factors affecting the reliability of data and limitations on data use</b>
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)**

Following is a project schedule showing specific tasks, the person or group responsible for their execution, and planned start and end dates.

<b>Activity</b>	<b>Responsible party</b>	<b>Planned start date</b>	<b>Planned completion date</b>	<b>Deliverable(s)</b>	<b>Deliverable due date</b>
Mailing of Residential Letters	AECOM	Upon EPA approval of this QAPP (mid-April 2016)	Within 3 days of QAPP approval date (mid-April 2016)	Letters mailed to residents	Within 3 days of QAPP approval date (mid-April 2016)
Call Center Activation and Sampling Qualification Determination	AECOM, EPA if residents have questions and the call center refers them to the EPA contact	Mid-April 2016	October 2016	Summary spreadsheet of call center contacts; complete spreadsheet will be provided in the final project report	May 2016 (monthly status reports) and October 2016 (Final Project Report)
Drinking Water Well Sampling	AECOM	Mid-April 2016	October 2016	Field notes	October 2016 (Final Project Report)
Analysis	TestAmerica Sacramento	May 2016	28 days from sample receipt	Report of Analyses/Data package	October 2016 (Final Project Report)
Validation - DVM	AECOM	May 2016	October 2016	Validation Summary report	October 2016 (Final Project Report)
Validation – third party (10%, see worksheet 36)	Environmental Standards	July 2016	September 2016	Validation Summary report	October 2016 (Final Project Report)
Usability assessment	AECOM	Ongoing with receipt of first data report	October 2016	Usability assessment summary report	October 2016 (Final Project Report)
Results to EPA and NJDEP	AECOM	May 2016	October 2016	Spreadsheet	Monthly Status Reports
Result letter to Resident	AECOM	May 2016	October 2016	Result Letter	Within 1 week of data finalization

Activity	Responsible party	Planned start date	Planned completion date	Deliverable(s)	Deliverable due date
Monthly Status Report	AECOM	May 2016	October 2016	Monthly Status Report	Monthly from May – October 2016
Following up with returned letters and residents that are non-responsive to the sampling offer*	AECOM/EPA	May 2016	October 2016	Door to door contacts and follow up letters	As needed
Final Project Report generation*	AECOM	May 2016	October 2016	Final Project Report	October 2016

Note all of the activities listed above, except the first and last, will be sequential and ongoing starting with the first call from a resident that is received at the call center.

**\* Indicates an activity that will be further defined during discussions with EPA and other stakeholders, as necessary.**

**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/537 Modified

Concentration level (if applicable): Low

Analyte	Project Action Limit (PAL)	PAL Reference	Project Quantitation Limit Goal	Laboratory-specific quantitation limit <sup>4</sup>	Laboratory-specific detection limit <sup>5</sup>
PFOA	70 ng/L	EPA 822-R-16-005 <sup>1</sup>	2 ng/L	2 – ng/L	0.748 ng/L
PFOS	70 ng/L	EPA 822-R-16-004 <sup>2</sup>	2 ng/L	2 ng/L	1.276 ng/L
PFNA	10 ng/L	NJDEP <sup>3</sup>	2 ng/L	2 ng/L	0.654 ng/L
PFASs (other compounds – see worksheet #11)	2 ng/L	Communication, EPA Region 2 to Chemours <sup>4</sup>	2 ng/L	2 – 20 ng/L	Varies (see table 3)

<sup>1</sup> EPA 822-R-16-005, Drinking Water Health Advisory for Perfluorooctanoic Acid (PFOA) – May 2016

<sup>2</sup> EPA 822-R-16-004, Drinking Water Health Advisory for Perfluorooctane Sulfonate (PFOS) – May 2016

<sup>3</sup> New Jersey Department of Environmental Protection (NJDEP) interim specific groundwater quality criterion for Perfluorononanoic acid (PFNA)

<sup>4</sup> 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 EPA Region 2 to Chemours.

Regarding the quantitation limit: The reporting limit (RL) of 2 ng/L for most compounds (see Table 3) was assigned by TestAmerica Sacramento based on an extraction factor and the lab's lower calibration limit, after evaluating the results of method detection limit (MDL) studies performed using seven replicates spiked at 2 ng/L. The lab regularly analyzes MDL/RL verifications spiked at 1 and 2 ng/L, respectively.

<sup>4</sup> Define quantitation limit terminology used by the project/laboratory

<sup>5</sup> Define detection limit terminology used by the project/laboratory



**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

In 2009, E. I. du Pont de Nemours and Company (DuPont) worked in collaboration with the New Jersey Department of Environmental Protection (NJDEP) to conduct a Perfluorooctanoic Acid (PFOA) Private Drinking Water Well Survey and Sampling Program within a two-mile radius surrounding the Chambers Works facility, located in Deepwater, New Jersey. DuPont sampled private drinking water wells to determine if PFOA concentrations in those wells measured 0.40 ppb or greater, which is the provisional health advisory established by the U. S. Environmental Protection Agency (EPA) Office of Water on January 8, 2009.

A total of 111 drinking water wells sampled during the DuPont 2009 drinking water well survey and sampling program may be qualified for resampling under the Chemours Residential Drinking Water Well Resampling Program<sup>6</sup>. Additional drinking water wells within a two-mile radius surrounding the Chambers Works facility, which were not sampled during previous events, will be identified for possible sampling. A list of these drinking water wells/residences and contact information for the residents is not included in this QAPP for privacy reasons. Prior to sending sampling or resampling offer letters to the private residents, a database of potentially qualified residents will be generated and a call center will be established. After residents contact the call center, information gathered by the call center will be used to determine if these wells are qualified for sampling or resampling, and if so, coordinate and schedule the sampling activities. Residents will be contacted and sampling or resampling may be offered if the wells are used as drinking water sources. (See Worksheet #6 for a description of information to be included in the call center master spreadsheet and in the summary spreadsheets to be included in the monthly status reports to be submitted to EPA and NJDEP.)

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<sup>6</sup> A total of 113 samples was collected from the 111 drinking water wells. One well had a sample and a duplicate collected, and another well was sampled and then resampled due to blank contamination in the initial sample.

**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	Matrix <sup>7</sup>	Type	Analyte/ Analytical Group	Sampling SOP	Comments
CWK-D-address	DW	Field sample	PFASs	QAPP (see worksheet 21)	New locations.
CWK-D-address- date sampled	DW	Field sample	PFASs	"	Repeat locations.
CWK-D-address-D	DW	Field duplicate	PFASs	"	All locations
FB-date sampled	W	Field blank	PFASs	"	Related to all locations collected on a given day

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<sup>7</sup>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):  
TestAmerica  
880 Riverside Parkway  
West Sacramento, CA 95605  
Jill Kellmann  
Jill.Kellmann@testamericainc.com  
Tel: 916.373.5600  
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	537 Mod. WS-LC-0025 (Appendix A)	Accreditation for method pending, NJDEP permit expires 6/30/2016, renewal is in progress.	2 x 250 mL HDPE Bottles	4 ± 2°C	7 days	40 days	28 days	30 days

The 7-day and 40-day holding times given above are based on the general EPA convention for the holding time of extractable organic compounds in water (see EPA SW-846 Chapter 4 at [https://www.epa.gov/sites/production/files/2015-10/documents/chap4\\_0.pdf](https://www.epa.gov/sites/production/files/2015-10/documents/chap4_0.pdf)).

**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.

Matrix	Analyte/Analytical Group	Field Samples	Field Duplicates	Matrix Spikes	Matrix Spike Duplicates	Field Blanks	Equipment Blanks	Trip Blanks	Other	Total # analyses*
Drinking Water	PFASs	240	12	12	12	20	0	0	N/A	296

\* 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. The estimated number of field blank samples is based on performing a bottle-to-bottle transfer of laboratory blank water during each day of sampling.

**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 (prior to any treatment systems).
2. Open the 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.

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.

**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	<sup>‡</sup> Modified for Project? Y/N
WS-LC-0025, Revision 1.7	"Perfluorinated Compounds (PFASs) in Water, Soils, Sediments and Tissue [Method 537 Modified]", effective 3/18/16.	Definitive	Drinking Water/PFASs	na	Y- see below.

‡ A brief summary of project-specific SOP modifications must be provided on this worksheet or referenced.

The laboratory Standard Operating Procedure (SOP) can be found in Appendix A and is described as follows:

Samples and QC are spiked prior to extraction with isotopically (either carbon-13, oxygen-18, or deuterium labeled) analogs for many of the compounds of interest. Following spiking, water samples are extracted using a solid phase extraction (SPE) cartridge. PFASs are eluted from the cartridge with ammonium hydroxide/methanol solution. Soil samples are extracted by shaking, followed by sonication with a potassium hydroxide/methanol solution. Following centrifugation and filtration, the extracts are cleaned using SPE cartridges.

Extracts are analyzed by LCMS on an instrument with an electrospray interface. The separation is performed using reverse phase chromatography, and the mass spectrometer operated in the negative ion mode.

Quantitation is performed using isotope dilution methodology (i.e., compounds are quantitated using the spiked isotopically labeled analog as a reference). Compounds that do not have a labeled analog are quantitated by the internal standard method using a closely related labeled analog. This technique allows correction for analytical bias encountered when analyzing more chemically complex environmental samples, as the isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related interferences to the same extent as the compounds of concern.

Recovery of the isotopically labeled compounds is calculated using external standard quantitation. Recovery of these compounds is reported with the sample results.

**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 target analytes, lowest concentration standard at or below the reporting limit	0.5 – 400 ng/mL (upper levels may not be included in order to improve calibration fit)	Initial calibration prior to sample analysis	One of the two options below: <u>Option 1:</u> RSD for each analyte calc'd by isotope dilution (ID) $\leq$ 35%, each analyte calc'd by internal standard (IS) & the labeled compounds (IDA) $\leq$ 50%. <u>Option 2:</u> linear least squares regression for each analyte: $r^2 \geq 0.99$ , intercept $< \frac{1}{2}$ reporting limit	Evaluate standards, chromatography, and mass spectrometer response. If problem found with above, correct as appropriate, then repeat initial calibration.	Lab Manager / Analyst <sup>b</sup>	WS-LC-0025



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 calib curve.	Once per initial calibration, following initial calibration.	%D for each analyte calc'd by ID $\leq$ 40%, each analyte calc'd by (IS) $\leq$ 50% & the labeled compounds (IDA) $\leq$ 50%.	Evaluate data. If problem (e.g., concentrated standard, plugged transfer line) found, correct, then repeat second source verification. If it still fails, then repeat initial calibration.	Lab Manager / Analyst <sup>b</sup>	WS-LC-0025
LC/MS/MS	Daily calibration verification	Per SOP, one of the mid-range calibration standards. Levels are varied through the analytical sequence.	Before sample analysis, after every 10 field samples, and at the end of the sequence.	%D for each analyte calc'd by ID $\leq$ 40%, each analyte calc'd by (IS) $\leq$ 50% & the labeled compounds (IDA) $\leq$ 50%.	Evaluate failure and impact on samples. If samples non-detect for analytes which have a high bias, report non-detect results with case narrative comment. Otherwise, perform any indicated maintenance, recalibrate, and reanalyze all affected samples since the last acceptable CCV.	Lab Manager / Analyst <sup>b</sup>	WS-LC-0025

<sup>b</sup> 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.

<b>Instrument / Equipment</b>	<b>Maintenance Activity</b>	<b>Testing Activity</b>	<b>Inspection Activity</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>	<b>Title/position responsible for corrective action</b>	<b>Reference</b>
LC/MS/MS	Replace columns as needed, check eluent reservoirs	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	TestAmerica Chemist	SOP WS-LC-0025

**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: 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, tbd	CRG Chain of Custody SOP (Appendix D)
Chain-of-custody form completion	AECOM, tbd	CRG Chain of Custody SOP (Appendix D)
Packaging	AECOM, tbd	Shipping SOP (Appendix E)
Shipping coordination	AECOM, tbd	Shipping SOP (Appendix E)
Sample receipt, inspection, & log-in	TestAmerica, Sample Receiving Technician	WS-QA-0003 (Appendix F)
Sample custody and storage	TestAmerica, Sample Receiving Technician	WS-QA-0003 (Appendix F)
Sample disposal	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/SOP: 537 Mod/SOP WS-LC-0025, Revision 1.7

<b>QC Sample</b>	<b>Number/Frequency</b>	<b>Method/SOP Acceptance Criteria</b>	<b>Corrective Action</b>	<b>Title/position of person responsible for corrective action</b>	<b>Project-Specific MPC</b>
Method Blank	1 per prep. batch of up to 20 samples.	No analytes detected > ½ reporting limit (RL), 1/10 the amount measured in any sample, or 1/10 the regulatory limit, whichever is greater.	Reprep and reanalyze the method blank and all samples processed with the contaminated blank. If problem persists, call PM.	Lab Manager / Analyst	All analytes in the method blank must be less than ½ the RL or 1/5 of the PAL's on WS#15, whichever is greater
LCS containing all analytes	One per preparatory batch of up to 20 samples.	Laboratory historically derived control limits.	Reanalyze LCS once. If acceptable, report. 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	Lab Manager / Analyst	All analytes are evaluated against the criteria in Table 1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	Project-Specific MPC
			and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.		
Internal Standards (Isotope Dilution Analytes, spiked prior to extraction)	Every sample, spiked sample, standard, and method blank	% recovery for each IS in the original sample (prior to dilutions) must be within 25-150%	Reanalyze once. Assess matrix, dilute and/or re-extract as needed. Evaluate impact on data.	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)	laboratory statistically derived control limits for LCS, RPD $\leq$ 30%.	Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair if laboratory error is indicated.	Lab Manager / Analyst	All analytes are evaluated against the criteria in Table 1.

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

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.

<b>Sample Collection and Field Records</b>			
<b>Record</b>	<b>Generation</b>	<b>Verification</b>	<b>Storage location/archival</b>
Field logbook or data collection sheets (Appendix C)	Field Task Leader (tbd)	Project Manager (Mark Houlday)	Project File
Chain-of-Custody Forms	Field Task Leader (tbd)	Project Manager (Mark Houlday)	Project File
Air Bills	Field Task Leader (tbd)	Project Manager (Mark Houlday)	Project File
Deviations	Field Task Leader (tbd)	Project Manager (Mark Houlday)	Project File
Corrective Action Reports	Field Task Leader (tbd)	Project Manager (Mark Houlday)	Project File
Correspondence	Project Technical Adviser (Kathy Davis)	Project Manager (Mark Houlday)	Project File
Resident Database/Spreadsheet	Project Technical Adviser (Kathy Davis)	Project Manager (Mark Houlday)	Project File
Monthly Status Reports	Project Technical Adviser (Kathy Davis)	Project Manager (Mark Houlday)	Project File

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

Project Assessments			
Data usability assessment report	Project Chemist (Mike Aucoin)	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 or 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)
<b>Planning Documents/Records</b>			
1	Approved QAPP	X	
2	Contract	X	
3	Field SOPs	X	
4	Laboratory SOPs	X	
<b>Field Records</b>			
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
<b>Analytical Data Package</b>			
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 preservation 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

<b>Records Reviewed</b>	<b>Requirement Documents</b>	<b>Process Description</b>	<b>Responsible Person, Organization</b>
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 disk 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
- US EPA 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.

In addition, laboratory results greater than the MDL but less than the RL are qualified J and should be considered to be estimated values.

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 EPA 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:

<b>Step 1</b>	<b>Review the project's objectives and sampling design</b>  <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>
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<b>Step 2</b>	<b>Review the data verification and data validation outputs</b>  <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>
<b>Step 3</b>	<b>Document data usability and draw conclusions</b>  <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 %
N-ethyl perfluorooctane sulfonamidoacetic acid	2991-50-6	NEtFOSAA	60	140	30
N-methyl perfluorooctane sulfonamidoacetic acid	2355-31-9	NMeFOSAA	60	140	30
Perfluorobutanesulfonic acid (PFBS)	375-73-5	PFBS	55	147	30
Perfluorodecanoic acid (PFDA)	335-76-2	PFDA	66	141	30
Perfluorododecanoic acid (PFDoA)	307-55-1	PFDoA	71	139	30
Perfluoroheptanoic acid (PFHpA)	375-85-9	PFHpA	63	135	30
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	PFHxS	58	138	30
Perfluorohexanoic acid (PFHxA)	307-24-4	PFHxA	70	136	30
Perfluorononanoic acid (PFNA)	375-95-1	PFNA	71	140	30
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	PFOS	47	162	30
Perfluorooctanoic acid (PFOA)	335-67-1	PFOA	63	141	30
Perfluorotetradecanoic acid (PFTeA)	376-06-7	PFTeA	47	130	30
Perfluorotridecanoic Acid (PFTrIA)	72629-94-8	PFTrDA	51	139	30
Perfluoroundecanoic acid (PFUnA)	2058-94-8	PFUnA	68	139	30

**Table 2**  
**Laboratory IDA Recovery Limits**  
**PFAS Drinking Water Sampling Program QAPP**

Analyte	Rec. Low %	Rec. High %
13C2 PFDA	25	150
13C2 PFDoA	25	150
13C2 PFHxA	25	150
13C2 PFUnA	25	150
13C2-PFTeDA	25	150
13C4 PFOA	25	150
13C4 PFOS	25	150
13C4-PFHpA	25	150
13C5 PFNA	25	150
18O2 PFHxS	25	150
d3-NMeFOSAA	25	150
d5-NEtFOSAA	25	150

**Table 3**  
**Laboratory Reporting Limits**  
**PFAS Drinking Water Sampling Program QAPP**

<b>Analyte</b>	<b>Method Abbreviation</b>	<b>CAS Num</b>	<b>MDL ng/L</b>	<b>RL ng/L</b>
N-ethyl perfluorooctane sulfonamidoacetic acid	NEtFOSAA	2991-50-6	5.02	20
N-methyl perfluorooctane sulfonamidoacetic acid	NMeFOSAA	2355-31-9	5.64	20
Perfluorobutanesulfonic acid (PFBS)	PFBS	375-73-5	0.918	2
Perfluorodecanoic acid (PFDA)	PFDA	335-76-2	0.44	2
Perfluorododecanoic acid (PFDoA)	PFDoA	307-55-1	0.584	2
Perfluoroheptanoic acid (PFHpA)	PFHpA	375-85-9	0.802	2
Perfluorohexanesulfonic acid (PFHxS)	PFHxS	355-46-4	0.87	2
Perfluorohexanoic acid (PFHxA)	PFHxA	307-24-4	0.786	2
Perfluorononanoic acid (PFNA)	PFNA	375-95-1	0.654	2
Perfluorooctanesulfonic acid (PFOS)	PFOS	1763-23-1	1.276	2
Perfluorooctanoic acid (PFOA)	PFOA	335-67-1	0.748	2
Perfluorotetradecanoic acid (PFTeA)	PFTA	376-06-7	0.199	2
Perfluorotridecanoic Acid (PFTrIA)	PFTrDA	72629-94-8	0.551	2
Perfluoroundecanoic acid (PFUnA)	PFUnA	2058-94-8	0.748	2

## Figures

Figure 1

**AUDIT CHECKLIST**  
**PFAS Drinking Water Sampling Program QAPP**

PROJECT: \_\_\_\_\_ PROJECT MANAGER: \_\_\_\_\_

SITE LOCATION: \_\_\_\_\_

AUDITOR: \_\_\_\_\_ DATE: \_\_\_\_\_

Question	Yes	No	Comment/Documentation
<b>Field:</b>			
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
<b>Field:</b>			
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: \_\_\_\_\_




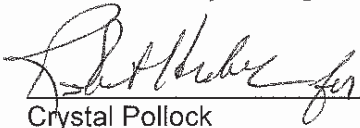
## Appendices



## **Appendix A**

**SOP WS-LC-0025, Revision  
1.7, Effective  
3/18/2016, "Perfluorinated  
Compounds (PFCs) in  
Water, Soils, Sediments and  
Tissue [Method 537  
Modified]"**

**Title: Perfluorinated Compounds (PFCs) in Water, Soils, Sediments and Tissue**  
**[Method 537 Modified]**

Approvals (Signature/Date):			
	5/9/16		3/9/16
Robert Hrabak Technical Manager	Date	Joe Schairer Health & Safety Manager / Coordinator	Date
	3/16/16		3/9/16
Lisa Stafford Quality Assurance Manager	Date	Crystal Pollock Laboratory Manager	Date

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## 1. SCOPE AND APPLICATION

- 1.1. This procedure describes the analysis of water, soil, sediment and tissue samples for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS).

Compound Name	Abbreviation	CAS #
<b>Perfluoroalkylcarboxylic acids (PFCAs)</b>		
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
Perfluoro-n-hexanoic acid	PFHxA	307-24-4
Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PFNA	375-95-1
Perfluoro-n-decanoic acid	PFDA	335-76-2
Perfluoro-n-undecanoic acid	PFUdA	2058-94-8
Perfluoro-n-dodecanoic acid	PFDoA	307-55-1
Perfluoro-n-tridecanoic acid	PFTTrDA	72629-94-8
Perfluoro-n-tetradecanoic acid	PFTeDA	376-06-7
Perfluoro-n-hexadecanoic acid	PFHxDA	67905-19-5
Perfluoro-n-octadecanoic acid	PFODA	16517-11-6
<b>Perfluorinated sulfonic acids (PFSAs)</b>		
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5
Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4
Perfluoro-1-heptanesulfonic acid	PFHpS	375-92-8
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1
Perfluoro-1-decanesulfonoc acid	PFDS	335-77-3
<b>Perfluorinated sulfonamides (FOSA)</b>		
Perfluoro-1-octanesulfonamide	FOSA	754-91-6
<u>N-ethylperfluoro-1-octanesulfonamide</u>	EtFOSA	4151-50-2
N-methylperfluoro-1-octansulfonamide	MeFOSA	31506-32-8
<b>Perfluorinated sulfonamidoacetic acids (FOSAA)</b>		
N-ethylperfluoro-1-octanesulfonamidoacetic acid	EtFOSAA	2991-50-6
N-methylperfluoro-1-octanesulfonamidoacetic acid	MeFOSAA	2355-31-9
<b>Fluortelomer sulfonates (FTS)</b>		

Sodium 1H,1H,2H,2H-perfluorooctane sulfonate (6:2)	6:2FTS	27619-97-2
Sodium 1H,1H,2H,2H-perfluorodecane sulfonate (8:2)	8:2FTS	39108-34-4

\* Chromatographs poorly and not reproducible, analyte is not analyzed.

Sample results for PFOA may also be reported as APFO, at the request of the client.  
(See Section 12.7)

- 1.2. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	500 mL	2.0 ng/L – 100 ug/L	2 to 400 ug/L
Soil/Sediment/Tissue	5 g	0.2 ug/kg – 20 ug/kg	0.2 to 100 ug/kg

- 1.3. Due to poor chromatographic peak shape which degraded with repeated injections for Perfluoro-1-octanesulfonamidoamide (FOSSA), this analyte is no longer included in the method.
- 1.4. 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.

## 2. SUMMARY OF METHOD

- 2.1. Water samples are extracted using a solid phase extraction (SPE) cartridge, unless EtFOSA and MeFOSA are requested. PFCs are eluted from the cartridge with ammonium hydroxide/methanol solution.
- 2.2. Soil samples are extracted with KOH/methanol solution using an orbital shaker for 3 hours followed by sonication for 12 hours. The mixture is centrifuged and the solvent filtered.
- 2.2.1. Optional cleanups may include sample freezing and/or cleanup by SPE cartridge, unless EtFOSE and MeFOSA are requested.
- 2.3. The final 80:20 methanol: water extracts are analyzed by LC/MS/MS. PFCs are separated from other components on a C18 column with a solvent gradient program using 20 mM Ammonium Acetate/water and methanol. The mass spectrometer detector is operated in the electrospray (ESI) negative ion mode for the analysis of PFC.
- 2.4. Isotope dilution technique is employed with this method for most compounds of interest. The internal standards (isotope dilution analytes or

IDA) consist of carbon-13 labeled analogs, oxygen-18 labeled analogs or deuterated analogs of the compounds of interest, and they are spiked into the samples at the time of extraction. This technique allows correction for analytical bias encountered when analyzing more chemically complex environmental samples, because the isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related interferences to the same extent as the compounds of concern. Compounds that do not have a labeled analog are quantitated by internal standard method using a closely related labeled analog.

- 2.5. Quantitation by the external standard method is employed for the IDA analytes and assumes a proportional relationship between the initial calibration and the analyte in the extract. The ratio of the peak response to mass or concentration injected is used to prepare a calibration curve. Peak response is measured as the area of the peak. Isotope dilution technique is employed with this method for most compounds of interest. The internal standards consist of carbon-13 labeled analogs or oxygen-18 labeled analogs of the compounds of interest, and they are spiked to the samples at the time of extraction. This technique allows correction for analytical bias encountered when analyzing more chemically complex environmental samples, because the isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related interferences to the same extent as the compounds of concern. Compounds that do not have a labeled analog are quantitated by internal standard method using a closely related labeled analog.

### 3. DEFINITIONS

- 3.1. PFCAs: Perfluorocarboxylic acids
- 3.2. PFSAs: Perfluorinated sulfonates
- 3.3. FOSA: Perfluorinated sulfonamides
- 3.4. PFOA: Perfluorooctanoic acid (may also be written PHOA).
- 3.5. APFO: Ammonium perfluorooctanoate
- 3.6. PFOS: Perfluorooctane sulfonate (may also be written PHOS)
- 3.7. MPFOA: Perfluoro-n-[1,2,3,4-<sup>13</sup>C<sub>4</sub>]octanoic acid. Carbon-13 labeled PFOA
- 3.8. MPFOS: Perfluoro-1-[1,2,3,4-<sup>13</sup>C<sub>4</sub>]octanesulfonate. Carbon-13 labeled PFOS
- 3.9. PTFE: Polytetrafluoroethylene (e.g., Teflon®)
- 3.10. SPE: Solid phase extraction.

- 3.11. PP: Polypropylene
- 3.12. PE: Polyethylene
- 3.13. HDPE: High density polyethylene
- 3.14. Further definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Assurance Manual (QAM).

#### **4. INTERFERENCES**

- 4.1. PFCs have been used in a wide variety of manufacturing processes, and laboratory supplies should be considered potentially contaminated until they have been tested and shown to be otherwise. The materials and supplies used during the method validation process have been tested and shown to be clean. These items are listed below in Section 6.
- 4.2. To avoid contamination of samples, standards are prepared in a ventilation hood in an area separate from where samples are extracted.
- 4.3. PTFE products can be a source of PFOA contamination. The use of PTFE in the procedure should be avoided or at least thoroughly tested before use. Polypropylene (PP) products may be used in place of PTFE products to minimize PFOA contamination.
  - 4.3.1. Standards and samples are injected from polypropylene autosampler vials with polypropylene screw caps once. Multiple injections may be performed on Primers when conditioning the instrument for analysis.
  - 4.3.2. Random evaporation losses have been observed with the polypropylene caps causing high IDA recovery after the vial was punctured and sample re-injected. For this reason, it is best to inject standards and samples once in the analytical sequence.
  - 4.3.3. Teflon-lined screw caps have detected PFCs at low concentrations. Repeated injection from the same teflon-lined screw cap have detected PFNA at increasing concentration as each repeated injection was performed, therefore, it is best to use polypropylene screw caps.
- 4.4. Volumetric glassware and syringes are difficult to clean after being used for solutions containing high levels of PFOA. These items should be labeled for use only with similarly concentrated solutions or verified clean prior to re-use. To the extent possible, disposable labware is used.

- 4.5. Commercial sources of PFOS may produce several peaks in the PFOS chromatogram. These adjacent peaks are either completely resolved or not resolved but with a profound deflection that can be resolved during peak integration. The later of the peaks matches the retention time of the single labeled PFOS peak. Earlier peaks are branched isomers of PFOS, rather than a result of peak splitting. The earlier peak is included during peak integration.
- 4.6. The phenomenon of the linear and branched isomers of PFOS exists for other PFAS, such as PFHxS and PFBS. Thus, in an attempt to reduce PFOS bias, it is required that  $m/z$  449>80 transition be used as the quantitation transition.
- 4.7. Both branched and linear PFAAs can potentially be found in the environment. For the compounds that give rise to more than one peak, all the chromatographic peaks observed in the standard must be integrated and the areas included.

## 5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported **immediately** to a supervisor, the EH&S Staff, or a senior manager.

### 5.1. Specific Safety Concerns

- 5.1.1. Preliminary toxicity studies indicate that PFCs could have significant toxic effects. In the interest of keeping exposure levels as low as reasonably achievable, PFCs must be handled in the laboratory as hazardous and toxic chemicals.
- 5.1.2. Exercise caution when using syringes with attached filter disc assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.
- 5.1.3. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.
- 5.1.4. Eye protection that satisfies ANSI Z87.1 (as per the TestAmerica Corporate Safety Manual), laboratory coat, and nitrile gloves must be worn

while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

- 5.1.5. Perfluorocarboxylic acids are acids and are not compatible with strong bases.
- 5.1.6. The use of vacuum systems presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.
- 5.1.7. Glass containers are not to be used for “tumbling” soil samples.

## 5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials action. 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 <sup>(1)</sup>	Hazards	Exposure Limit <sup>(2)</sup>	Signs and Symptoms of Exposure
Acetic Acid (3-2-1)	Corrosive Poison Flammable	10 ppm-TWA 15 ppm-STEL	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage to the upper respiratory tract. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent damage, including blindness. Brief exposure to 5000 PPM can be fatal.



Material <sup>(1)</sup>	Hazards	Exposure Limit <sup>(2)</sup>	Signs and Symptoms of Exposure
Hexane (2-3-0)	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Can cause pain and severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause deep ulcerations to skin, permanent eye damage, circulatory failure and swallowing may be fatal.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Potassium Hydroxide	Corrosive Poison		Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.
Sodium Hydroxide	Corrosive Poison	2 mg/cm <sup>3</sup> (Ceiling)	Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.
<p>(1) Always add acid to water to prevent violent reactions.            (2) Exposure limit refers to the OSHA regulatory exposure limit.</p>			

## 6. EQUIPMENT AND SUPPLIES

- 6.1. 8 mL test tubes, screw thread, with caps.
- 6.2. 15-mL polypropylene test tubes with polypropylene screw caps.
- 6.3. 50 mL graduated plastic centrifuge tubes.
- 6.4. 125 mL PPE wide-mouth bottles.
- 6.5. 16 oz or 500 mL PPE bottles with PPE screw caps.

- 6.6. Analytical balance capable of accurately weighing to the nearest 0.0001g, and checked for accuracy each day it is used in accordance with WS-QA-0041.
- 6.7. Syringe filter, Millipore Millex-HV 0.45 um, or equivalent. Do not use PTFE type filters.
- 6.8. 300- $\mu$ L autosampler vials, polypropylene, with polypropylene screw caps, Waters PN 1860004112, or equivalent.
- 6.9. SPE columns;
  - 6.9.1. Phenomenex Strata SPE C18, 6 mL, 500 mg, part number 8B-S002-HCH, Waters SepPak C18, 1 to 10g, or equivalent.
  - 6.9.2. Waters Oasis WAX 150 mg/6 cc (PN 186002493) for the cleanup of solids.
  - 6.9.3. Waters Oasis WAX 500 mg/6 cc (PN 186004647) for extraction of PFC from aqueous sample.
- 6.10. PFC Isolator column, Waters PN 186004476, plumbed between the UPLC pumps and autosampler valve to minimize PFC background from the UPLC solvent lines and filters.
- 6.11. Granulated carbon.
- 6.12. Vacuum manifold for Solid Phase Extraction (SPE).
- 6.13. Miscellaneous laboratory apparatus (beakers, test tubes, volumetric flasks, pipettes, etc.). These should be disposable where possible, or marked and segregated for high-level versus low-level use.
- 6.14. WATERS Acquity UPLC system connected to Triple Quad MS such as Waters Micromass Quattro Premier XE. The system utilizes Chrom Peak Review, version 2.1 or equivalent.
- 6.15. Acquity UPLC BEH C18 1.7 um, 3.0 mm x 150 mm, Part No. 186004690, or Phenomenex Gemini-NX C18 3 um, 3.0 mm x 100 mm, Part No. 00D-4453-Y0, or equivalent.
- 6.16. Bottle rotating apparatus for soil extractions.
- 6.17. Glass fiber filter, Whatman GF/F, catalog number 1825 090 or equivalent.
- 6.18. Preventive and routine maintenance is described in below

<b>HPLC/MS/MS Preventative Maintenance</b>
<p><b><u>As Needed:</u></b></p> <p>Change pump seals.            Change in-line filters in autosampler (HPLC).            Check/replace in-line frit if excessive pressure or poor performance.            Replace column if no change following in-line frit change.            Clean corona needle.            Replace sample inlet tube in APCI (10.1 cm).            Replace fused silica tube in ESI interface.            Clean lenses.            Clean skimmer.            Ballast rough pump 30 minutes.</p>
<p><b><u>Daily (When in use)</u></b></p> <p>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><b><u>Semi-Annually</u></b></p> <p>Replace rough-pump oil (4-6 months).            Replace oil mist and odor elements.            Replace activated alumina filter if applicable.</p>
<p><b><u>Annually</u></b></p> <p>Vacuum system components including fans and fan covers.            Clean/replace fan filters, if applicable.</p>

## 7. REAGENTS AND STANDARDS

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

7.1.1. Acetic Acid, glacial

7.1.2. Ammonium acetate (20 mM in water)

7.1.3. Ammonium Hydroxide (NH<sub>4</sub>OH), 0.3% in methanol

- 7.1.4. Hexane
  - 7.1.5. Hydrochloric Acid (HCl), 2.0 M solution in water
  - 7.1.6. Methanol
  - 7.1.7. Potassium Hydroxide (KOH), 0.4% in methanol
  - 7.1.8. Ottawa Sand
  - 7.1.9. Sodium Hydroxide (NaOH), 0.1N, in water
  - 7.1.10. Water, Nanopure or Millipore, must be free of interference and target analytes
- 7.2. Standards
- 7.2.1. PFCs are purchased as a high purity solids (96% or greater) or as certified concentration in solution. Standard materials are verified compared to a second source material at the time of initial calibration. The solid stock material is stored at room temperature or as specified by manufacturer or vendor.
  - 7.2.2. If solid material is used for preparing a standard, stock standard solutions are prepared from the solids and are stored at  $4 \pm 2^{\circ}\text{C}$ . Stock standard solutions should be brought to room temperature before using. Standards are monitored for signs of degradation or evaporation. Standard solutions must be replaced at least annually from the date of preparation.
  - 7.2.3. PFBS, PFHxS, PFHpS, PFOS, PFDS, MPFOS, and many other PFASs are not available as the acids, but rather as their corresponding salts, such as sodium or potassium. The standards are prepared and corrected for their salt content according to the equation below.

$$Mass_{acid} = Measured\ Mass_{salt} \times MW_{acid} / MW_{salt}$$

Where:  $MW_{acid}$  is the molecular weight of PFAA

$MW_{salt}$  is the molecular weight of the purchased salt.

*For example, the molecular weight of PFOS is 500.1295 and the molecular weight of NaPFOS is 523.1193. Therefore, the amount of NaPFOS used must be adjusted by a factor of 1.046*

## 7.3. Calibration Standards

The calibration stock solution is prepared by diluting the appropriate amounts of PFOA and PFOS stock solutions in 80% methanol/water. The calibration stock solution is diluted with methanol to produce initial calibration standards. These are the normal calibration levels used. A different range can be used if needed to achieve lower reporting limits or a higher linear range.

## 7.4. Initial Calibration (ICAL) Levels (ng/mL)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
<b>Perfluoroalkylcarboxylic acids (PFCAs)</b>							
PFBA	0.5	1.0	5.0	20	50	200	400
PFPeA	0.5	1.0	5.0	20	50	200	400
PFHxA	0.5	1.0	5.0	20	50	200	400
PFHpA	0.5	1.0	5.0	20	50	200	400
PFOA	0.5	1.0	5.0	20	50	200	400
PFNA	0.5	1.0	5.0	20	50	200	400
PFDA	0.5	1.0	5.0	20	50	200	400
PFUdA	0.5	1.0	5.0	20	50	200	400
PFDoA	0.5	1.0	5.0	20	50	200	400
PFTTrDA	0.5	1.0	5.0	20	50	200	400
PFTeDA	0.5	1.0	5.0	20	50	200	400
PFHxDA	0.5	1.0	5.0	20	50	200	400
PFODA	0.5	1.0	5.0	20	50	200	400
<b>Perfluorinated sulfonic acids (PFSAs)</b>							
PFBS	0.5	1.0	5.0	20	50	200	400
PFHxS	0.5	1.0	5.0	20	50	200	400
PFHpS	0.5	1.0	5.0	20	50	200	400
PFOS	0.5	1.0	5.0	20	50	200	400
PFDS	0.5	1.0	5.0	20	50	200	400
<b>Perfluorinated sulfonamides (FOSA)</b>							
FOSA	0.5	1.0	5.0	20	50	200	400
EtFOSA	0.5	1.0	5.0	20	50	200	400
MeFOSA	0.5	1.0	5.0	20	50	200	400
<b>Perfluorinated sulfonamidoacetic acids (FOSAA)</b>							
EtFOSAA	0.5	1.0	5.0	20	50	200	400
MeFOSAA	0.5	1.0	5.0	20	50	200	400
<b>Fluortelomer sulfonates (FTS)</b>							
6:2 FTS	0.5	1.0	5.0	20	50	200	400
8:2 FTS	0.5	1.0	5.0	20	50	200	400
<b>Labeled Isotope Dilution Analytes (IDA)</b>							
MPFBA	50	50	50	50	50	50	50

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
M5PFPeA	50	50	50	50	50	50	50
MPFHxA	50	50	50	50	50	50	50
MPFHpA	50	50	50	50	50	50	50
M4PFOA	50	50	50	50	50	50	50
MPFNA	50	50	50	50	50	50	50
MPFDA	50	50	50	50	50	50	50
MPFUdA	50	50	50	50	50	50	50
MPFDoA	50	50	50	50	50	50	50
MPFHxS	50	50	50	50	50	50	50
MPFOS	50	50	50	50	50	50	50
M8FOSA	50	50	50	50	50	50	50
D5-EtFOSA	50	50	50	50	50	50	50
D3-MeFOSA	50	50	50	50	50	50	50
D5-EtFOSAA	50	50	50	50	50	50	50
D3-MeFOSAA	50	50	50	50	50	50	50
M2-6:2FTS	50	50	50	50	50	50	50
M2-8:2FTS	50	50	50	50	50	50	50

Note: Sample extracts are in 80% MeOH/H<sub>2</sub>O.

FOSAA may be added to the mix and are added at the same concentration as FOSA.

*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.*

#### 7.5. Initial Calibration Verification Standard (ICV).

A second source solution for PFC is purchased from the same vendor; the PFC-MXB contains most of the target analytes in this mixture and is used as an ICV. A few compounds are not available in this mixture, may not be available as another lot, and are not available from another vendor. For these analytes only, a second analyst may prepare a second source standard from the same source as the ICAL to produce an ICV. The recommended concentration of the ICV standard should be in the mid-range of the calibration curve. The concentration may be adjusted if the initial calibration levels are changed or altered. The IS is added at a fixed concentration of 50 ng/mL.

#### 7.6. LCS/Matrix PFC Spike Solution, 1000 ng/mL.

The PFC spike solution is prepared by diluting all PFCs to produce a solution containing PFCs each at 1000 ng/mL in methanol.

#### 7.7. PFC Isotope Dilution Analyte Solution, 1000 ng/mL.

The PFC-IS solution is prepared by diluting all labeled PFCs to produce a solution each at 1000 ng/mL in methanol.

## 8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

- 8.1. Water samples are collected in pre-cleaned 500 mL HDPE containers. Soil samples are collected in pre-cleaned 250 mL HDPE containers. Other containers may also be suitable. Samples are chilled to  $4 \pm 2$  °C for shipment to the laboratory.
- 8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at  $4 \pm 2$  °C. Water samples must be extracted within 7 days of collection. Soil samples must be extracted within 14 days of collection. Tissue samples must be extracted within 1 year of collection if stored at -20°C. Extracts must be refrigerated at  $4 \pm 2$  °C, and analyzed within 40 days from extraction.

*NOTE: There is no prescribed regulatory holding time requirement for PFOA and PFOS. The scientific literature indicates that PFOA and PFOS are highly persistent compounds in the environment. TestAmerica Denver (formerly STL Denver) has conducted stability studies indicating that medium- and low-level standard solutions of PFOA are stable for at least three months in polystyrene, and polypropylene plastics at  $4 \pm 2$  °C. The 7 day / 14 day / and 40 day holding times given above are based on the general EPA convention for the holding time of extractable organic compounds in water and soil.*

## 9. QUALITY CONTROL

- 9.1. Initial Demonstration of Capability (IDOC)  
The initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.
- 9.2. Batches are defined at the sample preparation step. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC program document (WS-PQA-003) for further details of the batch definition.
  - 9.2.1. The quality control batch is a set of up to 20 samples of the same matrix processed using the same procedure and reagents within the same time period. The quality control batch must contain a matrix spike/matrix spike duplicate (MS/MSD), a laboratory control sample (LCS) and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count toward the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD, an LCSD may be substituted if batch precision is required by the program or client. In the event that multiple MS/MSDs are run with a batch due to client requirements, the additional MS/MSDs do not count toward the maximum 20 samples in a batch.

- 9.3. One method blank (MB, laboratory reagent blank) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water. For solid sample, the method blank is an aliquot of Ottawa sand. The method blank is processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above 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. See policy WS-PQA-003 for specific acceptance criteria.
- 9.3.1. If the MB produces a peak within the retention time window of any of the analytes determine the source of the contamination and eliminate the interference before processing samples.
- 9.3.2. The method blank must not contain any analyte at or above the reporting limit, or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.
- 9.3.3. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.
- 9.3.4. Re-extraction and re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.
- 9.3.5. Refer to WS-PQA-003 for further details of the corrective actions.
- 9.3.6. Projects performed under the auspices of the DOD/DOE must meet QSM specific criteria for method blanks. Results are acceptable if the blank contamination is less than  $\frac{1}{2}$  of the reporting limit for each analyte, or less than  $\frac{1}{10}$  of the regulatory limit, or less than  $\frac{1}{10}$  of the sample result for the same analyte, whichever is greater. If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. If contamination remains, the contaminated samples should be re-prepared and reanalyzed with a new MB and batch-specific QC 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 is an aliquot of laboratory matrix (e.g. water for aqueous samples and Ottawa sand for solids) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples.



Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside of the control limits. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See WS-PQA-0003 for specific acceptance criteria. The control limits for the LCS are stored in TALS.

- 9.5. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD pair is aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside of the control limits must be within the control limits in the LCS. Corrective actions must be documented on a nonconformance memo, then implemented when recoveries of any spiked analyte are outside of the control limits provided by the LIMS or by the client.
- 9.6. A duplicate control sample (LCSD or DCS) may be added when insufficient sample volume is provided to process an MS/MSD pair, or is requested by the client. The LCSD is evaluated in the same manner as the LCS. See WS-PQA-003 for specific acceptance criteria.
- 9.7. Initial calibration verification (ICV) – When available, a second source standard is analyzed with the initial calibration curve. The concentration should be at the mid range of the curve.
- Corrective actions for the ICV include:
- Rerun the ICV.
  - Remake or acquire a new ICV.
  - Evaluate the instrument conditions.
  - Evaluate the initial calibration standards.
- 9.8. Isotope Dilution Analytes
- 9.8.1. The IDA solution is added to each field and QC sample at the time of extraction, as described in Section 11. As described in Section 7, this solution consists of isotopically labeled analogs of the analytes of interest.
- 9.8.2. IDA recoveries are flagged if they are outside of the acceptance limits (25–150%). Quantitation by isotope dilution generally precludes any adverse effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-noise ratio is greater than 10:1.

- 9.8.2.1. Evaluate data quality for usability, flag and submit a non-conformance memo for any analytes outside of the recovery criteria, and report if data is deemed not adversely effected.
- 9.8.2.2. Re-extraction of samples should be performed if the signal-to-noise for any IDA is less than 10:1 or if the IDA recoveries fall below 10%.
  - 9.8.2.2.1. Re-extraction may be necessary under other circumstances when data quality has been determined to be adversely affected.

## 10. CALIBRATION

- 10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".
- 10.2. Routine instrument operating conditions are listed in the table in Section 11.6.1.
- 10.3. Instrument Tuning

Instrument tuning is done initially when the method is first developed and thereafter as needed to maintain the sensitivity and selectivity of the method. Tuning is done by infusing each individual compound (native, internal standard and recovery) into the mobile phase using a tee fitting at a point just before the entrance to the electrospray probe. The responses for the parent and daughter ions for each compound are observed and optimized for sensitivity and resolution. Mass assignments are reviewed and calibrated if necessary. The mass assignments must be within  $\pm 0.5$  amu of the values shown in the table in Section 11.5.1.
- 10.4. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include, but are not limited to new columns or pump seals. A new calibration is not required after minor maintenance.
- 10.5. With the exception of the circumstances delineated in policy P-T-001, 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.
- 10.6. A fixed injection volume is used for quantitation purposes and is to be the same for both the sample and standards.

- 10.7. All units used in the calculations must be consistently uniform, such as concentration in ng/mL.
- 10.8. Initial Calibration
- 10.8.1. A number of analytical standards of different analyte concentrations are used to generate the curve. Each standard is injected once to obtain the peak response for each analyte at each concentration. These standards define the working range of the analysis.
- 10.8.1.1. A minimum of five analytical standards is used when using average response factor and or linear calibration fits.
- 10.8.1.2. A minimum of six analytical standards is used for quadratic fit to generate the curve.
- 10.8.2. Calibration is by average response factor, linear fit, or by quadratic fit. Quadratic fit is used for the analyte if the response is non-linear.
- 10.8.2.1. For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated by isotope dilution must be < 35% for the curve to be valid.
- 10.8.2.2. For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated by internal standard must be < 50% for the curve to be valid.
- 10.8.2.3. For linear fit, the intercept of the line must be less than ½ the reporting limit, and the coefficient of determination (r<sup>2</sup>) must be greater than or equal to 0.990 for the curve to be considered valid (or the correlation coefficient (r) > 0.995).
- 10.9. Calibration Curve Fits
- 10.9.1. Linear regression or quadratic curves may be used to fit the data to a calibration function. Detailed descriptions and formulas for each fitting type can be found in SOP CA-Q-S-005, "Calibration Curves (General)".-
- 10.9.2. The linear curve uses the following function:

**Equation 1**

$$y = bx + c$$

Where:

$$y = \frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$$

x = concentration

b = slope

$c$  = intercept

10.9.3. The quadratic curve uses the following function:

**Equation 2**

$$y = ax^2 + bx + c$$

Where  $y$ ,  $x$ ,  $b$ , and  $c$  are the same as above, and  $a$  = curvature.

10.9.4. The external standard method uses the following equation:

**Equation 3**

$$\text{Response Factor} = \frac{\text{Peak Area}}{\text{Concentration of Solution (ng / mL)}}$$

10.9.5. Evaluation of Calibration Curves

The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- The absolute value of the intercept of a regression line (linear or non-linear) at zero response must be less than the reporting limit.
- There should be no carryover at or above 1/2 MRL after a high CAL standard.

If these criteria are not met, instrument conditions and standards will be checked, and the ICAL successfully repeated before continuing.

10.9.6. Weighting of Calibration Points

In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. Because accuracy at the low end of the curve is very important for this analysis, it is preferable to increase the weighting of the lower concentration points.  $1/\text{concentration}$  or  $1/x$  weighting is encouraged. Visual inspection of the line fitted to the data is important in selecting the best fit.

10.10. Initial Calibration Blank (ICB)

10.10.1. Immediately following the ICAL, a calibration blank is analyzed that consists of an injection of 80:20 methanol:water blank.

10.10.2. The result for the calibration blank must be less than the reporting limit.

10.10.3. If the ICB is greater than the reporting limit then the source of contamination must be identified and any necessary cleaning completed, and then the instrument should be recalibrated.

10.11. Initial Calibration Verification (ICV)

- 10.11.1. Following the ICAL and the ICB, an ICV standard obtained from a different source or vendor than the ICAL standards is analyzed. This ICV standard is a mid-range standard.
- 10.11.2. The recovery for the ICV must meet the appropriate following criteria;
  - 10.11.2.1. The native analyte must be within or equal to 60-140% for all natives quantitated by isotope dilution.
  - 10.11.2.2. The native must be within or equal to 50-150% for all natives quantitated by internal standard.
  - 10.11.2.3. The IDA must be  $\geq 50$  and  $\leq 150\%$ .
- 10.11.3. See Section 9.7 for corrective actions in the event that the ICV does not meet the criteria above.
- 10.12. Continuing Calibration Verification (CCV) – At the beginning of a run, the end of a run, and after every 10 samples are analyzed a CCV must be injected to determine if the calibration is still valid. The exception is after an acceptable curve and ICV are run 10 samples can be analyzed before a CCV is required. The CCVs are usually at the mid level range of the curve and should vary throughout the run. The curve and ICV do not need to be run every day. To start an analytical run a CCV can be analyzed and if it meets acceptance criteria a run can be started. In addition the low standard in the curve must be analyzed and must be within  $\pm 50\%$  of the expected value.
  - 10.12.1. The recovery for the CCV standards must be equal to or within 60-140% for all natives quantitated by isotope dilution and equal to or within 50% to 150% for all natives quantitated by internal standard. The recovery for the IDA  $\geq 50$  to  $\leq 150\%$ .
  - 10.12.2. If this is not achieved, the instrument has drifted outside the calibration limits. The instrument must be recalibrated.

## 11. PROCEDURE

- 11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Non-Conformance Memo (NCM). The NCM process is described in more detail in SOP WS-QA-0023. The NCM shall be filed in the project file and addressed in the case narrative.
- 11.2. Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

### 11.3. Water Sample Preparation

- 11.3.1. Visually inspect samples for the presence of settled and or suspended sediment. If sediment is apparent, filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent). Gravity or vacuum can be used to pass the sample through the filter. Prepare a filtration blank with any samples requiring filtration.

**Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.**

- 11.3.2. Measure 500 mL of each sample using a graduated cylinder and pour into a labeled 16 oz polypropylene (PP) bottle. *Prepare separate aliquots of 1.0 mL if EtFOSA and/or MeFOSA are requested.*
- 11.3.3. Prepare additional aliquots of a field sample for the MS/MSD, if requested.
- 11.3.4. Prepare two 500 mL aliquots of HPLC-grade water for the method blank and LCS. (or 1.0 mL if EtFOSA and/or MeFOSA are requested.)
- 11.3.5. Spike the LCS and MS/MSD (if requested) with 0.020 mL (20 uL) of the LCS/Matrix PFC Spike solution (Section 7.6). This will result in a sample concentration of 40 ng/L. If EtFOSA and/or MeFOSA are required, increase the amount of LCS Matrix PFC spike solution added to 100 uL.
- 11.3.6. Add 0.05 mL (50 uL) of the IDA PFC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial. If EtFOSA and/or MeFOSA are requested increase the amount of IDA added to 250 uL.
- 11.3.7. If EtFOSA and/or MeFOSA are requested adjust the final volume (FV) of these aliquots to 5.0 mL with MeOH. Vortex each sample. Then transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

### 11.4. Solid Phase Extraction (SPE) – *(Do not perform SPE clean up if EtFOSA and/or MeFOSA are requested.)*

*The automated Zymark Auto-Trace Workstation can be used as long as the program follows these conditions and passes the PFC background check.*

- 11.4.1. Condition the SPE cartridges (Waters WAX, 500 mg/6 cc) by passing the following without drying the column.

***NOTE:** The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

**WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.**

- 11.4.2. Wash with 5.0 mL of 0.3% NH<sub>4</sub>OH/methanol.
- 11.4.3. Wash with 5.0 mL of 0.1N NaOH/water. Close valve when ~ 200  $\mu$ L remains on top to keep column wet. *After this step, the columns cannot go dry until the completion of loading and rinsing samples.*
- 11.4.4. Appropriately label the columns and add the reservoir to the column.
- 11.4.5. Add samples to the columns and with vacuum, pull the entire 500 mL aliquot of the sample through the cartridge at rate of approximately 2 to 5 drops per second.
- 11.4.6. After the final loading of the sample but before completely passed through the column, rinse the SPE column with 1 mL of water.
- 11.4.7. After the sample and water rinse has completely passed through the cartridge, allow the column to dry well with vacuum for 15 minutes.
- 11.5. **SPE Column Wash of Aqueous** with hexane –
  - 11.5.1. Load the first 5 mL of hexane to soak for five minutes, then elute to waste.
  - 11.5.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).
  - 11.5.3. Allow the column to dry with vacuum for 5 to 10 minutes. Columns must be dried before continuing.
- 11.6. **SPE Elution** – using 15 mL polypropylene test tube as receiving tube in the SPE manifold.
  - 11.6.1. Rinse samples bottles with 5 mL of 0.3% NH<sub>4</sub>OH/methanol and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.
  - 11.6.2. Repeat sample bottle to column reservoir rinse and cartridge elution with a second 5 mL aliquot of 0.3% NH<sub>4</sub>OH/methanol. The total collection

should be approximately 10 mL.

#### 11.7. **Extract Concentration**

- 11.7.1. Concentrate each sample under a gentle stream of nitrogen to near dryness.
- 11.7.2. Add 800 uL of methanol to each extract, soak and vortex to mix well to reconstitute extract.
- 11.7.3. Add 200 uL of water to each sample for a final solvent composition of 80:20 Methanol:Water and vortex to mix the mixture well.
- 11.7.4. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.
- 11.7.5. Seal the vial with a polypropylene screw cap. *Note: Teflon lined caps can not be used due to detection of low level concentration of PFCs.*

#### 11.8. **Soil, Sediment and Tissue Sample Preparation and Extraction**

- 11.8.1. Visually inspect soil samples for homogeneity.
- 11.8.2. Weigh a representative 5 g aliquot of soil, sediment or tissue sample into a 50 mL polypropylene wide-mouth bottle. Weigh additional sample amounts for the matrix spike and matrix spike duplicate analyses if they are requested. (*Prepare separate aliquots if EtFOSA and/or MeFOSA are requested.*)
  - 11.8.2.1. For the method blank and LCS matrix, use 5 g each of Ottawa sand.
- 11.8.3. Spike the LCS and MS/MSD (if requested) with 0.020 mL (20 uL) of the LCS/Matrix PFC Spike solution (Section 7.6). This will result in a sample concentration of 4.0 ng/L.
- 11.8.4. Add 0.05 mL (50 uL) of the internal standard PFC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial.
- 11.8.5. Cap the bottles and allow the spike to settle into the sample matrix. Gently shake the bottles to mix the spike into the matrix.
- 11.8.6. Add 20 mL of 0.4% KOH/methanol to each sample.
- 11.8.7. Shake each sample on an orbital shaker at room temperature for 3 hours.



- 11.8.8. Following the shaking, extract the samples in an ultrasonic water bath for an additional 12 hours.
  - 11.8.9. After the completion of extraction, centrifuge each sample at 3500 rpm for 15 minutes.
  - 11.8.10. Collect and decant the KOH/methanol extract to a new 50-mL centrifuge tube.
  - 11.8.11. Add another 2 mL of 0.4% KOH/methanol solution to the residue, briefly shake to mix and centrifuge at 3500 rpm for 15 minutes.
  - 11.8.12. Combine the rinsate to the first corresponding tubes.
  - 11.8.13. To the final KOH/methanol extract, add 2 mL of water to each. (*Omit this step if EtFOSA and/or MeFOSA are requested.*)
  - 11.8.14. Concentrate the KOH/methanol/water extract under nitrogen to less than 2 mL, and dilute with water to 15 mL final volume. (*Omit this step if EtFOSA and/or MeFOSA are requested.*)
  - 11.8.15. Acidify with 80 uL of glacial acetic acid, and mix the contents well with vortex mixer. Check the pH to ensure pH is between 6 to 8.
  - 11.8.16. Centrifuge at 3500 rpm for 15 minutes.
- 11.9. **Solid Cleanup by SPE.** (Do not perform SPE clean up if EtFOSA and/or MeFOSA are requested. Proceed directly to Section 11.12)
- 11.9.1. Set up WAX 150 mg/6 cc SPE columns for sample cleanup using vacuum manifold.
  - 11.9.2. Condition the SPE cartridges by passing the following without drying the column.  
  
*NOTE: The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*
- WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.**
- 11.9.3. Wash with 5.0 mL of 0.3% NH<sub>4</sub>OH/methanol.
  - 11.9.4. Wash with 10 mL of 0.1N NaOH/water. Close valve when ~ 500uL

remains on top of column to keep column wet. *After this step, the columns cannot go dry until the completion of loading and rinsing samples.*

- 11.9.5. Add extracts to the columns and with vacuum, pull the entire extracts through the cartridge at rate of approximately 3 to 5 drops per second.
- 11.9.6. Rinse the sample tube with 5 mL of water and add to the SPE column.
- 11.9.7. Dry the columns with vacuum for 15 minutes.
- 11.10. **SPE Column Wash of solids** with hexane –
  - 11.10.1. Load the first 5 mL of hexane to soak for five minutes, and elute to waste.
  - 11.10.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).
  - 11.10.3. Allow the column to dry with vacuum for 10 minutes. Columns must be dried before continuing.
- 11.11. **SPE Elution** – using 15 mL polypropylene test tube as receiving tube in the SPE manifold.
  - 11.11.1. Elute the analytes from the cartridge with 5.0 mL of 0.3% NH<sub>4</sub>OH/methanol, first allow the solution to soak for 5 minutes, and then elute into the 15 mL collection tube.
  - 11.11.2. Add a second 5 mL of 0.3% NH<sub>4</sub>OH/methanol and collect the eluant into the collection tube. The total collection should be approximately 10 mL.
- 11.12. **Extract Concentration**
  - 11.12.1. Concentrate each sample under a gentle stream of nitrogen to near dryness.
  - 11.12.2. Add 800 uL of methanol to each extract, soak and vortex to mix well to reconstitute extract.
  - 11.12.3. Add 200 uL of water to each sample for a final solvent composition of 80:20 Methanol:Water and vortex to mix the mixture well.
  - 11.12.4. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.
  - 11.12.5. Seal the vial with a polypropylene screw cap. *Note: Teflon lined caps can not be used due to detection of low level concentration of PFCs.*

11.13. **Other types of Sample Cleanup**

11.13.1. **Freezing technique** to remove lipids.

11.13.1.1. If samples contain lipids then freeze the methanolic extract and QC extracts at -20°C for at least 1 hour. Collect the solvent layer.

11.13.2. **Cleanup with graphitized carbon** which may also be used to remove organic interferences.

11.13.2.1. Add 100 mg of graphitized carbon to each sample extract and QC extracts.

11.13.2.2. Shake vigorously and then let sit for 10 minutes.

11.13.2.3. Centrifuge each sample for 2 minutes at 1000 rpm.

11.13.2.4. Decant the solvent layer

11.13.3. Concentrate each sample under a gentle stream of nitrogen to approximately 0.5 mL.

11.13.4. Add 200 uL of Millipore water to each sample.

11.13.5. Bring the final volume to 1.0 mL with methanol (80% methanol/20% water).

11.13.6. Filter through a 0.45 µm syringe filter as necessary or centrifuge the extracts to obtain a clear supernant. *Note: Syringe filter should be checked for PFC background before using.*

**WARNING: Application of excessive pressure has caused disc filters to rupture and burst. Exercise discretion when filtering.**

11.14. **Instrument Analysis**

11.14.1. Suggested operation conditions are listed below:

<b>Routine Instrument Operating Conditions</b>					
<i>HPLC Conditions (Waters Acquity UPLC)</i>					
<b>Column (Column temp = 50°C)</b>	Waters Acquity BEH 1.7µm C18, 3.0 x 150 mm				
<b>Mobile Phase Composition</b>	A = 20 mM Ammonium Acetate in Water		B = Methanol		
<b>Gradient Program</b>	<b>Time</b>	<b>%A</b>	<b>%B</b>	<b>Curve</b>	<b>Flow Rate mL/min.</b>
	0	98	2	6	0.30
	1	98	2	6	0330

	2	50	50	6	0.30
	12	10	90	6	0.30
	12.5	0	100	6	0.30
	16	0	100	6	0.30
	16.2	98	2	6	0.30
	Maximum Pressure limit = 15,000 psi				
<b>Injection Size</b>	10 $\mu$ L (fixed amount throughout the sequence)				
<b>Run Time</b>	~20 minutes				

<b>Mass Spectrometer Interface Settings (Quattro Premier XE)</b>	
<b>MS Interface Mode</b>	ESI Negative Ion
<b>Capillary (kV)</b>	2.8
<b>Cone (V)</b>	Varies from 8.0 to 65
<b>Extractor (V)</b>	3
<b>Source Temp</b>	135°C
<b>Desolvation Temp</b>	350°C
<b>Cone Gas (nitrogen) Flow</b>	25 L/hour
<b>Desolvation Gas (nitrogen) Flow</b>	1100 L/hour

<b>Mass Spectrometer Scan Settings</b>						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
PFBA	Perfluorobutanoic acid	213 > 169	0.02	8	10	1
13C4-PFBA	IS	217 > 172	0.02	12	10	1
PFPeA	Perfluoropentanoic acid	263 > 219	0.02	10	10	2
13C5PFPeA	IS	268 > 223	0.02	11	9	2
PFBS	Perfluorobutanesulfonate	299 > 80	0.02	45	35	2
PFHxA	Perfluorohexanoic acid	313 > 269	0.02	10	10	3
13C2-PFHxA	IS	315 > 270	0.02	12	9	3
PFHpA	Perfluoroheptanoic acid	363 > 319	0.02	10	10	4
13C4-PFHpA	IS	367 > 322	0.02	12	10	4
PFHxS	Perfluorohexanesulfonate	399 > 80	0.02	55	35	4
18O2-PFHxS	Internal standard	403 > 84	0.02	50	40	4
PFOA	Perfluorooctanoic acid	413 > 369	0.02	12	10	5
13C4PFOA	IS	417 > 372	0.02	12	12	5
PFHpS	Perfluoroheptanesulfonate	449 > 80	0.02	60	38	5
PFNA	Perfluorononanoic acid	463 > 419	0.02	16	10	7
13C5-PFNA	IS	468 > 423	0.02	12	12	7
PFOS	Perfluorooctanesulfonate	499 > 80	0.02	60	40	6
13C4-PFOS	IS	503 > 80	0.02	35	48	6

<b>Mass Spectrometer Scan Settings</b>						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
PFDA	Perfluorodecanoic acid	513 > 469	0.02	16	12	8
813C2-PFDA	IS	515 > 470	0.02	14	12	8
PFUdA	Perfluoroundecanoic acid	563 > 519	0.02	15	12	10
13C2-PFUdA	IS	565 > 520	0.02	14	12	10
PFDS	Perfluorodecanesulfonate	599 > 80	0.02	74	48	10
FOSA	Perfluorooctanesulfonamide	498 > 78	0.02	40	32	9
13C8-FOSA	IS	506 > 78	0.02	48	32	9
PFDoA	Perfluorododecanoic acid	613 > 569	0.02	15	14	11
13C2-PFDoA	IS	615 > 570	0.02	16	12	11
PFTTrDA	Perfluorotridecanoic acid	663 > 619	0.02	12	12	11
PFTeDA	Perfluorotetradecanoic acid	713 > 669	0.02	12	18	11
PFHxDA	Perfluorohexadecanoic acid	813 > 769	0.02	18	15	12
PFODA	Perfluorooctadecanoic acid	913 > 869	0.02	20	16	12
FOSAA *	Perfluorooctanesulfonamidacetic acid	556 > 498	0.02	40	28	13
PFPeS	Perfluoropentanesulfonate	3749 > 80	0.02	55	32	3
PFNS	Perfluorononanesulfonate	549 > 80	0.02	65	54	8
PFDoS	Perfluorododecanesulfonate	699 > 80	0.02	80	55	11
<sup>13</sup> C <sub>2</sub> PFTeDA	<sup>13</sup> C <sub>2</sub> -Perfluorotetradecanoic acid IS for PFTeDA	715 > 670	0.02	15	15	11
<sup>13</sup> C <sub>2</sub> PFHxDA	<sup>13</sup> C <sub>2</sub> -Perfluorohexadecanoic acid IS for PFHxDA	815 > 770	0.02	18	15	12
EtFOSA	N-ethylperfluoro-1-octanesulfonamide	525.7 > 168.8	0.02	45	36	11
d5EtFOSA	IS for EtFOSA	530.7 > 168.7	0.02	40	30	11
MeFOSA	N-methylperfluoro-1-octanesulfonamide	511.7 > 168.8	0.02	45	25	11
d5MeFOSA	IS for MeFOSA	514.6 > 168.7	0.02	40	30	11
EtFOSAA	N-ethylperfluoro-1-octanesulfonamidoacetic acid	583.6 > 418.8	0.02	35	20	9
d5-EtFOSAA	IS for EtFOSAA	588.6 > 418.8	0.02	30	25	9
MeFOSAA	N-methylperfluoro-1-octanesulfonamidoacetic acid	569.6 > 418.8	0.02	30	28	9
d5-MeFOSAA	IS for MeFOSAA	572.6 > 418.8	0.02	30	25	9
6:2FTS	Sodium 1H,1H,2H,2H-perfluorooctane sulfonate	426.7 > 406.8	0.02	40	30	5
M2-6:2FTS	IS for 6:2FTS	428.7 > 408.8	0.02	40	28	5
8:2FTS	Sodium 1H,1H,2H,2H-perfluorodecane sulfonate	526.6 > 506.7	0.02	40	28	8

<b>Mass Spectrometer Scan Settings</b>						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
M2-8:2FTS	IS for 8:2FTS	528.6 > 508.7	0.02	40	28	8

\* Chromatographs poorly, analyte is not analyzed.

Native Compounds	Native RT (minutes)	IS analog	IS RT (minutes)	Quantitation Method
PFBA	4.77	13C4-PFBA	4.79	Isotope Dilution
PFPeA	5.90	13C5-PFPeA	5.92	Isotope Dilution
PFBS	6.01	18O2-PFHxS	8.64	IS calculation
PFHxA	7.22	13C2-PFHxA	7.25	Isotope Dilution
PFPeS	7.30	18O2-PFHxS	7.32	IS calculation
PFHpA	8.57	13C4-PFHpA	8.59	Isotope Dilution
PFHxS	8.60	18O2-PFHxS	8.64	Isotope Dilution
PFOA	9.80	13C4-PFOA	9.83	Isotope Dilution
PFHpS	9.80	13C4-PFOS	10.90	IS calculation
PFNA	10.88	13C5-PFNA	10.92	Isotope Dilution
PFOS	10.87	13C4-PFOS	10.90	Isotope Dilution
PFNS	11.80	13C4-PFOS	10.90	IS calculation
PFDA	11.82	13C2-PFDA	11.86	Isotope Dilution
FOSA	12.41	13C8-FOSA	12.46	Isotope Dilution
PFDS	12.57	13C4-PFOS	10.90	IS calculation
PFUdA	12.62	13C2-PFUdA	12.66	Isotope Dilution
PFDoA	13.32	13C2-PFDoA	13.34	Isotope Dilution
PFDoS	13.89	13C4-PFOS	10.90	IS calculation
PFTTrDA	13.91	13C2-PFDoA	13.34	IS calculation
PFTeDA	14.39	13C2-PFDoA	13.34	IS calculation
PFHxDA	15.16	13C2-PFDoA	13.34	IS calculation
PFODA	15.57	13C2-PFDoA	13.34	IS calculation
EtFOSA	14.13	d-EtFOSA	14.11	Isotope Dilution
MeFOSA	13.73	d-MeFOSA	13.73	Isotope Dilution
EtFOSAA	12.63	D5-EtFOSAA	12.62	Isotope Dilution
MeFOSAA	12.3	D3-MeFOSAA	12.28	Isotope Dilution
6:2FTS	10.08	M2-6:FTS	10.08	Isotope Dilution
8:2FTS	11.95	M2-8:FTS	11.95	Isotope Dilution

11.14.2. Tune and calibrate the instrument as described in Section 10.

11.14.3. A typical run sequence is as follows:

- Primer (A number of primers are injected for conditioning of the instrument before analysis, especially when the instrument was idled or changed from a different analysis).
- Blank
- Calibration Curve
- ICB
- ICV
- MB
- LCS
- LCSD (if applicable)
- Sample 1
- Sample 1 MS (if applicable)
- Sample 1 MSD (if applicable)
- Sample 2 (up to sample 10 before next CCV)
- CCV
- Up to 10 samples.
- End sequence with CCV

## 12. CALCULATIONS

12.1. If the concentration of the analyte ions exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. It may be necessary to dilute samples due to matrix.

12.2. Qualitative Identification

12.2.1. The retention times of PFC's with labeled standards must be the same as that of the labeled internal standards to within 0.05 min. For PFC's with no labeled standards, the RT must be within  $\pm 0.3$  minutes of the ICV and CCV standards. *Note: The IS RT and native RT may be offset by 0.02 to 0.04 minutes.*

12.3. The ICAL established in Section 10 is used to calculate concentrations for the extracts.

12.4. Extract concentrations are calculated as below. The first equation applies to the linear fit, the second to the quadratic line fit.

**Equation 4**                      Concentration, ng/mL =  $\frac{y - c}{b}$

**Equation 5**                      Concentration, ng/mL =  $\frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$

Where:

$$y = \frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$$

x = concentration  
a = curvature  
b = slope  
c = intercept

12.5. Water Sample Result Calculation:

**Equation 6**                      Concentration, ng/L =  $\frac{C_{ex} V_t}{V_o}$

Where:

$C_{ex}$  = Concentration measured in sample extract (ng/mL)  
 $V_t$  = Volume of total extract (mL)  
 $V_o$  = Volume of water extracted (L)

12.6. Soil Sample Result Calculation:

**Equation 7**                      Concentration, ng / g =  $\frac{C_{ex} V_t}{W_s D}$

Where ng/g = μg/kg and:

$C_{ex}$  = Concentration measured in sample extract (ng/mL)  
 $V_t$  = Volume of total extract (mL)  
 $W_s$  = Weight of sample extracted (g)  
 $D$  = Fraction of dry solids, which is calculated as follows:  

$$\frac{100 - \% \text{ moisture in sample}}{100} \quad (\text{for dry weight result})$$

12.7. IDA Recovery Calculation:

**Equation 8**                      % Recovery =  $\frac{RF_{ex} A_t}{Amt}$

Where ng/g = μg/kg and:

$RF_{ex}$  = Response Factor for IDA compound  
 $A_t$  = Area response for IDA compound  
 $Amt$  = Amount spike of IDA



- 12.8. If results are to be reported as ammonium perfluorooctanoate (APFO), instead of PFOA, apply a multiplier of 1.0406 to the sample results to correct for the molecular weight differences between PFOA and APFO or this adjustment can be made during the preparation of the standards used for calibration. (Use one, not both.)
- 12.9. Raw data, calibration summaries, QC data, and sample results are reviewed by the analyst. These must also be reviewed thoroughly by a second qualified person. See the Data Review Policy (WS-PQA-0012). These reviews are documented on the Data Review Checklist.

### **13. METHOD PERFORMANCE**

- 13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.
- 13.2. **Method Detection Limit**  
The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.
- 13.3. **Initial Demonstration of Capability (IDOC)**  
Each analyst performing this procedure must successfully analyze four LCS QC samples using current laboratory LCS control limits. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.
- 13.4. The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in WS-QA-0006 and policy WS-PQA-003.

### **14. POLLUTION PREVENTION**

- 14.1. All waste will be disposed of in accordance with Federal, State and Local regulations.
- 14.2. Solid phase extraction used for water samples greatly reduces the amount of solvent used compared to liquid-liquid extraction.

- 14.3. Standards and reagents are purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.
- 14.4. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 14.5. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless waste is being transferred.
- 14.6. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

## 15. WASTE MANAGEMENT

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

- 15.1. Assorted test tubes, autovials, syringes, filter discs and cartridges. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the correct steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.
- 15.2. Extracted soil samples, used sodium sulfate, paper funnel filters, glass wool, thimbles, and extracted solids contaminated with solvents. Dump these materials into an orange contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the appropriate steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.
- 15.3. Waste Methanol. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.
- 15.4. Mixed water/methanol waste from soil extraction. Collect the waste in the HPLC waste carboy. When full, or after no more than one year, dump into the blue plastic HPLC collection drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.

- 15.5. Aqueous acidic waste from the LCMS instrument contaminated with methanol. This is collected in a 1-gallon carboy at the instrument. When the carboy is full, or after no more than one year, it is emptied into the blue plastic HPLC collection drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.
- 15.6. Autovials contaminated with methanol. As the autovials are removed from the instrument after analysis, they are collected in open containers at the instrument. After all autovials are removed, the open container must be dumped into a closed satellite collection container in a fume hood, as the punctured septa in the autovial can allow methanol and other contaminants to evaporate into the atmosphere. The satellite collection containers are transferred to the waste disposal area when full or after no more than one year, where they are disposed through the vial eater.

## 16. REFERENCES

- 16.1. Cheryl Moody, Wai Chi Kwan, Johnathan W. Martin, Derek C. G. Muir, Scott A. Mabury, "Determination of Perfluorinated Surfactants in Surface Water Samples by Two Independent Analytical Techniques: Liquid Chromatography/Tandem Mass Spectrometry and <sup>19</sup>F NMR," *Analytical Chemistry* 2001, 73, 2200-2206.
- 16.2. John Giesy et al., "Accumulation of Perfluorooctane Sulfonate in Marine Mammals", *Environmental Science & Technology*, 2001 Vol. 35, No. 8, pages 1593-1598.
- 16.3. U.S. EPA, "Residue Chemistry Test Guidelines, OPPTS 860.1340, Residue Analytical Method", EPA 712-C-95-174, August 1995.
- 16.4. STL Denver White Paper DEN-W-LC-002, "Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, September 5, 2003.
- 16.5. STL Denver White Paper DEN-W-LC-003, "Addendum A to Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, August 6, 2003.
- 16.6. STL Denver White Paper DEN-W-LC-004, "Method Validation Study for Analysis of Perfluorooctanoic Acid in Waters by High Performance Liquid Chromatography/Tandem Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, January 26, 2005.
- 16.7. Waters application note; "Acquity UPLC System for Quantifying Trace Levels of Perfluorinated Compounds with an Acquity PFC Analysis Kit", Peter J. Lee, Evan

T. Bernier, Gordon T. Fujimoto, Jeremy Shia, Michael S. Young, and Alice J. Di Gloia, Waters Corporation, Milford, MA. USA.

- 16.8. US EPA, "Method 537 - Determination of Selected Perfluorinated alkyl acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)", Version 1.1, September 2009, J.A. Shoemaker, P.E. Grimmer, B.K. Boutin, EPA Document #: EPA/600/R-08/092

## 17. METHOD MODIFICATIONS

Modifications from Method 537 are detailed below:

- 17.1. Waters are extracted at 500 mL, not 250 mL.
- 17.2. Water sample containers are not preserved with Trizma. Holding time has been changed to 7 days for extraction.
- 17.3. The method has been modified to address soil/solid matrices. The extraction holding time is set at 14 days.
- 17.4. The analyte list has been expanded. The number of labeled analytes has been expanded as well to improve quantitation.
- 17.5. The reporting limits differ as they are all set at one consistent value.
- 17.6. Calibration levels differ from the referenced method.
- 17.7. More labeled analytes are fortified into the samples prior to the extraction process. Most target analytes are quantitated against a labeled analyte.
- 17.8. There is no symmetry requirement.
- 17.9. Calibration, both initial and continuing, has different acceptance criteria due to the longer list of analytes, and the use of internal standard/external standard quantitation.
- 17.10. The eluents and HPLC configuration differs. As a result the final extract is in 80:20 methanol: water.
- 17.11. The LCS and MS/MSD are spiked at one concentration and do not rotate between a low to high levels.
- 17.12. Samples are not checked for residual chlorine or pH.
- 17.13. A different SPE cartridge (Waters OASIS WAX) is used for the extraction process. As a result solvents and elution procedures are different.

## 18. ATTACHMENTS

There are no attachments to this SOP.

## 19. REVISION HISTORY

- 19.1. WS-LC-0025 Rev. 1.7, Effective 03/18/2016
- 19.1.1. Section 4.5 – Deleted the last sentence in this section: “Until more information is available” and changed “excluded” to “included”.
  - 19.1.2. Section 4.7 - Deleted the last sentence. “Chromatographic peaks in a sample must be integrated in the same way as the CAL standard.”
  - 19.1.3. Section 7.4 – Changed upper calibration limit (CS-7) for all analytes from 500 ng/mL to 400 ng/mL
  - 19.1.4. Section 9.8.2 – Revised 1<sup>st</sup> sentence to “IDA recoveries are flagged if they are outside of the acceptance limits (25–150%)”
  - 19.1.5. Section 11.3.5 – Added to end of Section, “If EtFOSA and/or MeFOSA are required, increase the amount of LCS Matrix PFC spike solution added to 100 uL.”
  - 19.1.6. Editorial changes.
- 19.2. WS-LC-0025 Rev. 1.6, Effective January 22, 2016
- 19.2.1. Section 11.6.1 – Revised to include rinse of sample container
  - 19.2.2. Section 11.6.2 – Revised to include rinse of sample container.
  - 19.2.3. Editorial changes
- 19.3. WS-LC-0025 Rev. 1.5, Effective November 1, 2015
- 19.3.1. Added the analytes EtFOSA, MeFOSA, EtFOSAA, MeFOSAA, 6:2FTS and 8:2FTS to Sections 1.1, 7.4 and 11.14.
  - 19.3.2. Added Sections 2.5, 10.9.4 and 12.7 to address external standard quantitation.
  - 19.3.3. Section 9.8 was updated and Section 12.7 added to address the calibration and quantitation of IDA compounds.
  - 19.3.4. Added verbiage to Section 11 to address specifics if EtFOSA and/or MeFOSA are requested.

- 19.3.5. Added Section 11.3.7 for the specific processing of aqueous samples for EtFOSA and/or MeFOSA.
- 19.3.6. Added analytes and pertinent information to Section 11.14.1.
- 19.3.7. Editorial changes
- 19.4. WS-LC-0025 Rev. 1.4, Effective August 28, 2015
  - 19.4.1. Updated copyright statement on cover page.
  - 19.4.2. Section 1.1 – Renamed sulfonates to sulfonic acids and corrected the CAS# for PFHpS and PFOS. Removed FOSSA from table.
  - 19.4.3. Section 7.4 – Renamed sulfonates to sulfonic acids and removed FOSSA remark.
  - 19.4.4. Section 6.18 – Routine and Preventative maintenance table added.
  - 19.4.5. Added Section(s) 17.1 thru 17.13 - Method modifications to Method 537.
  - 19.4.6. Editorial changes.
- 19.5. WS-LC-0025 Rev. 1.3, Effective Oct. 31, 2014
  - 19.5.1. Removed references to glass containers in Section 8.1, 8.2 and Notes following Section 8.
  - 19.5.2. Editorial changes
- 19.6. WS-LC-0025 Rev. 1.2, Effective July 5, 2013
  - 19.6.1. Updated Tables in Section 11.14 with current specifications.
  - 19.6.2. Editorial changes.
- 19.7. WS-LC-0025 Rev. 1.1, Effective May 25, 2012
  - 19.7.1. Editorial revisions.
- 19.8. WS-LC-0025 revision 1.0, Effective May 3, 2011
  - 19.8.1. This is the original SOP. SOP was created from WS-LC-0020, Revisio

## **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
<b>Field Equipment</b>	
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
<b>Field Clothing and PPE</b>	
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><b>Sunscreens</b> - 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><b>Insect Repellents</b> - Jason Natural Quit Bugging Me, Repel Lemon Eucalyptus Insect repellent, Herbal Armor, California Baby Natural Bug Spray, BabyGanics</p> <p><b>Sunscreen and insect repellent</b> - Avon Skin So Soft Bug Guard Plus – SPF 30 Lotion</p>
<b>Sample Containers</b>	
LDPE or glass containers	HDPE or polypropylene
Teflon-lined caps	Unlined polypropylene caps
<b>Rain Events</b>	
Waterproof or resistant rain gear	Gazebo tent that is only touched or moved prior to and following sampling activities
<b>Equipment Decontamination</b>	
Decon 90®	Alconox® and/or Liquinox®
Water from an on-site well	Potable water from municipal drinking water supply
<b>Food Considerations</b>	
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: \_\_\_\_\_  
 Analyst Signature: \_\_\_\_\_ Date: \_\_\_\_\_ 04451

## **Appendix D**

### **Chain-of-Custody SOP**

# Corporate Remediation Group Chain-of-Custody Standard Operating Procedure

## Purpose

The purpose of this Standard Operating Procedure (SOP) is to establish a 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 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. Either ADQM or the project team marks up the COC field copy. All of the corrections will 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., 8/31/14).
2. Write the time on COC in 24-hour or military time (e.g., 1330). 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.”

# **Appendix E**

## **Shipping SOP**

# Standard Operating Procedure For Sample Packing and Shipping

Date: March 2014

Project No.:



URS Corporation  
Sabre Building, Suite 300  
4051 Ogletown Road  
Newark, DE 19713

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### Appendices

Appendix A Sample Packing and Shipping with Pictures

## 1.0 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.

## 2.0 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

## 3.0 Related Procedures

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

## 4.0 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.

### 4.1 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.

## 4.2 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.

## 5.0 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”.

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**Hazardous Materials** – “A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. The term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials”.

Quantities of certain dangerous goods may be transported as “Small Quantity Exception” or “Limited Quantity Exception” (49 CFR 173.156).

Shippers and transporters of hazardous materials or dangerous goods are required to have specialized training.

Prior to collection and/or packaging of samples in this category for shipment to a laboratory, a member of Hazardous Waste Management Network should be consulted.


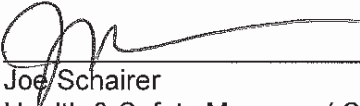




## **Appendix F**

**SOP WS-QA-0003, Revision  
12.1, Effective 4/11/2016,  
“Sample Receipt and  
Procedures [Quality  
Assurance Procedure]”**

**Title: Sample Receipt and Procedures**

**[Quality Assurance Procedure]**

Approvals (Signature/Date):			
	4/7/16		4/6/16
Koroush Vaziri Department Supervisor	Date	Joe Schairer Health & Safety Manager / Coordinator	Date
	4/7/2016		4.7.16
Lisa Stafford Quality Assurance Manager	Date	Crystal Pollock Laboratory Director	Date

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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 scissor 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, immediately close the ice chest and move it into a fume hood before continuing to unpack the ice chest.
- 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 7 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 6 of this document

before opening any coolers containing these types of samples.

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 <sup>3</sup>	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 <sup>3</sup>	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. IR thermometer calibrated at a minimum of once per quarter against an NIST reference thermometer.
- 6.2. Filament thermometer calibrated at a minimum of once annually against an NIST reference thermometer.
- 6.3. Probe thermometer capable of reading to 0.1°C calibrated at a minimum of once annually against an NIST reference thermometer.
- 6.4. pH paper (Range pH 2 to pH 12 or equivalent).
- 6.5. Pipette.

## **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.
- 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 an NIST reference thermometer.

## **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 Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance 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 Nonconformance memo shall be used for this documentation.

- 11.2. 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 11.13.

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 record sheet. Refer to this time when entering the receipt time into the LIMS 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 Shipment

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

11.4.2. 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:

- 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,
- The shipment contains concentrated product samples, or
- The client program requires opening within a fume hood.

- 11.4.3. If a shipment contains broken or leaking samples, notify the project manager via e-mail. Continue opening the cooler under the hood with cut resistant gloves. Digital pictures may be taken of the broken samples and the files attached to the project manager's email. If there are no intact samples for a given sample point and analysis, call the project manager, so that the client can be notified quickly. 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. Assume custody of the samples by signing and dating the COC in the section marked "Received for Lab By" or "Received By". This is also required for legal chain of custody. Immediately after opening, 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:
- 11.4.4.1. Use the infrared thermometer by directing the thermometer at sample containers making sure no labels or packaging materials are interfering with the direct contact of the infrared beam and the sample container. 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.
- 11.4.4.2. 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 the LIMS 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.
- 11.4.4.3. 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.
- 11.4.5. Start a folder for the login. Place the CoC in the folder. Photocopy the shipping manifest/airbill and add to the sample receiving notes page and include the 8½" x 11" facsimile in the folder. 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.
- 11.4.5.1. Certain clients (such as those in Alaska) may require that the custody seals be scanned/photocopied for inclusion in the permanent record.
- 11.4.6. Examine accompanying documentation. If documentation is absent and the shipment arrival was unexpected, contact the lead project manager for assistance.
- 11.4.7. 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.
- 11.4.8. Complete the bottle inventory (see Appendix 5), if it is necessary to aid in logging in. As a part of this, examine any VOA vials for bubbles, and note the number of containers with bubbles in the upper left of the box, with the total number of VOA vials in the lower right of the box. Compare the containers and their state of preservation with the list of analyses requested. Although some exceptions are permitted regarding particular clients, container types must compare to those listed in the TestAmerica Sacramento Quality Assurance Manual Tables 22.1 through 22.8. Discrepancies of this nature must also be annotated on the COC and a non-conformance memo filed.
- 11.4.9. If Encore® sample containers are present in the shipment, take the following action:
- 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 file the SDG 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" 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".

11.4.10. 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.

#### 11.5. 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.

- 11.5.1. 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.
- 11.5.2. 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 (Methods 8280 and 8290 dioxin/furan analyses are an exception, requiring extraction within thirty days).
- 11.5.3. 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 3). The receiving chemist initials and records the records the date and time on the SHT form. Any requested TAT of 10 days or less is also considered a priority.

#### 11.6. Sample Login

- 11.6.1. 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)
- 11.6.2. 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.
- 11.6.3. 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. 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.
- 11.6.4. 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.*

- 11.6.5. 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 8). VOA vials are excepted 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.

- 11.6.5.1. 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.
- 11.6.5.2. For metals samples which should be preserved and 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.
- 11.6.5.3. Store the completed preservation check form in the folder.
- 11.6.6. 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:
  - 11.6.6.1. R-14 (V) - volatile containers awaiting transfer to the VOA group refrigerators.
  - 11.6.6.2. 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."
  - 11.6.6.3. 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.
  - 11.6.6.4. C1 - dry pulp, paper samples or cassettes.
  - 11.6.6.5. 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.
  - 11.6.6.6. 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.*

- 11.6.7. Open the LIMS. Enter the login function. 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.
- 11.6.7.1. 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.
- 11.6.7.2. Complete the receipt/checklist tab. Complete anomalies if prompted.
- 11.6.7.3. Complete the information on the Login/Samples and Login/Samples tabs.
- 11.6.7.4. 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.
- 11.6.7.5. When all information has been entered, print sample labels.
- 11.6.8. Label sample containers. 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.
- 11.6.9. 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.

11.6.10. Scan the CoC and attach to the job in TALS.

11.6.11. 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.
- Bottle lot inventory.
- Temp record form.

#### 11.7. Shipping Container Return

11.7.1. 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.

11.7.2. 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, be certain the return address is recorded before stripping the container of used tape and labels. 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.

11.7.3. Packing, artificial ice, and temperature blanks are returned to the client. Packing which is deemed re-usable may be returned. 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. Secure it with cellophane tape and seal the container.

11.8. 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.

11.8.1. 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.

## 11.9. Disposal of Ice

11.9.1. 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 and the amounts disposed in this way are recorded. 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.**

## 11.10. Refrigerator and Freezer Temperatures

11.10.1. 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.

## 11.11. Internal Sample Tracking

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

11.11.1. In the LIMS, 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.

11.11.2. 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.

11.11.3. 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. Transfer of volatile containers from VOA to RD and RF do not require verification.

#### 11.12. Sending Out Subcontracted Work

- 11.12.1. 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 11.6 of this SOP. The project manager should notify the sample receiving staff as to all samples they require to be shipped out.
- 11.12.2. 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.
- 11.12.3. Samples should be packaged in a manner in accordance with the TestAmerica Sacramento Receiving Acceptance Policy. Samples are packaged in a plastic bag to insure labels do not get wet. Additional packing may be used to insure 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.
- 11.12.4. Insure 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).
- 11.12.5. 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.

#### 11.13. Client Deliveries ("Over the Counter")

- 11.13.1. 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.

11.13.2. 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.

11.14. For after hours sample receipt instructions for non-sample administrative personnel, refer to Appendix 8.

## **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 to the dumpster at the end of the day.
- 15.2. Contaminated solid packing materials, including broken glass, caused by the breakage of sample containers during shipment. Dump the solid waste into a yellow 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 - TestAmerica Sacramento Sample Receiving Acceptance Policy

- 18.2. Appendix 2 – Short Hold Test Notification
- 18.3. Appendix 3 – Bottle Lot Inventory
- 18.4. Appendix 4 – Sample Receiving Preservation Log
- 18.5. Appendix 5 – Example Bottle and other Labels
- 18.6. Appendix 6 – Flow Chart
- 18.7. Appendix 7 – Chemical Warfare Degradates – Potential Hazards in Sample Receipt
- 18.8. Appendix 8 – Handling of Blood or Other Potential Infectious Materials
- 18.9. Appendix 9 – After Hours Sample Receipt Procedures for Non-Sample Administrative Personnel
- 18.10. Appendix 10 – Sample Receipt Notes Form
- 18.11. Appendix 11 – Canister Receipt Notes Form
- 18.12. Appendix 12 – Revenue Source Tracking Procedure

## **19. REVISION HISTORY**

- 19.1. WS-QA-0003, Revision 12.1, Effective DD/MM/YYYY
  - 19.1.1. 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.
  - 19.1.2. Added paragraph 5.1.3 regarding lifting and movement of full ice chests.
  - 19.1.3. Edited paragraphs 11.8 and 14.2 regarding cleaning of ice chests that had soil samples in them.
  - 19.1.4. Added paragraph 16.2, Glass Safety SOP.
  - 19.1.5. Editorial changes
- 19.2. WS-QA-0003, Revision 12.0, Effective 12/11/2015
  - 19.2.1. Inserted Appendix 11, Canister Receipt Notes Form
  - 19.2.2. Updated Appendix 10 – Sample Receipt Notes Form

- 19.2.3. Editorial comments
- 19.3. WS-QA-0003, Revision 11.9, Effective 09/30/2014
  - 19.3.1. Added to Section 10.2. “ IR thermometers will be checked against mercury thermometer ID 4881 and the corrected temperature must be with 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 an NIST reference thermometer.”
  - 19.3.2. Editorial comments.
- 19.4. WS-QA-0003, Revision 11.8, Effective 07/12/2013
  - 19.4.1. Deleted Section 11.3.3 – computer entry SRL (Sample Receipt Login) form is not used.
  - 19.4.2. Modified Section 11.44.1 to include - Note the uncorrected and corrected sample temperature on the sample receiving notes page and write the corrected temperature on the COC.
  - 19.4.3. Editorial changes.
- 19.5. WS-QA-0003, Revision 11.7, Effective 5/3/2013
  - 19.5.1. Inserted new Sample Acceptance Policy (Appendix 1)
  - 19.5.2. Editorial revisions.
- 19.6. WS-QA-0003, Revision 11.6, Effective 12/15/2012
  - 19.6.1. Updated forms to TALS specifications.
  - 19.6.2. Editorial revisions.
- 19.7. WS-QA-0003, Revision 11.5, Effective 11/18/2011
  - 19.7.1. Deleted “purple folders” form Sections 11.5.3 and 11.6.1.4.
  - 19.7.2. Section 11.4.2.2 - Changed temperature range from 2 - 6°C to 0 - 6°C.
  - 19.7.3. Updated LRC forms in Appendices 3 – 7.
  - 19.7.4. Editorial revisions.

19.8. WS-QA-0003, Revision 11.4, Effective 06/17/2011.

19.8.1. Added Section 11.4.3 – Photocopy the shipping manifest/airbill and include the 8½“ x 11” facsimile in the folder with the Chain of Custody and LRC documentation. The shipping manifest is an integral part of the project documentation and will be scanned and included with the finalized project.

## APPENDIX 1



### SACRAMENTO LABORATORY SAMPLE ACCEPTANCE POLICY *(Effective 04/11/2013)*

The TNI Standard and TestAmerica Sacramento have specific requirements under which all samples will be received by the laboratory for analysis. 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.
- Each sample shall be collected in the appropriate sample container and labeled with unique, durable and indelible identification.
- Drinking waters samples for Method 1813B that may have residual chlorine must be checked and treated in the field, or collected in sodium thiosulfate preserved containers.
- 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.
- 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).
- 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**

<p><b>TestAmerica West Sacramento</b>  <b>SHORT HOLDING TEST NOTIFICATION</b></p>		 THE LEADER IN ENVIRONMENTAL TESTING
Client _____ Lot ID _____ Date & time received by Sample Control _____ Date & time received in Gen Chem _____ Chemist Notified _____ 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 # _____	<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	
<p>General Analysis (select those that apply)</p> <input type="checkbox"/> pH <input type="checkbox"/> Alkalinity <input type="checkbox"/> Cr6+ <input type="checkbox"/> EC <input type="checkbox"/> pH-Soils <input type="checkbox"/> Color <input type="checkbox"/> TDS/TSS <input type="checkbox"/> SO <sub>3</sub>	<p>Sample #</p> _____ _____ _____ _____	<p>Expiration Date &amp; Time</p> _____ _____ _____ _____
<p>Lachat</p> <input type="checkbox"/> NO <sub>2</sub> +NO <sub>3</sub> <input type="checkbox"/> NO <sub>2</sub> <input type="checkbox"/> NO <sub>3</sub>	_____ _____ _____	_____ _____ _____
<p>Method 365.3</p> <input type="checkbox"/> OPO <sub>4</sub>	_____ _____	_____ _____
<p>Method 300.0</p> <input type="checkbox"/> NO <sub>3</sub> <input type="checkbox"/> NO <sub>2</sub> <input type="checkbox"/> OPO <sub>4</sub> <input type="checkbox"/> Br <input type="checkbox"/> Cl <input type="checkbox"/> SO <sub>4</sub> <input type="checkbox"/> F	_____ _____	_____ _____
<p>Metals</p> <input type="checkbox"/> Dissolved Metals, Filter & Pres <input type="checkbox"/> Other _____ _____ _____	_____ _____ _____	_____ _____ _____
<p>Comments: _____          _____          _____</p>		
<p>QA-356</p>	<p>RBH 11/14/11</p>	





**APPENDIX 4**

Sample Receiving Preservation Check

**TestAmerica**

**Sacramento**  
**Sample Receiving Preservation Check**


Lot ID: \_\_\_\_\_

Instructions: Check or x the container type, then check or x the pH measurement if it meets. Write the actual pH if out.


Container	pH																				
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	
AGBs	500PJ																				
AGBs	na																				
AGBs	500PJ																				
AGBs	na																				
AGBs	500PJ																				
AGBs	na																				
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**APPENDIX 5**  
**Example Labels**

1. Label for the Folder


	<b>Loc: 320</b> <b>1197</b>
Login: 320-1197 PM: Sadler, Jeremy Company: TestAmerica Laboratories, Inc.	

2. Label for the COC

	<b>Loc: 320</b> <b>1197</b>
320-1197 Chain of	

3. Labels for the Samples



a. For the side of the container


<b>320-1197-A-1</b> 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

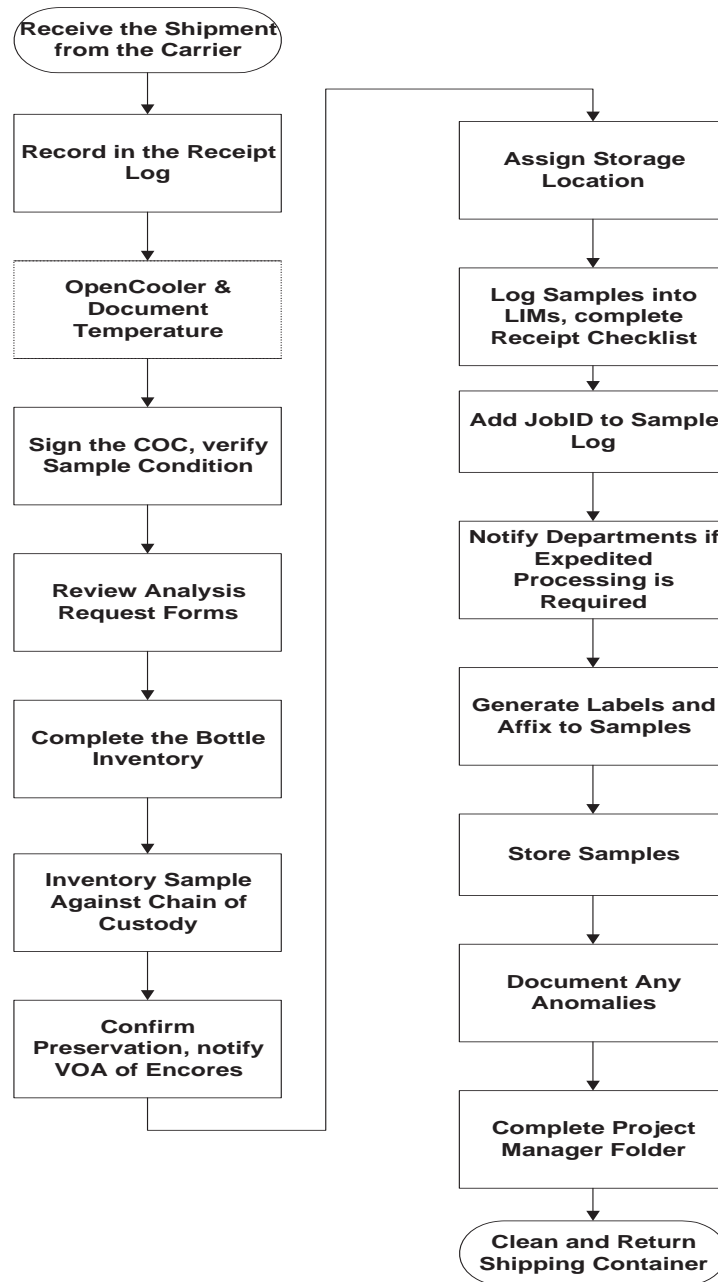
<b>Loc: 320</b> <b>1197</b> <b>#1</b> <b>A</b>
---

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 and 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-003

⇒ 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 sample 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 require 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 pair 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, 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**

**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 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 10**  
**Sample Receipt Notes**



Sacramento  
Sample Receiving Notes –

Job: \_\_\_\_\_

Tracking #: \_\_\_\_\_

Use this form to record Sample Custody Seal, Cooler Custody Seal, Temperature & corrected Temperature & other observations. File in the job folder with the COC.

Notes: _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____	Therm. ID 11 / 12 / AK / Other _____
	Cooler Custody Seal: _____
	Sample Custody Seal: _____
	Temp: Observed _____
	Corrected: _____
	From: Temp Blank <input type="checkbox"/> Sample <input type="checkbox"/>
	NCM : Yes <input type="checkbox"/> No <input type="checkbox"/>
	<b>Yes No NA</b>
	Perchlorate has headspace? <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>
	CoC is complete w/o discrepancies? <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>
	Samples received within holding time? <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>
Sample preservatives verified? <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	
Initial / Date: _____	

**APPENDIX 11**  
**Canister Receipt Notes**

Airbill Here



Sacramento

Sample Receiving Notes - Job \_\_\_\_\_

Use this form to record Sample Custody Seal, Cooler Custody Seal, Temperature & corrected Temperature & other observations.  
 File in the job folder with the COC.

Notes: _____ _____ _____ _____ _____ _____ _____ _____ _____ _____	Cooler Custody Seal: _____ # Bags: _____ 1L, _____ 2L, _____ 10L # Canisters: _____ 1L, _____ 8L, TA <input type="checkbox"/> Non TA <input type="checkbox"/> Transferred by Sacramento - Yes <input type="checkbox"/> No <input type="checkbox"/> # Canisters Unused: _____ 1L, _____ 8L # Flow Regulators: _____, # Gauges: _____ Co-locator: _____
---	---

## APPENDIX 12

### 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 7/1/2012 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.

#### Label Generation

Labels will be printed centrally and supplied to each location.

Labels will be generated with location name and a bar code.

Labels will be generated for all service center locations, including those labs that house service center functions.

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

When a location runs low on labels, they need to contact [Janet.McAuluse@testamerica.com](mailto:Janet.McAuluse@testamerica.com) to request additional labels. Two week's notice should be provided to obtain additional labels.

#### Service Center Responsibility (Beginning 7/1/2012)

Each service center will be responsible for affixing a label on the inside of the cooler lid in the upper left corner.

Service Center personnel will label every cooler that they ship out, drop off, pick up, or that is delivered to them from a client, every time.

Service Center personnel need to ensure that any old labels are removed prior to affixing their labels.

**NOTE: Labels are to be removed by lab personnel, but in order to ensure integrity of the system, the service center needs to check before applying their label!**

In cases where the service center is tracking multiple sources of revenue (such as Boston) the staff at the service center needs to distinguish between clients and / or jobs that were previously sent to a re-mission location such as Westfield. If work for a particular client or job was previously shipped to Westfield, the alternate sticker of "Boston/Westfield" is to be used on the cooler.

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

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 ensure their label is the only label on the cooler when shipping it to a lab.

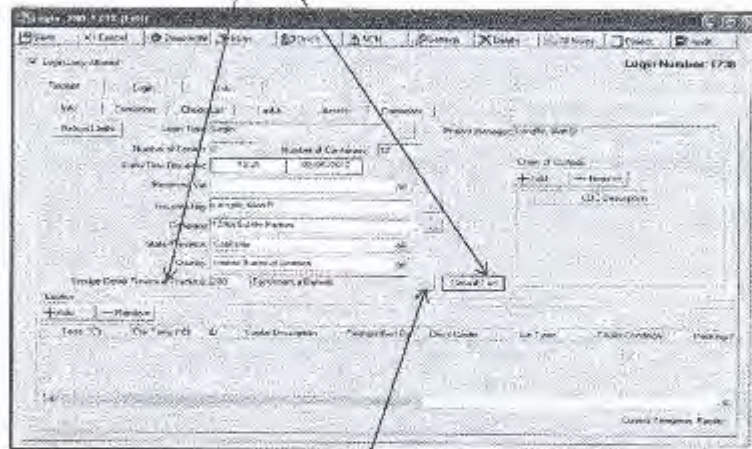


Corporate Quality  
Work Instruction for  
Laboratory / Service Center Operations

**Lab / Sample Receiving Responsibility (Beginning 7/1/2012)**

For every log in, sample receiving personnel are responsible to scan the label from the inside of the cooler lid, which will enter the "Service Center Revenue Tracking" into TALs.

If there is no label present on the inside of the cooler lid, sample receiving will select their home location by using the "Default Loc" button.



If the scanning process is not successful (scanning failure, or other reason), the service center location must be selected using the list feature in TALs.

As of July 1, 2012 this data will be considered mandatory in TALs. The login cannot be saved successfully until the Service Center Tracking field information has been completed.

**Once the label from the cooler is scanned, the label must be removed from the cooler and thrown away.**



Corporate Quality

Work Instruction for  
 Laboratory / Service Center Operations

**Project Manager (PM) Responsibility (Beginning 1/1/2012)**

The PM's should endeavor to identify the "source" of revenue on the project, or job. 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.

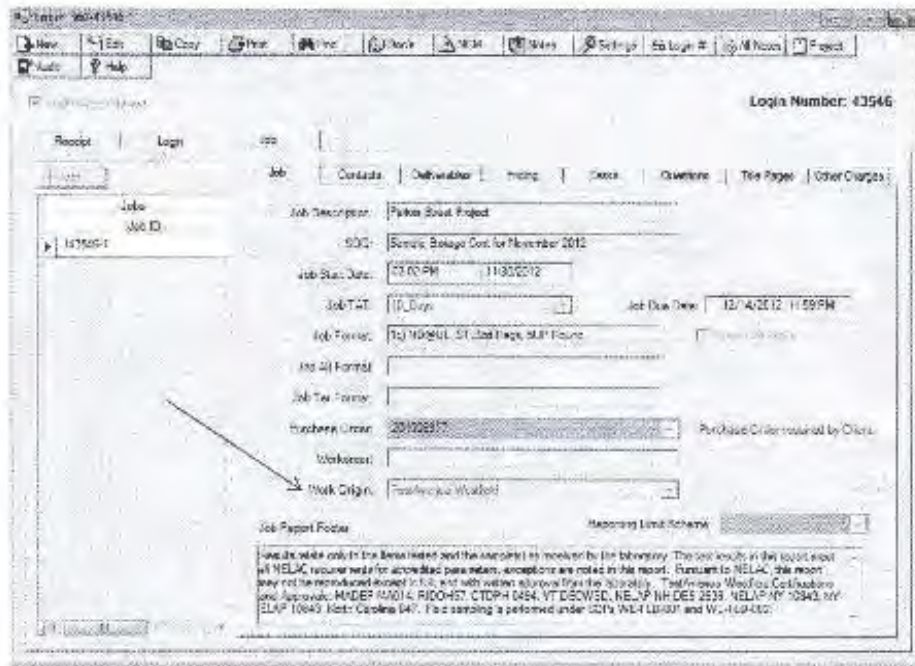
The screenshot shows a software application window with a menu bar (File, Edit, Report, Delete, etc.) and a toolbar. The main area is a form for project details. A red arrow points to the 'Service Location' dropdown menu, which is currently set to 'Westfield'. Other visible fields include 'Location' (Buffalo), 'Project Manager' (Robert Ray), 'Project No.' (00000000), and 'TAT Calculation Type' (Standard). The bottom right of the form shows financial summary fields like 'Total Reference Price' and 'Total Invoice'.



Corporate Quality

Work Instruction for  
 Laboratory / Service Center Operations

The PM 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:




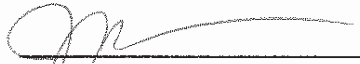
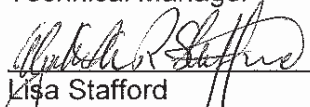
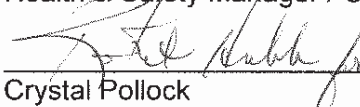
Invoices associated with projects / jobs / logins that are tagged with this information will be tracked as being sourced from a service center and / or re-mission location. It is the responsibility of PM, Service Center and Sample Login staff to ensure every effort is made to properly identify the "Source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



## **Appendix G**

**SOP WS-EHS-0001,  
Revision 4.5, Effective  
9/18/2015, “Waste Disposal”**

**Title: Waste Disposal**

Approvals (Signature/Date):			
	9-9-15		9/9/15
Richard Kester Technical Manager	Date	Joe Schairer Health & Safety Manager / Coordinator	Date
	9/11/2015		9/11/15
Lisa Stafford Quality Assurance Manager	Date	Crystal Pollock Laboratory Director	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 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, 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 in general chemistry.

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 LLE waste, landfill and high solvent lab trash.

5.1.3.3. At the organic prep/low resolution dioxin prep H-3, there are 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 (two 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.  
*Note: There is a \$5 reward for turning in solvent drums that are properly filled.*
  - 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 should 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 drum.
  - 5.4.2. VOA vials: VOA analysts collect 40 ml vials of unused sample in cardboard boxes lined with plastic bags. 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 20-L steel drum is used to collect the vials, then pour 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 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 for solvent and HPLC, and 8 for Acid 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). 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 under the radio. 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 and PPQ Form 550 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 chime 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 chime 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 chime 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. Note: Flammable drums such as Solvent or HPLC may not be stacked.

#### 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 (FS02/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).
- 5.10.2. Use the Excel spreadsheet at FS02/Public/aaSacsafe/waste/counts/shipments to estimate weights and disposal costs for the shipment. Request a Purchase Order (PO) through JD Edwards. Use the vendor number 667711 for Clean Harbors, or 544420 for Nexeo if applicable, from the safety “Purchases” folder. The cost center for waste disposal is 320.23000. Enter item one, one shipment, and the full cost of the shipment on one line. 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. 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 three (T-3). Daily procedures are posted on the inside door of the treatment room. The logbook is kept on T-2 next to the broken flow meter. Check that pH in T-3 is between 5 and 11 before discharging.
- 5.11.1. Fill tank two (T-2) at the beginning of each month. Take VOA and metals samples to Sample Control. Hold the water in T-2 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 (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.

- 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.
- 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 rampacking 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 SOP's.
- 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 4.5, Effective 09/18/2015

9.1.1. Updated copyright statement on cover page.

9.1.2. Section 5.14.9 – Added chemical oxygen demand waste to sentence.

9.1.3. Section 5.14.9 – Deleted sentence referencing Heritage recycling for COD waste.

9.1.4. Editorial changes.

9.2. WS-EHS-0001, Revision 4.3, Effective 09/05/2014

9.2.1. 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.”

9.2.2. Appended to Section 3.2 – “The West Sacramento Fire Department may be able to provide the needed fall protection/extraction harness.”

9.2.3. 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.”

9.2.4. Replaced previous Section 5.2. with the current Section 5.2 detailing criteria for disposing of sample waste.

9.2.5. 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.”

9.3. WS-EHS-0001, Revision 4.3, Effective 9/12/2013

9.3.1. Updated disposal procedures – edits to sections 2.1, 5.2, 5.4.3, 5.10.1, 5.10.2, 5.11.2, 5.12, 5.14.10, and 5.14.

9.3.2. Removed the following items from Responsibilities, Section 6.1, as they were marked “delete” in the text:

- Waste minimization plan, (Delete as we have a waiver).
- Tax return for (Delete as this is done by Bill Nash in Irvine)
  - Environmental Fee,
  - Generator Fee,
  - EPA ID verification and manifest fee,
  - Yolo County hazardous material fee, and
  - Sacramento city hazardous materials fees,
- Quarterly reports to the Sacramento Publicly Owned Treatment Works (POTW). Delete as the Sacramento municipal POTW does not require reports.

9.3.3. Removed Revision history items older than 2010. They may be found in previous versions of this SOP.

9.3.4. Editorial changes

9.4. WS-EHS-0001, Revision 4.2, Effective 9/07/2012

9.4.1. Updated disposal procedures

9.4.2. Editorial changes

9.5. WS-EHS-0001, Revision 4.1, Effective 03/18/2011

9.5.1. Editorial changes.

## **Appendix H**

### **Data Package Review Checklist**



Location –

Jobname -

Lab – TestAmerica Sacramento

Lab Report No. -

*Note – the following checklist is intended to allow an organized review of the laboratory data package for compliance with the laboratory SOP and general data usability. Results and the supporting raw data will not be deleted or discarded. Comments herein will be provided to the laboratory who will generate a revised laboratory data package, if necessary, and an electronic disk deliverable (EDD). The EDD will be uploaded to the Locus EIM database and the Data Validation 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, review text against SOP for sample prep/analysis, note if QC criteria not being met

#### RESULTS SUMMARY –

Confirm sample results reported

Compare results between executive summary spreadsheet, LIMS reports, and Quantify Compound Summary Report – no differences beyond those that can be explained by rounding.

Confirm measures of accuracy and precision per batch of samples analyzed

Location –

Jobname -

Lab – TestAmerica- Sacramento

Lab Report No. -

RESULTS SUMMARY (cont) –

Method Blanks – less than RL – further evaluated using DVM (see below)

*Field 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 lab or field blanks)*

LCS (recovery per the table below)

Internal Standard (recovery should be 25-150%)

FIELD DUPLICATES – calculate the RPD for field duplicates. Criteria are 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 is exceeded, the sample and field duplicate results only will be qualified J and considered to be estimated.

MATRIX SPIKE RECOVERY – spike recovery per the table below unless sample concentration is > 4X spike concentration. .

Confirm at least 1 matrix spike per batch of samples analyzed

If the lab uses the same sample for the matrix spike and duplicate analyses, then confirm the sample result (not the duplicate) was used for recovery calculation

Location –

Jobname -

Lab – TestAmerica- Sacramento

Lab Report No. -

#### 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 6 C)

HOLD TIME – per SOP – water samples will be extracted within 7 days from collection, soils will be extracted within 14 days from collection) and analysis within 40 days of extraction. Up to 2X the hold time results will be qualified J (estimated) or UJ (detection limit is estimated). Beyond 2X the hold time results will be qualified J and non-detect results may be qualified R (unusable).

#### RUN SEQUENCE –

Compare raw data results throughout package

Check for:

Verify initial calibration (minimum 5 points for average response factor and/or linear fit, or 6 points for quadratic fit)

ICB < RL

Low-level verification standard (recovery should be 50-150%)

Check Standard (ICV) – Second Source (recovery should be 60-140% if quant by isotope dilution, 50 – 150% if quant by internal standard )

CCV (recovery should be 60-140% if quant by isotope dilution, 50 – 150% if quant by internal standard )

#### LC/MS/MS OPERATING CONDITIONS

Scanning method – evaluate

Location –

Jobname -

Lab – TestAmerica- Sacramento

Lab Report No. -

#### MASS CALIBRATION

All masses must be found within 0.5 amu of known masses

INITIAL CALIBRATION – initial 5 standards

Correlation coefficient  $R \geq 0.990$  (Linear)

Coefficient of determination  $R^2 \geq 0.990$  (Quadratic)

#### MDL/RL DETERMINATION

Determined at least semiannually

Method or reagent blank used

$MDL < \frac{1}{2} RL$  for each target

MDL verification run at 1 ng/L

RL verification run at 2 ng/L

RETENTION TIMES – Retention time of sample peaks must be within 0.05 minutes of the retention time observed for the labeled internal standard.

#### COMMENTS

Reviewer -

Date -

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

Analyte	CAS Num	Type	LCS/LCSD/MS/MSD Limits		
			Rec. Low %	Rec. High %	Precision %
N-ethyl perfluorooctane sulfonamidoacetic acid	2991-50-6	NEtFOSAA	60	140	30
N-methyl perfluorooctane sulfonamidoacetic acid	2355-31-9	NMeFOSAA	60	140	30
Perfluorobutanesulfonic acid (PFBS)	375-73-5	PFBS	55	147	30
Perfluorodecanoic acid (PFDA)	335-76-2	PFDA	66	141	30
Perfluorododecanoic acid (PFDoA)	307-55-1	PFDoA	71	139	30
Perfluoroheptanoic acid (PFHpA)	375-85-9	PFHpA	63	135	30
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	PFHxS	58	138	30
Perfluorohexanoic acid (PFHxA)	307-24-4	PFHxA	70	136	30
Perfluorononanoic acid (PFNA)	375-95-1	PFNA	71	140	30
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	PFOS	47	162	30
Perfluorooctanoic acid (PFOA)	335-67-1	PFOA	63	141	30
Perfluorotetradecanoic acid (PFTeA)	376-06-7	PFTeA	47	130	30
Perfluorotridecanoic Acid (PFTrIA)	72629-94-8	PFTrDA	51	139	30
Perfluoroundecanoic acid (PFUnA)	2058-94-8	PFUnA	68	139	30

## **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;

## FINAL

- 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.



FINAL

**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 ( <b>RESULT_TYPE_CODE = TIC</b> ): <ul style="list-style-type: none"> <li>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.</li> <li>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.</li> <li>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).</li> <li>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. <b>RESULT_TYPE_CODE should be set to "TRG"</b>.</li> </ul>
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)

## FINAL

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	<b>SAMPLE_PURPOSE</b>	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	<b>LAB_REPORTING_LIMIT_TYPE</b>	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	<b>BASIS</b>	C3	Yes	Basis for reporting the result. Only valid values are W, D, or N.

# FINAL

Table 1. Chemours/DuPont EIM EDD format				
Field	Field Name	Length	VVL	Field Contents
<p>Notes:</p> <p>a. <i>Fields in Bold Regular font are required for all records (e.g., LAB_ID).</i></p> <p>b. <i>Fields in Italic font are required for various subsets of samples and/or analyses.</i></p> <p>c. <i>Fields In Regular font are optional.</i></p>				

## **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 Perfluorinated Compound (PFC) data for drinking water samples generated by TestAmerica Sacramento SOP “Perfluorinated Compounds (PFCs) in Water, Soils, Sediments and Tissue [Method 537 Modified]” (SOP No. WS-LC-0025, Rev 1.7) for the Chambers Works/PFC Drinking Water Sampling Program (PFC DW Sampling Program). Validation will be performed to assess the compliance of the sample data to the PFC DW Sampling Program Quality Assurance Project Plan (QAPP) and the aforementioned SOP (as applicable to the PFC DW Sampling Program). In addition, the usability of the PFC data provided by the analytical laboratory will be determined based on the general guidance provided in the “US EPA Contract Laboratory National Functional Guidelines for Organic Data Review” (10/1999; National Functional Guidelines) and “US EPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review” (08/2014 National Functional Guidelines). It should be noted that the National Functional Guidelines apply strictly to data generated by the Contract Laboratory Program (CLP) protocol and are not directly applicable to validation of data generated by Modified EPA Method 537; therefore, this SOP presents the specific data qualification actions that will be used for validation.

The validation findings will be presented in a quality assurance review (QAR) 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.

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## 2.0 EVALUATION TOOLS

- field duplicate form (DVF\_DUP.xls)

Chemistry Applications:

- Curve fitting software (DVF\_CAL.xls)
- Methods Database

## 3.0 REFERENCE DOCUMENTS

- US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (10/1999).
- US EPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (08/2014).
- TestAmerica Sacramento SOP No. WS-LC-0025, Rev 1.7.
- US EPA Method 537.
- 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 PFC data based on an evaluation of information presented in the data package deliverables. Compliance to the PFC DW Sampling Program Quality Assurance Project Plan (QAPP) including the aforementioned SOP (as applicable to the PFC DW Sampling Program) 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 Chemours). 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 PFC data based on an evaluation of the information presented in the data package deliverables. The findings of the PFC 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), estimated results (J/UJ), tentative identifications of target compound results (N), and a general qualifier for all results reported below the quantitation limit (if applicable).

The data reviewer's criteria for evaluating the usability of the PFC data and the resultant qualifications will be as stipulated on the attached Table for the Validation of PFC Data Generated for the PFC DW Sampling Program. 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 PFC Data Generated for the PFC DW Sampling Program**

Quality Control Item	Usability Criteria	Action
Temperature Upon Receipt	4±2°C	<p>If temperature is &lt;2°C, no action is required unless samples were frozen/broken and then professional judgment should be used.</p> <p>If temperature is &gt;6°C but ≤20°C, qualify positive results as estimated (“J”) and qualify “not-detected results as estimated (“UJ”).</p> <p>If temperature is &gt; 20°C, 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 &lt; 8 hours has elapsed from collection to receipt at the laboratory to determine if qualification due to elevated temperature applies.</p>
Technical Holding Time	Aqueous samples should be extracted within 7 days of sample collection and extracts should be analyzed within 40 days after extraction.	<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>, &gt; twice the holding time), qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“R”).</p>
Initial Calibration (See Note #1 for additional information.)	<p>One of the two options below:</p> <p>Option 1: RSD for each analyte calc’d by isotope dilution (ID) ≤ 35% &amp; the labeled compounds (IDA) ≤ 50%.</p> <p>Option 2: linear least squares regression for each analyte: <math>r^2 \geq 0.99</math>, intercept &lt; ½ reporting limit (RL).</p> <p>(All target PFCs are by ID for the PFC DW Sampling Program)</p>	<p><b>When evaluating initial calibration for target PFCs by ID, use professional judgment to first assess impact of any out-of-criteria labeled PFC on the corresponding target PFC.</b></p> <p>If target PFCs have %RSD &gt; 35%, qualify positive results as estimated (“J”).</p> <p>If target compounds have %RSD &gt; 70%, use professional judgment to qualify “not-detected” results as estimated (“UJ”) or unusable (“R”).</p> <p>Use professional judgment when evaluating correlation coefficients (r) and coefficients of determination (COD). If <math>r^2 &lt; 0.99</math> but <math>\geq 0.85</math>, qualify positive results as estimated (“J”) and do not qualify “not-detected” results. If <math>r^2</math> is &lt;0.85, qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“R”) using professional judgment. If intercept is <math>\geq \frac{1}{2}</math> RL (absolute value), evaluate sample data for false positives and “not-detects.”</p>

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**Table for the Validation of PFC Data Generated for the PFC DW Sampling Program**

Quality Control Item	Usability Criteria	Action
Second Source Calibration Verification	%D for each analyte calc'd by ID $\leq 40\%$ & the labeled compounds (IDA) $\leq 50\%$ .	<p><b>Qualification is for all samples associated with initial calibration being verified. When evaluating second source calibration verification for target PFCs by ID, use professional judgment to first assess impact of any out-of-criteria labeled PFC on the corresponding target PFC.</b></p> <p>If target PFC by ID has %D&gt;40% with the response indicating a sensitivity increase, qualify positive results as estimated (“J”) and do not qualify “not-detected” results.</p> <p>If target PFC by ID has %D&gt;40% but <math>\leq 90\%</math>, with the response indicating a sensitivity decrease, qualify positive results as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p> <p>If target PFC by ID has %D&gt;90% with the response indicating a sensitivity decrease, qualify positive results as estimated (“J”) and qualify “not-detected” results as unusable (“R”).</p>
Reporting Limit (RL) Verification Standard	Recovery within $\pm 50\%$ of RL	<p><b>Qualify samples analyzed after a non-compliant RL Verification.</b></p> <p>If the recovery is <math>&gt; 150\%</math>, qualify positive results <math>\leq 3\times</math> the spike level as estimated (“J”) and do not qualify “not-detected” results.</p> <p>If the recovery <math>&lt; 50\%</math>, qualify positive results <math>\leq 3\times</math> the spike level as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).</p>

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**Table for the Validation of PFC Data Generated for the PFC DW Sampling Program**

Quality Control Item	Usability Criteria	Action
Daily Calibration Verification (See Note #2 for additional information.)	%D for each analyte calc'd by ID $\leq 40\%$ & the labeled compounds (IDA) $\leq 50\%$ .	<p><b>Qualification is for all samples on both sides of the out-of-criteria calibration verification standards.</b></p> <p><b>When evaluating calibration verification for target PFCs by ID, use professional judgment to first assess impact of any out-of-criteria labeled PFC on the corresponding target PFC.</b></p> <p>If target PFC by ID has <math>\%D &gt; 40\%</math> with the response indicating a sensitivity increase, qualify positive results as estimated ("J") and do not qualify "not-detected" results.</p> <p>If target PFC by ID has <math>\%D &gt; 40\%</math> but <math>\leq 90\%</math>, with the response indicating a sensitivity decrease, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ").</p> <p>If target PFC by ID has <math>\%D &gt; 90\%</math> with the response indicating a sensitivity decrease, qualify positive results as estimated ("J") and qualify "not-detected" results as unusable ("R").</p>
Blanks (See Note #3 for additional information.)	Summarize all results greater than the method detection limit (MDL) present in the blanks. The highest positive result associated with a sample should be utilized for evaluation of contamination.	<p>If a target PFC is found in the blank but not in the associated sample(s), no action is required.</p> <p>If a sample result is <math>\leq 5\times</math> the blank result, qualify the positive result as "not detected" ("B").</p> <p>If a sample result is <math>&gt; 5\times</math> blank result, qualification is not required..</p>
Internal Standards (Labelled Analytes, spiked prior to extraction) (See Note #4 for additional information.)	% recovery for each IS in the original sample (prior to dilutions) must be within 25-150%.	<p><b>The recovery limits do not apply to samples analyzed at dilutions greater than the dilution that will result in the lower recovery limit being <math>&lt; RL</math> (typically, five-fold).</b></p> <p>If an IS recovery is outside of 25-150% but <math>\geq 10\%</math>, qualify positive results as estimated ("J") and qualify "not-detected" results as estimated ("UJ") for PFCs quantitated using that IS.</p> <p>If an IS recovery is <math>&lt; 10\%</math>, qualify positive results for the associated PFCs as estimated ("J") and qualify "not-detected" results for the associated PFCs as unusable ("R") using professional judgment (based on the signal-to-noise ratio for the IS and the expected response for the target PFC at the RL).</p>

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**Table for the Validation of PFC Data Generated for the PFC DW Sampling Program**

Quality Control Item	Usability Criteria	Action
Laboratory Control Samples (LCS)/ Laboratory Control Sample Duplicates (LCSD)	Laboratory historically derived control limits, RPD $\leq$ 30%.	<p><b>The LCS qualification will be applied to all samples in the preparation batch.</b></p> <p>If the recovery is &gt; the upper acceptance limit, qualify positive results in all associated samples as estimated (“J”) and do not qualify “not-detected” results.</p> <p>If the recovery is &lt; the lower acceptance limit, qualify positive results in all associated samples as estimated (“J”) and qualify “not-detected” results in all associated samples as estimated (“UJ”). Use professional judgment to determine whether “not-detected” results should be qualified as unusable (“R”) due to very low recovery (taking into account the historically derived control limits, possibly recoveries &lt;10%).</p> <p>If the precision between recoveries exceeds the RPD criterion, qualify positive results as estimated (“J”) and do not qualify “not-detected” results.</p>
Matrix Spike/Matrix Spike Duplicate (MS/MSD) (If performed)	Laboratory statistically derived control limits for LCS, RPD $\leq$ 30%.	<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 PFC is &gt;4× the spiking level for that compound. RPDs calculated using MS/MSD results can be used to evaluate precision.</p> <p>If the recovery is &gt; the upper acceptance limit, qualify the positive result in the native sample as estimated (“J”) and do not qualify the “not-detected” result.</p> <p>If the recovery is &lt; the lower acceptance limit, qualify the positive result in the native sample as estimated (“J”) and qualify the “not-detected” result in the native sample as estimated (“UJ”). Use professional judgment to determine whether “not-detected” results should be qualified as unusable (“R”) due to very low recovery (taking into account the historically derived control limits, possibly recoveries &lt;10%).</p> <p>If the precision between recoveries exceeds the RPD criterion, qualify the positive result in the native sample as estimated (“J”) and do not qualify the “not-detected” result.</p>

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**Table for the Validation of PFC Data Generated for the PFC DW Sampling Program**

Quality Control Item	Usability Criteria	Action
Field/Laboratory Duplicate (See Note #5 for additional information)	RPD $\leq$ 30% when PFCs are detected in both samples $\geq$ 5 $\times$ sample-specific RL. Difference < sample specific RL when one or both samples < 5 $\times$ sample specific RL.	<b>Use the MDL or RL (depending on reporting requirements) as a numerical value for any “not-detected” result in the difference calculation. If both results are “not-detected”, a quantitative assessment of duplicate precision is not performed.</b> If the criteria are not met, qualify positive results for the out-of-criteria PFCs in the original sample and its duplicate as estimated (“J”) and qualify “not-detected” results as estimated (“UJ”).
Compound Quantitation and Qualitative Identification (See Notes #6 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.	If a target PFC result exceeds the instrument calibration range, qualify positive result as estimated (“J”). 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”). If a target PFC is <RL but $\geq$ MDL, qualify positive results as estimated (“J”).
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.	Use professional judgment to determine the need to qualify data not qualified based on the QC previously discussed. Write a brief narrative to give the 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.

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### Notes for the Validation of PFC Data Generated for the PFC DW Sampling Program

1. If the initial calibration curve %RSD is  $>2\times$  the criterion, the linearity of the first three initial calibration standards should be evaluated. If the first three initial calibration standards for the compound are linear (*i.e.*,  $r \geq 0.99$ ), do not qualify “not-detected” results. If the first three initial calibration standards for the compound are not linear, qualify “not-detected” results as estimated (“UJ”) or unusable (“R”) using professional judgment.

Use professional judgment when evaluating the concentration intercept of a calibration curve. If the concentration intercept is positive, the samples should be evaluated for false positives. If the concentration intercept is negative, the sample should be evaluated for false negatives.

2. If instrument instability (*i.e.*, several calibration verification standards with PFCs 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 (increase in instrument sensitivity).

If the continuing calibration verification standard is  $\%D >$  criterion in the direction of increased instrument sensitivity and it is determined that “not-detected” results should not be qualified, the data reviewer should note this within the QAR support documentation.

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### Notes for the Validation of PFC Data Generated for the PFC DW Sampling Program

3. 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.

4. The IS recovery limits do not apply to samples analyzed at dilutions greater than the dilution that would bring the lower recovery limit below the RL (typically, five-fold dilutions). Write a comment in the QAR addressing the issue that sample-specific method performance based on IS recoveries could not be evaluated due to the dilution required for sample analysis.
5. Duplicate samples may be collected and analyzed as an indication of overall precision. Field duplicate analyses measure both field and laboratory precision; 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

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### **Notes for the Validation of PFC Data Generated for the PFC DW Sampling Program**

original sample and the laboratory/field duplicate. Solid duplicate results are expected to have greater variance than aqueous duplicate results.

6. If a sample result exceeds the instrument calibration range (lower dilution analysis) or is less than the RL (secondary dilution), do not utilize this result when comparing an original analysis and a diluted reanalysis.

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