Quality Assurance Project Plan: Per- and Polyfluoroalkyl Substances (PFAS) Sampling in Community Water Supplies

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SECTION A. PROJECT MANAGEMENT

A1 Approval Sheet

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Kim Harris — Date

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Michelle Rousey — Date

Illinois EPA BOW, Quality Assurance Officer (QAO), Headquarters

A2 Table of Contents

Acronyms and Abbreviations

9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid; Potassium 9-chlorohexadecafluoro-3-oxanone-1-sulfonate
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid; Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate
ADONA	4,8-dioxa-3H-perfluorononanoic acid; Sodium dodecafluoro-3H-4,8-dioxanonate
ASDWA	Association of State Drinking Water Administrators
BOW	Bureau of Water
CAR	Corrective Action Report
CASRN	Chemical Abstract Service Registry Number
CCC	Continuing Calibration Check
CCH	Continuing Calibration Check High
CCL	Continuing Calibration Check Low
CCM	Continuing Calibration Check Mid
CWS	Community Water Supplies
DL	Detection Limit
DPWS	Division of Pubic Water Supplies
DQOs	Data Quality Objectives
EPA	Environmental Protection Agency
EPG	Environmental Protection Geologist
FD	Field Duplicate
FRB	Field Reagent Blank
g/L	Grams per Liter
GWDWB	Ground Water and Drinking Water Branch
GWS	Groundwater Section

HDPE	High-Density Polyethylene
HFPO-DA	Hexafluoropropylene oxide dimer acid; Perfluoro-2-propoxypropanoic acid (GenX)
IEPA	Illinois Environmental Protection Agency
IS	Internal Standard
LC	Liquid Chromatography
LCMRL	Lowest Concentration Minimum Reporting Level
LC/MS/MS	Liquid Chromatography/Mass Spectroscopy/Mass Spectroscopy
LDPE	Low-Density Polyethylene
LFB	Laboratory Fortified Blank
LFSM	Laboratory Fortified Sample Matrix
LFSMD	Laboratory Fortified Sample Matrix Duplicate
LSASD	Laboratory Services and Applied Sciences Division
MDL	Method Detection Limit
mL	Milliliters
MRL	Minimum Reporting Level
MS	Mass Spectrometer
NELAC	National Environmental Laboratory Accreditation Conference
NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid
ng/L	Nanograms per Liter
NIST	National Institute for Standards and Technology
NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid
PFAS	Per- and Polyfluoroalkyl Substances
PFBS	Perfluorobutanesulfonic acid
PFDA	Perfluorodecanoic acid
PFDoA	Perfluorododecanoic acid

PFHpA	Perfluoroheptanoic acid
PFHxA	Perfluorohexanoic acid
PFHxS	Perfluorohexanesulfonic acid
PFNA	Perfluorononanoic acid
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctanesulfonic acid
PFTA	Perfluorotetradecanoic acid
PFTrDA	Perfluorotridecanoic acid
PFUnA	Perfluoroundecanoic acid
ppt	Parts per Trillion
PVC	Polyvinyl Chloride
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QMP	Quality Management Plan
RPD	Relative Percent Difference
SDVB	Polystyrenedivinylbenzene
SDWIS	Safe Drinking Water Information System
SOP	Standard Operating Procedure
SPE	Solid Phase Extraction
SQAB	Science and Quality Assurance Branch
STPSB	State and Tribal Program and Support Branch
TAT	Turn-Around-Time
µg/L	Micrograms per Liter

µL	Microliters
U.S. EPA	United States Environmental Protection Agency
USGS	United States Geological Survey

A3 Distribution List

A copy of this Quality Assurance Project Plan (QAPP) will be distributed in electronic format via e-mail to each person signing the approval sheet and to the individuals listed in Section A4 Project/Task Organization. Individuals participating in this study may request additional copies of the QAPP from personnel listed in Section A4.

An electronic copy of the QAPP will be posted on the Illinois Environmental Protection Agency (EPA) Intranet system via the Illinois EPA Portal: Illinois EPA Compass > SharePoint Site Directory > Bureau of Water > Bureau of Water Quality System > BOW Quality Assurance Project Plans.

The original approved QAPP containing the Approval Sheet with signatures and dates will be retained by the Illinois EPA Bureau of Water (BOW) Quality Assurance Officer (QAO).

This document has been prepared according to the United States Environmental Protection Agency (U.S. EPA) publication *EPA Requirements for Quality Assurance Project Plans (QA/R-5)* (May 2001).

A4 Project/Task Organization

An organizational chart illustrating the Per- and Polyfluoroalkyl Substances (PFAS) Sampling in Community Water Supplies study group hierarchy is presented in Figure 1. (Table 1 contains contact information for the PFAS study group.)

The following individuals are responsible for the project management:

U.S. EPA Technical Lead – Kim Harris, Technical Lead, U.S. EPA Region 5, Ground Water and Drinking Water Branch (GWDWB), Water Division

U.S. EPA Project Officer – Katharine Marko, Project Officer, U.S. EPA Region 5, State and Tribal Program and Support Branch (STPSB), Water Division

U.S. EPA QA Manager – Louis J. Blume, Physical Scientist, U.S. EPA Region 5, Science and Quality Assurance Branch (SQAB), Laboratory Services and Applied Sciences Division (LSASD)

Study Director/Manager –

Rick Cobb, P.G., Acting Division Manager, Illinois EPA, Headquarters, Division of Public Water Supplies (DPWS), BOW.

Michael Summers, P.G., Groundwater Section (GWS) Manager, Illinois EPA, Headquarters, DPWS, BOW.

Network Design Coordinator – Anthony Dulka, P.G., Planning & Assessment Unit Manager, Illinois EPA, Headquarters, GWS, DPWS, BOW.

Quality Assurance Officer – Michelle Rousey, QAO, Illinois EPA, BOW.

Data Coordinator – Alan Fuhrmann, EPG III, Illinois EPA, Headquarters, GWS, DPWS, BOW.

Compliance Assurance Assistant – Connie Sullinger, Retired, Illinois EPA, Toxicity Assessment Unit.

Sample Collectors –

Greg White, P.G., Environmental Protection Specialist III, Illinois EPA, Rockford Regional Office, GWS, DPWS, BOW.

Ryan Bennett, Environmental Protection Geologist (EPG) III, Illinois EPA, Headquarters, GWS, DPWS, BOW.

Alan Fuhrmann, EPG III, Illinois EPA, Headquarters, GWS, DPWS, BOW.

Kari Beckum, EPG I, Illinois EPA, Headquarters, GWS, DPWS, BOW.

Karen Bridges, EPG I, Illinois EPA, Headquarters, GWS, DPWS, BOW.

Logan Schippert, Environmental Protection Associate, Illinois EPA, Headquarters, Springfield Regional Office, Surface Water Section, BOW.

Drew Podlewski, Environmental Protection Specialist, Illinois EPA, Headquarters, Watershed Management Section, BOW.

Bruce Wilkinson, Retired, U.S. EPA Region 5, Pesticides and Toxics Compliance Section.

Dan Hopkins, Retired, U.S. EPA Region 5, Pesticides Section, Land and Chemicals Division.

Bill Morrow, Retired Hydrologist, United States Geological Survey (USGS).

Patrick Mills, Retired Hydrologist, USGS.

Eurofins Eaton Analytical, LLC:

Matthew Hartz, Laboratory Director

Bill Reeves, Laboratory Quality Assurance Officer

The U.S. EPA Technical Lead is responsible for assisting with the QAPP/Standard Operating Procedure (SOP) review and advising on sampling protocol and data reports.

The U.S. EPA Project Officer is responsible for managing the grant and reviews and coordinates the Quality Assurance and technical documents.

The U.S. EPA QA Manager is responsible for reviewing and approving the QAPP.

The Study Director/Manager is responsible for oversight of the entire project including design, funding, implementation, data receipt and review, reporting, and preparation of the QAPP.

The Network Design Coordinator, in consultation with the Study Director/Manager, is responsible for the design of the network and selection of sampling sites. The Network Design Coordinator, along with the QAO, will ensure any additional Sample Collectors receive sample collection training prior to collecting samples.

The QAO, in consultation with the Study Director/Manager, is responsible for oversight of quality control (QC) for the project and will assist in preparing the QAPP. The QAO will review and approve this QAPP as meeting the QAPP requirements in U.S. EPA's publication QA/R-5. The QAO, in consultation with the Network Design Coordinator, will provide and document sample collection training. The QAO will conduct audits as deemed necessary.

The Data Coordinator is responsible for reviewing the laboratory data.

The Compliance Assurance Assistant, in consultation with the Data Coordinator, is responsible for processing and tracking monitoring results.

The Sample Collectors are responsible for coordinating the execution of field activities.

The Laboratory Director is responsible for the preparation, analysis, data generation, and data reporting of the environmental samples for the PFAS parameters.

The Laboratory QAO will oversee the laboratory's internal quality assurance/quality control (QA/QC) program.

A5 Problem Definition and Background

Perfluorooctane Sulfonate (PFOS) and Perfluorooctanoic Acid (PFOA) are fluorinated organic chemicals that are part of a larger group of chemicals referred to as Per- and Polyfluoroalkyl Substances (PFAS). These chemicals are manmade and do not occur naturally in the environment. They have been used in non-stick cookware, water-repellent clothing, stain resistant fabrics and carpets, some cosmetics, some firefighting foams, and products that resist grease, water, and oil. Some PFAS do not break down easily and persist for a long time in the environment, especially in water. Their toxicity and persistence in the environment indicate they are a potential danger to public health and the environment. PFOS and PFOA have been the most extensively produced and studied of these chemicals. The U.S. EPA finalized a lifetime Health Advisory (HA) of 0.07 micrograms per liter (µg/L) for both PFOS and PFOA in May 2016 (see Appendices A, B, and C). Even though U.S. EPA has not proposed Maximum Contaminant Levels for PFOS and PFOA, the final Health Advisory recommended actions for drinking water systems if water sampling results confirm PFOS and PFOA concentrations are greater than 70 parts per trillion (ppt).

A6 Project/Task Description

Recognizing the national prevalence of these emerging contaminants of concern (AECOM, 2019 and KDEP, 2019), the Illinois EPA will sample 1,455 active entry points to the distribution system providing drinking water at all 1,749 Community Water Supplies (CWS) (includes purchase systems) in Illinois for PFAS contaminants. When initial sampling results indicate a detection of PFAS contaminants at or above the minimum reporting level, follow-up confirmation samples will be collected from entry points to the distribution system as soon as possible.

Illinois EPA will collect up to an estimated 5000 samples to assess finished drinking water collected from CWS. The samples will be collected following procedures outlined in Section B2 - Sampling Methods. Collected samples will be transported to Eurofins Eaton Analytical, LLC as stated in Section B3 - Sample Handling and Custody. The laboratory will analyze the samples for 18 PFAS contaminants using U.S. EPA Method 537.1. Table 2 contains the PFAS study analytes, acronyms, and Chemical Abstract Services Registry Numbers (CASRNs).

Eurofins Eaton Analytical, LLC is a National Environmental Laboratory Accreditation Conference (NELAC)-certified laboratory holding accreditation for drinking water analysis of the PFAS contaminants listed above with minimum reporting levels (MRLs) of 2 - 14 parts per trillion (preferably lower as achievable by the laboratory)[1]. If any field sample has a level of PFAS detected at or above the MRL, the accompanying field reagent blank (FRB) will be analyzed.

The NELAC Institute's Mission Statement:

> The NELAC Institute (TNI) is a 501(c)(3) non-profit organization whose mission is to foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community. The organization is managed by a Board of Directors and is governed by organizational Bylaws.
>
> TNI's vision is a true national accreditation program, whereby all entities involved in the generation of environmental measurement data within the United States are accredited to one uniform, rigorous, and robust program that has been implemented consistently nationwide and focuses on the technical competence of the entity pursuing accreditation. TNI believes such a program will improve the quality and reliability of environmental data used by federal and state agencies. (https://nelac-institute.org/content/aboutus.php)

Eurofins Eaton Analytical, LLC in South Bend, Indiana has NELAC accreditation for PFAS analysis in drinking water by U.S. EPA Method 537.1. The laboratory is accredited by the Oregon Environmental Laboratory Accreditation Program (which is a NELAP Recognized Accreditation Body) as the Primary Accrediting Authority (Appendix D).

[1] Per Method 537.1 Section 1.2, Minimum Reporting Level (MRL) is the lowest analyte concentration that meets Data Quality Objectives (DQOs) that are developed based on the intended use of this method. The single laboratory lowest concentration MRL (LCMRL) is the lowest true concentration for which the future recovery is predicted to fall, with high confidence (99%), between 50 and 150% recovery. Single laboratory LCMRLs for analytes in this method range from 0.53-6.3 ng/L, and are listed in Table 5 of Method 537.1.

The Illinois EPA BOW QAO, in consultation with the Network Design Coordinator and cooperation with Eurofins Eaton Analytical, LLC, will plan the logistics and training on proper sample containers, volumes, parameters, method, preservatives, holding times, and QA/QC for PFAS sample collection and transportation.

As part of this initial statewide assessment, monitoring results will be made available to U.S. EPA for review via upload into the Safe Drinking Water Information System (SDWIS) database.

A7 Quality Objectives and Criteria

A summary of the minimum measurement criteria and data quality objectives (DQOs) for samples analyzed by Eurofins Eaton Analytical, LLC are presented in Table 3.

A7.1 Precision

Precision is a measure of agreement among repeated measurements of the same property under identical, or substantially similar, conditions; calculated as either the range or as the standard deviation. Precision may also be expressed as a percentage of the mean of the measurements, such as relative range or relative standard deviation (coefficient of variation).

Precision will be measured in the laboratory during the analysis of field duplicate (FD) samples (FD1 and FD2) and laboratory fortified sample matrix (LFSM)/LFSM duplicate (LFSMD) samples. The frequency of the analysis will be one set of field duplicates or LFSM/LFSMD per batch of 20 environmental samples.

The analyses of the duplicate samples are considered acceptable if the calculated relative percent difference (RPD) of the measurements is within the acceptance limits listed in Table 3. If the RPD falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the Continuing Calibration Checks (CCC), the recovery is judged matrix biased. The results for the sample are labeled as suspect based on poor RPD values to inform the data user that the results are suspect.

The results of the duplicate analyses are used to calculate the RPD for evaluating precision using the following formula:

$$RPD = [(A - B) / (A + B)/2] * 100$$

where

A = Original sample concentration

B = Duplicate sample concentration

A7.2 Bias

Bias is the systematic or persistent distortion of a measurement process that causes errors in one direction.

Analytical bias will be assessed in the laboratory during the analysis of LFSM samples. The frequency of the analysis will be one per batch of 20 environmental samples.

The analyses of the LFSM samples are considered acceptable if the calculated percent recovery of the measurements is within the acceptance limits listed in Table 3. If the results are outside of the designated recovery range, and the recovery for that analyte is shown to be in control in the CCCs, the recovery problem for the LFSM is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect based on poor recoveries in the LFSM to inform the data user that the results are suspect.

The results of the spiked samples are used to calculate the percent recovery using the following formula:

Percent Recovery = [(S - U) / T] * 100

Where

S = Spiked sample concentration

U = Unspiked sample concentration

T = True spike concentration

A7.3 Accuracy

Accuracy is a measure of the overall agreement of a measurement to a known value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations.

Error due to sampling will be assessed during the analysis of sample duplicates (Section A7.1) and field reagent blanks (Section A7.4).

Accuracy will be measured during the analysis of environmental water by using Laboratory Fortified Blanks (LFBs) and LFSM/LFSMDs. LFBs are prepared in the laboratory by fortifying (or spiking) samples of deionized water with the analytes of interest. These LFBs will be analyzed with each batch of water extracts, at a frequency of one per 20 environmental samples. LFSM/LFSMDs are prepared in the laboratory by fortifying (or spiking) field samples with the analytes of interest. LFSM/LFSMDs will be analyzed at a frequency of one per 20 environmental samples. The analyses of the LFBs and LFSM/LFSMDs are considered acceptable if the calculation concentrations for all analytes of interest are within the acceptance limits listed in Table 3. If the criteria are not met for the LFB, then

all data must be considered invalid for samples in the extraction batch. Corrective actions must be taken. The analyst should reanalyze the LFB as part of the same or a new analysis batch after the problems have been resolved. If the reanalysis meets the LFB criteria, report only the reanalysis data. If the LFSM/LFSMD results are outside of the designated recovery range, and the recovery for that analyte is shown to be in control in the CCCs, the recovery problem for the LFSM is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect based on poor recoveries in the LFSM to inform the data user that the results are suspect.

The results of the spiked samples are used to calculate the percent recovery for evaluating accuracy using the following formula:

$$\text{Percent Recovery} = [(S - U) / T] * 100$$

Where

S = Spiked sample concentration

U = Unspiked sample concentration

T = True spike concentration

A7.4 Representativeness

Representativeness is the measure of the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Representativeness of data will be ensured using established field and laboratory procedures and their consistent application. To aid in the evaluation of the representativeness of the sample data, field reagent blanks (FRBs) will be evaluated for the presence of contaminants.

One FRB per sampling site will be collected and sent to the laboratory along with the accompanying site samples. The laboratory is responsible for deciding whether to analyze the FRB based on the following criteria:

> Per U.S. EPA Method 537.1 Section 9.3.8, the analysis of the FRB is required only if a field sample contains a method analyte or analytes at or above the minimum reporting level (MRL). If the method analyte(s) found in the field sample is present in the FRB at a concentration greater than 1/3 the MRL, then all samples collected with that FRB are invalid and must be recollected and reanalyzed.

The FRB results will also be reviewed by the Data Coordinator to evaluate the effectiveness of sample collection procedures in avoiding PFAS contamination. If a pattern of reoccurring contamination is found, the sample collection procedures will be reevaluated and revised if necessary.

A7.5 Comparability

Comparability is a measure of the confidence with which one data set or method can be compared to another.

Comparability will be maximized by using standard analytical methods and standardized, documented sampling techniques. Documentation will include all sampling locations, conditions, and field sampling methods. All results will be reported in standard units or, for field parameters, as defined in the method. All laboratory calibrations will be performed using standards traceable to the National Institute for Standards and Technology (NIST) or another certified reference standard source.

A7.6 Completeness

Completeness is a measure of the amount of valid data needed to be obtained from a measurement system.

The percent completeness is calculated by dividing the number of valid sample results by the total number of samples planned and multiplying the result by 100 percent. Completeness will be reported as the percentage of all measurements judged valid. The following equation will be used to determine completeness:

Percent Completeness = (V/T) * 100

where

V = Valid number of sample results

T = Total number of samples planned

For this project, the QA objective for degree of completeness for both field and laboratory data is 90 percent. If completeness is less than the target of 90 percent, the Study Director/Manager and QAO will evaluate the data to determine whether there are enough data to complete the study or if additional data collection is necessary.

A7.7 Sensitivity

Sensitivity is the capability of a method or instrument to discriminate between measurement responses representing different levels of the variable of interest. The intent of this project is to employ methods of measurements that detect and quantify all analytes of interest wherever possible. The Minimum Reporting Level (MRL), also known as reporting limit, has been established at the lowest level of quantitation (lowest analyte concentration) achievable by the laboratory.

Although there are many intended and potential uses of the data, minimum measurement criteria are typically established at the lowest analyte concentration required for planned uses of the measurement data. Since no minimum measurement criteria can be identified, the water samples will be analyzed to the lowest concentration MRL for each PFAS analyte achievable by the Eurofins Eaton Analytical, LLC.

The monitored parameters, MRLs, MDLs, and QC acceptance criteria are shown in Table 3.

The minimum measurement objective for any analyte will be achieved when the analytical procedure selected for sample analysis can be shown to have a Method Detection Limit (MDL) at or below the minimum measurement objective. Table 3 compares the minimum measurement objective against the MDL achieved by the laboratory. All analytes meet the minimum measurement objective.

Per U.S. EPA Method 537.1 Section 1.4, the detection limit (DL) is defined as "the statistically calculated minimum concentration that can be measured with 99% confidence that the reported value is greater than zero. The DL is compound dependent and is dependent on extraction efficiency, sample matrix, fortification concentration, and instrument performance."

Analyte MDLs shall be determined by the Procedure for the Determination of the Method Detection Limit – Revision 2 given in the August 28, 2017 Code of Federal Regulations (CFR), Volume 40, Part 136, Appendix B. The MDL is defined as "the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results." Since the MDL procedure is based upon precision obtained for a standard greater than the MDL, it also is a measure of method sensitivity at concentrations near the MDL. Eurofins Eaton Analytical, LLC will determine MDLs by following the updated 40 CFR Part 136 Appendix B and the October 2017 memo from the U.S. EPA Office of Ground Water and Drinking Water, Technical Support Center. After the initial MDL determination, quarterly spikes will be analyzed and every 13 months put together and compared to the blank data to determine the new MDL for each analyte.

A8 Special Training/Certifications

Prior to sample collection, all Sample Collectors shall be trained in the proper sample collection procedures. PFAS sample collection shall follow U.S. EPA Method 537.1 Section 8 Sample Collection, Preservation, and Storage (Appendix E) and Illinois EPA's SOP for Sample Collection of PFAS at CWS in Illinois (Appendix F). The Illinois EPA BOW QAO, in consultation with the Network Design Coordinator, will provide and document sample collection training. Documentation of training will be in the form of training attendance sheet(s) or electronic acknowledgement of training. The training documentation will be retained with the sample collection records in Section A9. Any additional Sample Collectors added to this project will receive sample collection training prior to collecting samples.

A9 Documentation and Records

Sample collection training documentation and sample collection records of all field activities shall be retained in the permanent record. The Sample Collectors are responsible for coordinating the execution of those field activities. Sample collection records shall document proper sampling protocol performed in the field.

The Study Director/Manager shall retain all laboratory analytical results with accompanying Chain-of-Custody sheets (Appendix F) and all correspondence with Eurofins Eaton Analytical, LLC.

All records will be archived with the Agency's Division of Records Management, following their protocols, and maintained in a secure location at the Agency Headquarters in Springfield.

The QAO shall be made aware of any problems encountered during any phase of the project.

SECTION B. DATA GENERATION AND ACQUISITION

B1 Sampling Process Design (Experimental Design)

The Network Design Coordinator is responsible for the design of the network and selection of sampling sites. The network has been designed to collect PFAS samples from all active entry points to the distribution systems of "parent" community water supplies in the state as efficiently as possible given limited resources. The Network Design Coordinator will maintain the list of sampling sites and track sample collection progress. Further, the Network Design Coordinator will be responsible for addressing site-specific sample collection issues/problems as they arise and keep sample collection staff on schedule for project completion.

Samples for analysis of PFAS will be collected using Eurofins Eaton Analytical, LLC sample collection kits. PFAS sample collection shall follow U.S. EPA Method 537.1 Section 8 Sample Collection, Preservation, and Storage (Appendix E) and Illinois EPA's SOP for Sample Collection of PFAS at CWS in Illinois (Appendix F).

PFAS samples will be shipped via FedEx or UPS overnight priority using the forms provided by Eurofins Eaton Analytical, LLC.

B2 Sampling Methods

PFAS samples will be collected following U.S. EPA Method 537.1 Section 8 Sample Collection, Preservation, and Storage (Appendix E) and Illinois EPA's SOP for Sample Collection of PFAS at CWS in Illinois (Appendix F). Sample containers, volumes, parameters, methods, preservatives, and holding times are summarized in Table 4.

Table 5 contains a summary of prohibited and acceptable items for sampling PFAS.

The PFAS samples will be analyzed by Eurofins Eaton Analytical, LLC.

Refer to Section B5.1 Field Quality Control for individuals responsible for corrective action.

B3 Sample Handling and Custody

All samples are to be collected, preserved, stored and transported to the laboratory within the prescribed holding times as described in Illinois EPA's SOP for Sample Collection of PFAS at CWS in Illinois (Appendix F).

All samples are to be accompanied by completed Chain-of-Custody Sheets (Appendix G).

All samples will be iced or refrigerated to chill them prior to shipping to the laboratory. Blue ice or chemical ice packs are prohibited.

The laboratory shall record sample temperature upon arrival at the laboratory using a thermometer calibrated against a NIST-traceable certified thermometer. Samples requiring thermal preservation are refrigerated after sample acceptance at the laboratory.

When received by the laboratory, the samples are logged into the laboratory logbook and/or laboratory database. Maximum holding times before sample preparation and analysis, as stated in applicable methods and SOPs, are followed.

Sample disposal shall follow the procedures stated in the laboratory's Quality Manual.

Refer to Section B5.2 Laboratory Quality Control for individuals responsible for corrective action.

B4 Analytical Methods

Eurofins Eaton Analytical, LLC shall use U.S. EPA Method 537.1 (Appendix H) approved for drinking water for the preparation and analysis of PFAS samples. The Eurofins Eaton Analytical, LLC SOP for PFAS Extraction and Analysis following this method is in Appendix I.

A summary of the method, per U.S. EPA Method 537.1 Section 2:

> A 250-milliliter (mL) water sample is fortified with surrogates and passed through a solid phase extraction (SPE) cartridge containing polystyrenedivinylbenzene (SDVB) to extract the method analytes and surrogates. The compounds are eluted from the solid phase sorbent with a small amount of methanol. The extract is concentrated to dryness with nitrogen in a heated water bath, and then adjusted to a 1-mL volume with 96:4% (vol/vol) methanol:water and addition of the internal standards. A 10-µL injection is made into a liquid chromatography (LC) equipped with a C_{18} column

that is interfaced to a mass spectrometer (MS)/MS. The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under identical LC/MS/MS conditions. The concentration of each analyte is determined by using the internal standard technique. Surrogate analytes are added to all Field and QC Samples to monitor the extraction efficiency of the method analytes.

The PFAS analytical method and laboratory SOP are listed in Table 6.

The PFAS turn-around-time (TAT) from sample receipt at the laboratory to reporting of the sample results is 30 days, unless the Illinois EPA has requested an accelerated TAT of 14, 7, or an emergency response time of 24 hours.

B5 Quality Control

B5.1 Field Quality Control

All Sample Collectors are responsible for ensuring proper sampling methods, sample preservation, and sample custody of the delivered samples to the designated laboratory are followed.

An investigation and corrective action report (CAR) prepared by the Network Design Coordinator in the event of a QC issue will be submitted to the Study Director/Manager. The Study Director/Manager will then forward this report to the QAO.

The accuracy and precision of all data measurements must be quantifiable. Analytical procedures used for data analysis must be performed in accordance with approved methods. Data measurements should be recorded in a controlled environment in which a QC program is maintained.

B5.2 Laboratory Quality Control

Eurofins Eaton Analytical, LLC is responsible for implementing their QA/QC Manual which is an internal QA plan for laboratory procedures. The laboratory is responsible for the accuracy and reliability of analytical methods and final data reports according to their QA/QC Manual.

An investigation and CAR will be submitted to the Study Director/Manager and the QAO as QC issues arise. The laboratory is responsible for providing data qualifiers and/or case narratives to inform the Study Director/Manager and the QAO of any analytical exceptions that fall outside of routine method protocols. The laboratory's QA/QC Manual will contain the procedures for QC and for calculating QC statistics.

Table 7 contains the laboratory QC frequency and acceptance criteria for each parameter.

Appendix I contains the Eurofins Eaton Analytical, LLC SOP for PFAS Extraction and Analysis. Linear or quadratic calibration curves including a minimum of five points are generated for the analytes using peak area and an internal standard technique. Linear calibration curves including the origin and multiple points at a single concentration level are generated for the surrogate and internal standards using peak areas. The surrogate standards use an internal standard technique. The internal standards use an external calibration technique. Per the laboratory SOP, the laboratory QC with frequency and acceptance criteria are as follows:

Initial Calibration –

- Concentration levels are 2.0, 10, 50, 100, 200, and 250 ng/L.
- A minimum of five calibration points (six for quadratic curves per 2016 TNI Standard), the lowest must be at or lower than the MRL.
- Run with each analysis batch.
- Analyte recoveries must be within 50-150% for points at or below the MRL and within 70-130% for other points.

Continuing Calibration Check (CCC) –

- Concentration levels are CC Low (CCL) (2.0 ng/L), CC Mid (CCM) (100 ng/L), or CC High (CCH) (200 ng/L).
- A CCL must be analyzed immediately after the initial calibration curve, prior to any QC or samples. Rotate CCM and CCH after every tenth field sample and at the end of each analysis batch.
- Recovery must be within 50-150% of the true value for CCL and within 70-130% for CCM and CCH.

Internal Standard (IS) –

- Concentration is 40 ng/L in terms of PFOA-$^{13}C_2$.
- Introduce into every field sample, calibration, and QC sample.
- IS area counts must be 70-140% of the response in the most recent previous CCC and 50-150% of the average IS area count from the initial calibration.

Surrogate –

- Concentration is 40 ng/L in terms of PFHxA-$^{13}C_2$.
- Introduce into every field sample, calibration, and QC sample.
- Surrogate recovery must be within 70-130% of the target.

Appendix J contains the Method 537.1 Table 12 – Initial Demonstration of Capability Quality Control Requirements.

Appendix K contains the Method 537.1 Table 12 – Ongoing Quality Control Requirements (Summary).

B6 Instrument/Equipment Testing, Inspection, and Maintenance

All laboratory equipment shall be routinely maintained according to the manufacturer's manuals. Any equipment used for field data measurements shall be tested and inspected prior to sampling events.

An adequate supply of spare parts shall be maintained by the Sample Collectors (with oversight by the Network Design Coordinator) and the laboratory for equipment maintenance. Spare parts shall be routinely inventoried.

B7 Instrument/Equipment Calibration and Frequency

Instruments used in the field and in the laboratory shall be calibrated prior to use according to the manufacturer's manual. The laboratory shall calibrate instruments and equipment according to their internal QA plan.

The laboratory shall use Certified Reference Material standards with Certificate of Analysis documentation from a vendor accredited to ISO17034.

The laboratory's initial calibration shall be a minimum of five calibration points (six for quadratic curves per 2016 TNI Standard), the lowest must be at or lower than the MRL. The concentration levels are 2.0, 10, 50, 100, 200, and 250 ng/L. The initial calibration is run with each analysis batch. The acceptance criteria are as follows: Analyte recoveries must be within 50-150% for points at or below the MRL and within 70-130% for other points.

B8 Inspection/Acceptance of Supplies and Consumables

Supplies and consumables used in the field shall be inspected by the Sample Collectors to guarantee their usability. Supplies and consumables used in the laboratory shall be inspected by the laboratory director to confirm compliance with the laboratory Quality Management Plan (QMP) and SOPs.

B9 Non-direct Measurements

Non-direct measurements are not required for this study.

B10 Data Management

Logbooks, field measurement records, and other data gathered in the field (i.e., chain-of-custody sheets) shall be maintained in the permanent record. All records will be archived with the Agency's Division of Records Management, following their protocols, and maintained in a secure location at the Agency Headquarters in Springfield.

The laboratory shall convey all laboratory analytical data by providing hard and electronic copies of analytical samples results to the Study Director/Manager. The laboratory will also submit analytical data via electronic data deliverables into SDWIS. All data communicated to the Illinois EPA shall be verified by the Data Coordinator for reliability and the Study Director/Manager for usability.

Electronic data sent from the laboratory will be automatically uploaded into SDWIS. The electronic results will be compared by the Data Coordinator to the hardcopy results to confirm that the data has been uploaded and is correct. For electronic data that has not been successfully uploaded, the Data Coordinator will work with the laboratory to resolve the issue. The corrected data will be resent by the laboratory to SDWIS. Minor changes to data can be made manually in SDWIS, and all such changes will be reviewed by the Data Coordinator.

The laboratory shall provide an alert of any preliminary PFAS detection to the Illinois EPA via email. Initial detections of PFAS will be resampled by the Sample Collectors; these confirmation sample results will be delivered to SDWIS.

All sample results and associated information (such as case narrative, original Chain of Custody forms, quality control results, and data qualifiers) shall be provided by the laboratory to the Illinois EPA via email.

The PFAS TAT from sample receipt at the laboratory to reporting of the sample results is 30 days, unless the Illinois EPA has requested an accelerated TAT of 14, 7, or an emergency response time of 24 hours.

SECTION C. ASSESSMENT AND OVERSIGHT

C1 Assessments and Response Actions

Field audits will be performed by the QAO to evaluate the Sample Collectors' adherence to this QAPP, U.S. EPA Method 537.1 Section 8 Sample Collection, Preservation, and Storage (Appendix E) and Illinois EPA's SOP for Sample Collection of PFAS at CWS in Illinois (Appendix F). The frequency of field audits will be at least one per each Sample Collector during the project. Additional field audits will be conducted if deemed necessary. The field audits will be documented and submitted to the Study Director/Manager.

QC issues related to field activities will require an investigation and corrective action plan submitted to the Study Director/Manager and QAO.

The laboratory involved in data analysis shall maintain an internal QA program described in their QMP. The laboratory shall maintain QC checks for procedures. When the possibility of QC problems arise that may affect the usability of data, an investigation and CAR will be submitted by the Laboratory Director to the Study Director/Manager and reviewed by the QAO.

Also, the Study Director/Manager shall make certain that the project data associated with any quality control or other nonconformance issue is made available to data users with the appropriate data qualification. When data previously released to data users may have been affected by a quality control problem or other nonconformance issue, the Study Director/Manager shall notify other data users of the problem.

C2 Reports to Management

The Study Director/Manager, in coordination with the Network Design Coordinator, Quality Assurance Officer, Data Coordinator, and Compliance Assurance Assistant, will prepare monthly project status reports and distribute to management and U.S. EPA.

The Study Director/Manager will receive investigation and CARs in case of any QC issue and will forward these reports to the QAO. Reports shall be prepared by the QAO documenting the QC issue, the corrective action taken, and the outcome of the corrective action and how it impacts the data.

Any QA problems affecting the final reported values shall be reported to the Study Director/Manager and any other data users.

SECTION D. DATA VALIDATION AND USABILITY

D1 Data Review, Verification, and Validation

The Study Director/Manager and the Data Coordinator will review final analytical reports and address any issue related to data reliability as mentioned in pertinent investigation and correction action plans. Laboratory results with accompanying data qualifiers will be listed as such in any reports or data submitted to the Study Director/Manager. It will be the responsibility of the Study Director/Manager to determine the usability of any qualified data.

D2 Verification and Validation Methods

Sample collection and field measurement records shall be verified by Sample Collectors and sent to the Study Director/Manager. Laboratory data shall be verified by the Laboratory Director. Field and laboratory records will

be archived with the Agency's Division of Records Management, following their protocols, and maintained in a secure location at the Agency Headquarters in Springfield.

In the case of data verification resulting in a change to data, the Study Director/Manager shall document any corrections.

The Study Director/Manager shall be informed if data accuracy, reliability, or usability has been reduced as the result of errors in stored data or corrupted data files. The Study Director/Manager shall make any necessary corrections to the data and document the reasons the corrections were made.

D3 Reconciliation with User Requirements

The Study Director/Manager shall review data and its usability and determine if it meets the requirements of the study objectives as stated in Section A5, Problem Definition and Background.

The execution of the study shall follow the procedures outlined in this QAPP. Personnel listed in Section A4, Project/Task Organization are responsible for implementation of the QC measures during each stage of the project.

The QAPP shall be reviewed by all persons listed on the approval page. The review shall determine issues to be addressed as the project progresses. Issues to be discussed may include:

1. The number and location of sampling stations.
2. The frequency of sampling.
3. Sampling procedures.
4. Parameters measured.
5. Data quality objectives and minimum measurement criteria.
6. Analytical procedures.
7. Project reporting.
8. Corrective actions taken.

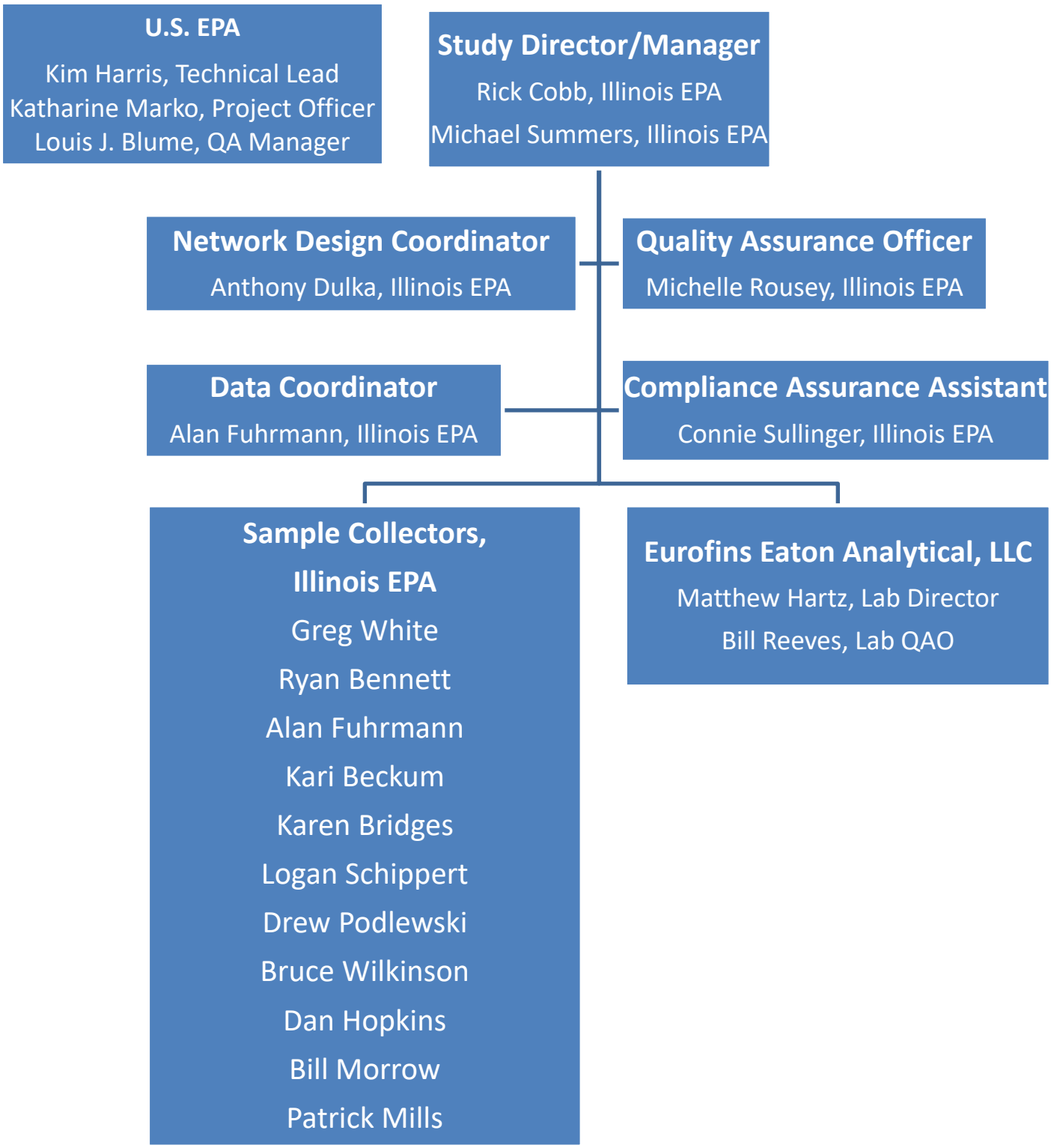
The study shall be modified only as directed by the Study Director/Manager. Changes in procedures shall not be made without the approval of the Study Director/Manager. All changes shall be documented in a memorandum that will be distributed to those listed on the Approval Sheet.

The QAO shall update the QAPP after review and keep a separate record of changes.

FIGURES

Figure 1 – Study Organizational Chart

Figure 1 – Study Organizational Chart



TABLES

Table 1 – Study Contact Information

Name	Title	Affiliation	Phone Number	E-Mail	Project Role
Kim Harris	Region 5, GWDWB, Water Division Technical Lead	U.S. EPA	(312) 886-4239	Harris.Kimberly@epa.gov	U.S. EPA Technical Lead
Katharine Marko	Region 5, STPSB, Water Division, Project Officer	U.S. EPA	(312) 885-1473	Marko.Katharine@epa.gov	U.S. EPA Project Officer
Louis J. Blume	Region 5, SQAB, LSASD, Physical Scientist	U.S. EPA	(312) 353-2317	Blume.Louis@epa.gov	U.S. EPA QA Manager
Rick Cobb	BOW, DPWS Deputy Division Manager	Illinois EPA	(217) 524-5377	Rick.Cobb@illinois.gov	Study Director/Manager
Michael Summers	BOW, DPWS, GWS Manager	Illinois EPA	(217) 557-8086	Michael.Summers@illinois.gov	Study Director/Manager
Anthony Dulka	BOW, DPWS, GWS Planning & Assessment Unit Supervisor	Illinois EPA	(217) 524-7923	Anthony.Dulka@illinois.gov	Network Design Coordinator
Michelle Rousey	BOW Quality Assurance Officer	Illinois EPA	(217) 785-3944	Michelle.Rousey@illinois.gov	Quality Assurance Officer
Alan Fuhrmann	BOW, DPWS, GWS Env. Prot. Geologist	Illinois EPA	(217) 557-3179	Alan.Fuhrmann@illinois.gov	Sample Collector/ Data Coordinator
Connie Sullinger	Toxicity Assessment Unit	Retired, Illinois EPA	(217) 782-1020	Connie.Sullinger@gmail.com	Compliance Assurance Assistant
Greg White	BOW, DPWS, GWS Env. Prot. Specialist	Illinois EPA	(815) 987-7760	Gregory.White@illinois.gov	Sample Collector

Table 1 – Study Contact Information (Cont.)

Name	Title	Affiliation	Phone Number	E-Mail	Project Role
Ryan Bennett	BOW, DPWS, GWS Env. Prot. Geologist	Illinois EPA	(217) 524-8114	Ryan.Bennett@illinois.gov	Sample Collector
Kari Beckum	BOW, DPWS, GWS Env. Prot. Geologist	Illinois EPA	(217) 558-6187	Kari.Beckum@illinois.gov	Sample Collector
Karen Bridges	BOW, DPWS, GWS Env. Prot. Geologist	Illinois EPA	(217) 782-1020	Karen.Bridges@illinois.gov	Sample Collector
Logan Schippert	BOW, DWPC, SWS Env. Prot. Associate	Illinois EPA	(217) 785-9949	Logan.Schippert@illinois.gov	Sample Collector
Drew Podlewski	BOW, DWPC, WMS Env. Prot. Specialist	Illinois EPA	(217) 558-0416	Drew.Podlewski@illinois.gov	Sample Collector
Bruce Wilkinson	Pesticides and Toxics Compliance	Retired, U.S. EPA Region 5	(847) 778-8332	bwwilkinson56@gmail.com	Contract Sample Collector
Dan Hopkins	Pesticides Section	Retired, U.S. EPA Region 5	(773) 401-8822	hopkins.danj@gmail.com	Contract Sample Collector
Bill Morrow	Retired Hydrologist	USGS	(217) 898-1440	wsmorrow@mchsi.com	Contract Sample Collector
Patrick Mills	Retired Hydrologist	USGS	(217) 898-9190	pcmills1@comcast.net	Contract Sample Collector
Matthew Hartz	Laboratory Director	Eurofins Eaton Analytical, LLC	(574) 472-5578	matthewhartz@eurofinsUS.com	Laboratory Director
Bill Reeves	Quality Assurance Manager	Eurofins Eaton Analytical, LLC	(574) 472-5568	williamreeves@eurofinsUS.com	Laboratory Quality Assurance Officer

Table 2 – Per- and Polyfluoroalkyl Substances

Analyte[a]	Acronym	Chemical Abstract Services Registry Number (CASRN)
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6[b]
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	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	PFTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnA	2058-94-8
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9[c]
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid	9Cl-PF3ONS	756426-58-1[d]
4,8-dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4[e]

a = Some PFAS are commercially available as ammonium, sodium and potassium salts. This method measures all forms of the analytes as anions while the counterion is inconsequential. Analytes may be purchased as acids or as any of the corresponding salts (see U.S. EPA Method 537.1 Section 7.2.3 regarding correcting the analyte concentration for the salt content).

b = HFPO-DA is one component of the GenX processing aid technology.

c = 11Cl-PF3OUdS is available in salt form (e.g. CASRN of potassium salt is 83329-89-9).

d = 9Cl-PF3ONS analyte is available in salt form (e.g. CASRN of potassium salt is 73606-19-6)

e = ADONA is available as the sodium salt (no CASRN) and the ammonium salt (CASRN is 958445-448).

Table 3 – Minimum Measurement Criteria and Objectives (Eurofins Eaton Analytical, LLC)

Parameter	Minimum Measure-ment Criteria ng/L	Minimum Measure-ment Objectives* ng/L	Minimum Reporting Levels (MRL) ng/L	Method Detection Limit (MDL) ng/L	Accuracy[A] (% recovery) LFB	Accuracy[A] (Matrix) (% recovery) LFSM/LSFMD	Precision[P] (RPD) LFSM/ LFSMD and Duplicates	Complete-ness (%)
HFPO-DA	NA	2	2	0.5	70-130	70-130	< 30%	90
NEtFOSAA	NA	2	2	0.6	70-130	70-130	< 30%	90
NMeFOSAA	NA	2	2	0.5	70-130	70-130	< 30%	90
PFBS	NA	2	2	0.4	70-130	70-130	< 30%	90
PFDA	NA	2	2	0.5	70-130	70-130	< 30%	90
PFDoA	NA	2	2	0.6	70-130	70-130	< 30%	90
PFHpA	NA	2	2	0.4	70-130	70-130	< 30%	90
PFHxS	NA	2	2	0.5	70-130	70-130	< 30%	90
PFHxA	NA	2	2	0.4	70-130	70-130	< 30%	90
PFNA	NA	2	2	0.5	70-130	70-130	< 30%	90
PFOS	NA	2	2	0.4	70-130	70-130	< 30%	90
PFOA	NA	2	2	0.4	70-130	70-130	< 30%	90
PFTA	NA	2	2	0.6	70-130	70-130	< 30%	90
PFTrDA	NA	2	2	0.5	70-130	70-130	< 30%	90
PFUnA	NA	2	2	0.5	70-130	70-130	< 30%	90
11Cl-PF3OUdS	NA	2	2	0.5	70-130	70-130	< 30%	90
9Cl-PF3ONS	NA	2	2	0.5	70-130	70-130	< 30%	90
ADONA	NA	2	2	0.6	70-130	70-130	< 30%	90

* = Minimum Measurement Objectives are set at the Minimum Reporting Level for each PFAS analyte.
A = Refer to Table 7 for low-level accuracy quality control criteria.
P = Refer to Table 7 for low-level precision quality control criteria.
ng/L = parts per trillion (ppt)
NA = Not Applicable
LFB = Laboratory Fortified Blank
LFSM = Laboratory Fortified Sample Matrix
LFSMD = Laboratory Fortified Sample Matrix Duplicate
RPD = Relative Percent Difference

Table 4 – Sample Containers, Volumes, Parameters, Method, Preservatives, and Holding Times

Sample Container (per sample site)	Parameter	Method Number	Preservative	Holding Time
250-mL polypropylene bottles fitted with polypropylene screw-caps, in following quantity: • Two bottles per sample ➢ (One is back-up bottle for lab) • Four bottles total if collecting duplicate samples* • Four bottles additional if requesting LFSM/LFSMD analyzed*	PFAS	U.S. EPA 537.1	Chemical: 5.0 g/L Trizma® Thermal: ≤ 10° C – Collection to receipt at laboratory ≤ 6° C – Storage at laboratory	Collection – Extraction: 14 days Extraction – Analysis: 28 days
Quantity: • One 250-mL polypropylene bottle filled with reagent water and preservative at Eurofins Eaton Analytical, LLC. • One empty (no preservative) 250-mL polypropylene bottle	PFAS Field Reagent Blank	U.S. EPA 537.1	Chemical: 5.0 g/L Trizma® Thermal: ≤ 10° C – Collection to receipt at laboratory ≤ 6° C – Storage at laboratory	Collection – Extraction: 14 days Extraction – Analysis: 28 days

The frequency of collecting additional samples to be used for duplicate and LFSM/LFSMD analysis will be determined by the Network Design Coordinator and the Data Coordinator as sampling sites are assigned to the Sample Collectors.

The PFAS TAT from sample receipt at the laboratory to reporting of the sample results is 30 days, unless the Illinois EPA has requested an accelerated TAT of 14, 7, or an emergency response time of 24 hours.

Table 5 – Summary of Prohibited and Acceptable Items for PFAS Sampling

PROHIBITED ITEMS	ACCEPTABLE ITEMS
FIELD EQUIPMENT ITEMS	
No Teflon™ containing materials	High-density polyethylene (HDPE) and polypropylene (PP) materials
Do not store samples in containers made of LDPE (Low-Density Polyethylene) materials	Acetate liners
No Teflon™ tubing	Silicon tubing
No waterproof field books	Loose paper (non-waterproof)
No plastic clipboards, binders, or spiral hard cover notebooks	Aluminum, polypropylene or Masonite field clipboards
No Post-It Notes	Indelible ball point pens
No chemical (blue) ice packs	Regular ice
FIELD CLOTHING AND PPE ITEMS	
No new clothing or water resistant, waterproof, or stain-treated clothing, clothing containing Gore-Tex™	Well-laundered clothing, defined as clothing that has been washed 5-6 times after purchase, made of synthetic or natural fibers (preferably cotton)
No clothing laundered using fabric softener or dried with anti-static sheets	No fabric softener or anti-static drier sheets
No boots containing Gore-Tex™	Boots made with polyurethane and polyvinyl chloride (PVC)
No Tyvek®	Cotton Clothing
No cosmetics, moisturizers, hand cream or other related products as part of personal cleaning/showering routine on the morning of sampling	**Sunscreens:** Do not use. **Insect Repellents:** Do not use
SAMPLE CONTAINERS ITEMS	
No LDPE or glass containers	HDPE or polypropylene
No Teflon™-lined caps	Lined or unlined HDPE or polypropylene caps
RAIN GEAR ITEMS	
No waterproof or resistant rain gear	PVC, polyurethane, polyethylene or rubber rain gear can be worn
DECONTAMINATION ITEMS	
No Decon 90	Alconox® and/or Liquinox®
No water from an on-site well	Potable water from municipal drinking water supply
FOOD ITEMS	
No food and drink, with exceptions noted on the right	Bottled water and hydration drinks (i.e. Gatorade® and Powerade®) to be brought and consumed only in the staging area.

Table 6 – Method and Standard Operating Procedures for PFAS

Parameter	Document Number	Title
PFAS	U.S. EPA Method: EPA/600/R-18/352	Method 537.1 – Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)
PFAS	Eurofins Eaton Analytical, LLC SOP: LCMS – SOP26645	LCMS – EPA 537.1 – Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and LC/MS/MS.

Table 7 – Laboratory Quality Control: Frequency and Criteria

Parameter	Laboratory Reagent Blank	Laboratory Fortified Blank	Laboratory Fortified Sample Matrix/Laboratory Fortified Sample Matrix Duplicate and Duplicates	Field Reagent Blank
PFAS	1/batch, up to 20 samples; < 1/3 minimum reporting level	1/20 samples; Low-level LFB 50-150%; Medium-level LFB 70-130%; High-level LFB 70-130%	1/20 samples; Low-level LFSM 50-150%, RPD ≤ 50% for analytes within 2X the MRL, RPD ≤ 30% for analytes higher than 2X the MRL; Medium-level LFSM 70-130%, RPD ≤ 30%; High-level LFSM 70-130%, RPD ≤ 30%	1/site; Analyze if field sample has analyte(s) ≥ MRL; < 1/3 minimum reporting level

REFERENCES

AECOM. July 26, 2019. *2019 PFAS Sampling of Drinking Water Supplies in Michigan*, Prepared for: Michigan Department of Environment, Great Lakes, and Energy, 172 pps.

Kentucky Department for Environmental Protection (KDEP). November 18, 2019. *Evaluation of Kentucky Community Drinking Water for Per- & Poly-Fluoroalkyl Substances,* Division of Water, 92 pps.

Mills, P.C. and Cobb, R.P. 2015. *Hexavalent and total chromium at low reporting concentrations in source-water aquifers and surface waters used for public supply in Illinois*, 2013: U.S. Geological Survey Scientific Investigations Report 2015–5020, 58 p., http://dx.doi.org/10.3133/sir20155020.

U.S. Environmental Protection Agency. May 2001. *EPA Requirements for Quality Assurance Project Plans (QA/R-5)*. (EPA/240/B-01/003). Washington, D.C.

APPENDICES

Appendix A – PFOA Health Advisory

Appendix B – PFOS Health Advisory

Appendix C – PFOA-PFOS-Office of Water HAs, Briefing for Association of State Drinking Water Administrators (ASDWA)

Appendix D – Eurofins Eaton Analytical, LLC Certificate of NELAC Accreditation

Appendix E – Method 537.1 Section 8 - PFAS Sample Collection Instructions

Appendix F – Illinois EPA's SOP for Sample Collection of PFAS at CWS in Illinois

Appendix G – Eurofins Eaton Analytical, LLC Chain-of-Custody Sheet

Appendix H – U.S. EPA Method 537.1 for PFAS Analysis

Appendix I – Eurofins Eaton Analytical, LLC SOP for PFAS Extraction and Analysis

Appendix J – Method 537.1 Table 12 – Initial Demonstration of Capability Quality Control Requirements

Appendix K – Method 537.1 Table 13 – Ongoing Quality Control Requirements (Summary)

Appendix A – PFOA Health Advisory

United States Environmental Protection Agency | Office of Water Mail Code 4304T | EPA 822-R-16-005 May 2016

Drinking Water Health Advisory for Perfluorooctanoic Acid (PFOA)

NOTE: See the attached hard copy or the file *Appendix A - PFOA Health Advisory.pdf*

Appendix B – PFOS Health Advisory

United States Environmental Protection Agency | Office of Water Mail Code 4304T | EPA 822-R-16-004 May 2016

Drinking Water Health Advisory for Perfluorooctane Sulfonate (PFOS)

NOTE: See the attached hard copy or the file *Appendix B - PFOS Health Advisory.pdf*

Appendix C – PFOA-PFOS-Office of Water HAs, Briefing for Association of State Drinking Water Administrators (ASDWA)

Lifetime Health Advisories (HA) for Perfluorooctane Sulfonate (PFOS) and Perfluorooctanoic Acid (PFOA)

Briefing for Association of State Drinking Water Administrators
May 30, 2016

NOTE: See the attached hard copy or the file *Appendix C - PFOA-PFOS-OW HAs ASDWA.pdf*

Appendix D – Eurofins Eaton Analytical, LLC Certificate of NELAC Accreditation

OREGON

Environmental Laboratory Accreditation Program

NELAP Recognized

Eurofins Eaton Analytical, LLC

4074

110 South Hill Street

South Bend, IN 46617

IS GRANTED APPROVAL BY ORELAP UNDER THE 2009 TNI STANDARDS, TO PERFORM ANALYSES ON ENVIRONMENTAL SAMPLES IN MATRICES AS LISTED BELOW :

Air	Drinking Water	Non Potable Water	Solids and Chem. Waste	Tissue
	Chemistry	Microbiology		
	Microbiology			
	Radiochemistry			

AND AS RECORDED IN THE LIST OF APPROVED ANALYTES, METHODS, ANALYTICAL TECHNIQUES, AND FIELDS OF TESTING ISSUED CONCURRENTLY WITH THIS CERTIFICATE AND REVISED AS NECESSARY.

ACCREDITED STATUS DEPENDS ON SUCCESSFUL ONGOING PARTICIPATION IN THE PROGRAM AND CONTINUED COMPLIANCE WITH THE STANDARDS.

CUSTOMERS ARE URGED TO VERIFY THE LABORATORY'S CURRENT ACCREDITATION STATUS IN OREGON.

Travis Bartholomew
Oregon State Public Health Laboratory
ORELAP Program Manager
7202 NE Evergreen Parkway, Suite 100
Hillsboro, OR 97124

EFFECTIVE DATE : 05/5/2020

EXPIRATION DATE : 09/16/2020

Certificate No : 4074 - 012

Appendix E – Method 537.1, Section 8 - PFAS Sample Collection Instructions

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. SAMPLE BOTTLE PREPARATION

8.1.1. Samples must be collected in a 250-mL polypropylene bottle fitted with a polypropylene screw-cap.

8.1.2. The preservation reagent, listed in the table below, is added to each sample bottle as a solid prior to shipment to the field (or prior to sample collection).

Compound	Amount	Purpose
Trizma® (Sect. 7.1.5)	5.0 g/L	buffering reagent and removes free chlorine

8.2. SAMPLE COLLECTION

8.2.1. The sample handler must wash their hands before sampling and wear nitrile gloves while filling and sealing the sample bottles. PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

8.2.2. Open the tap and allow the system to flush until the water temperature has stabilized (approximately 3 to 5 min). Collect samples from the flowing system.

8.2.3. Fill sample bottles, taking care not to flush out the sample preservation reagent. Samples do not need to be collected headspace free.

8.2.4. After collecting the sample, cap the bottle and agitate by hand until preservative is dissolved. Keep the sample sealed from time of collection until extraction.

8.3. FIELD REAGENT BLANKS (FRB)

8.3.1. A FRB must be handled along with each sample set. The sample set is composed of samples collected from the same sample site and at the same time. At the laboratory, fill the field blank sample bottle with reagent water and preservatives, seal, and ship to the sampling site along with the sample bottles. For each FRB shipped, an empty sample bottle (no preservatives) must also be shipped. At the sampling site, the sampler must open the shipped FRB and pour the preserved reagent water into the empty shipped sample bottle, seal and label this bottle as the FRB. The FRB is shipped back to the laboratory along with the samples and analyzed to ensure that PFAS were not introduced into the sample during sample collection/handling.

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

Appendix E – Method 537.1, Section 8 - PFAS Sample Collection Instructions (Cont.)

8.3.3. The reagent water used for the FRBs must be initially analyzed for method analytes as a LRB (using the same lot of sample bottles as the field samples) and must meet the LRB criteria in Section 9.3.1 prior to use. This requirement will ensure samples are not being discarded due to contaminated reagent water or sample bottles rather than contamination during sampling.

8.4. SAMPLE SHIPMENT AND STORAGE – Samples must be chilled during shipment and must not exceed 10 °C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10 °C when the samples are received at the laboratory. Samples stored in the lab must be held at or below 6 °C until extraction, but must not be frozen.

NOTE: Samples that are significantly above 10° C, at the time of collection, may need to be iced or refrigerated for a period of time, in order to chill them prior to shipping. This will allow them to be shipped with sufficient ice to meet the above requirements.

8.5. SAMPLE AND EXTRACT HOLDING TIMES – Results of the sample storage stability study (Table 10) indicated that all compounds listed in this method have adequate stability for 14 days when collected, preserved, shipped and stored as described in Sections 8.1, 8.2, and 8.4. Therefore, water samples should be extracted as soon as possible but must be extracted within 14 days. Extracts must be stored at room temperature and analyzed within 28 days after extraction. The extract storage stability study data are presented in Table 11.

Appendix F – Illinois EPA's SOP for Sample Collection of PFAS at CWS in Illinois

Illinois Environmental Protection Agency

Bureau of Water

Document Control Number 248

Standard Operating Procedure for

Sample Collection of

Per- and Poly-Fluorinated Alkyl Substances (PFAS)

at Community Water Supplies in Illinois

Division of Public Water Supplies (DPWS), Groundwater Section
1021 North Grand Avenue East
P.O. Box 19276
Springfield, Illinois 62794-9276

Approved:

Anthony Dulka, P.G.
Illinois EPA Bureau of Water (BOW),
Planning & Assessment Unit Manager, Headquarters, DPWS

02/25/2020
Date

Michelle Rousey
Illinois EPA BOW, Quality Assurance Officer, Headquarters, BOW

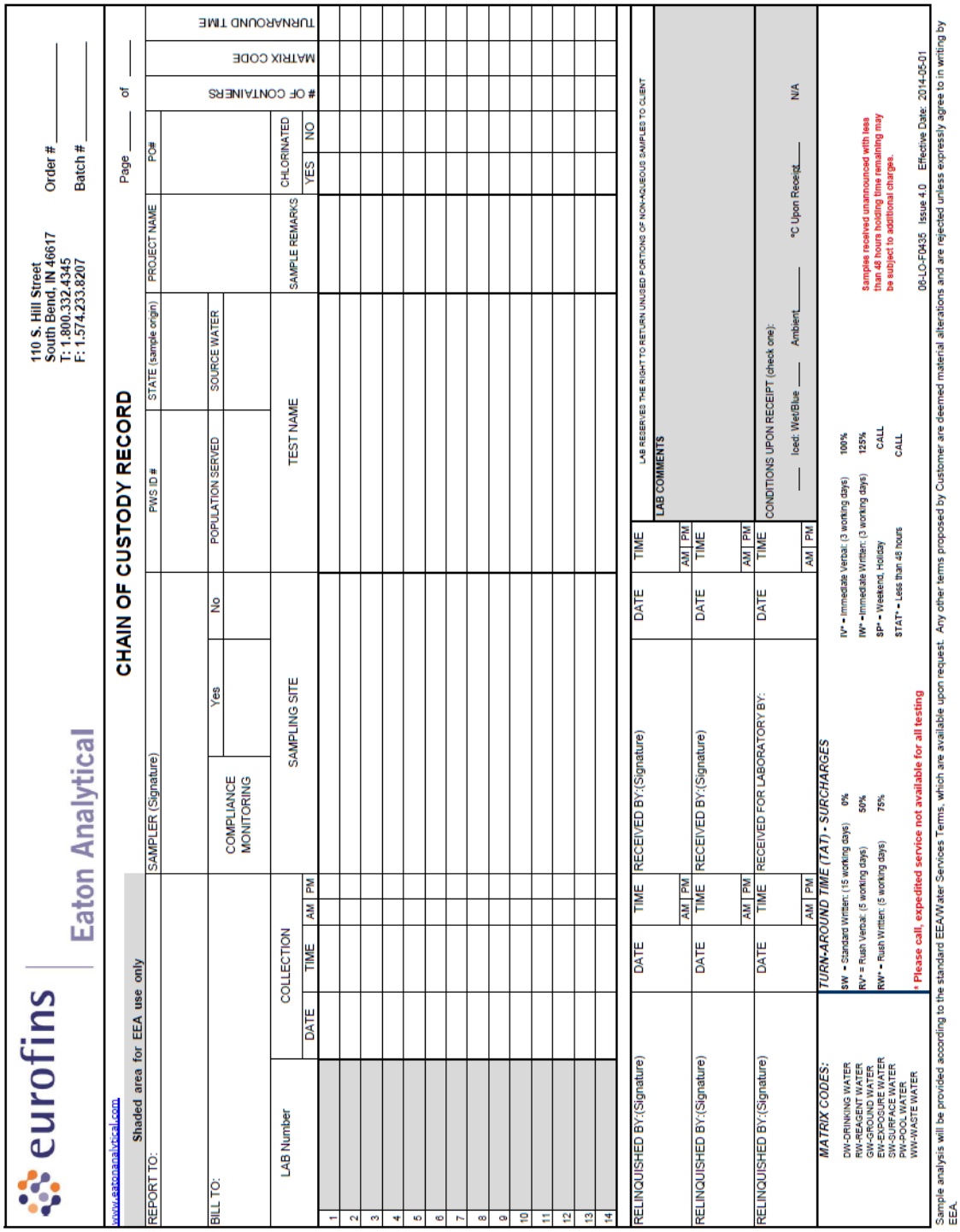
2/25/2020
Date

Annual Review (no changes):

	2021	2022	2023	2024	2025
Initials/Date					Full review and approval needed
Initials/Date					

NOTE: See the attached hard copy or the file *Appendix F – Illinois EPA's SOP for Sample Collection of PFAS at CWS in Illinois.pdf.*

Appendix G – Eurofins Eaton Analytical, LLC Chain-of-Custody Sheet



eurofins | Eaton Analytical

www.eatonanalytical.com

110 S. Hill Street
South Bend, IN 46617
T: 1.800.332.4345
F: 1.574.233.8207

Order # _____
Batch # _____

CHAIN OF CUSTODY RECORD

Page ____ of ____

Shaded area for EEA use only

REPORT TO:

SAMPLER (Signature)

PWS ID #

STATE (sample origin)

PROJECT NAME

PO#

BILL TO:

COMPLIANCE MONITORING: Yes / No

POPULATION SERVED

SOURCE WATER

LAB Number	COLLECTION DATE	COLLECTION TIME	AM	PM	SAMPLING SITE	TEST NAME	SAMPLE REMARKS	CHLORINATED YES	CHLORINATED NO	# OF CONTAINERS	MATRIX CODE	TURNAROUND TIME
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												

RELINQUISHED BY:(Signature) DATE TIME AM PM RECEIVED BY:(Signature) DATE TIME AM PM

RELINQUISHED BY:(Signature) DATE TIME AM PM RECEIVED BY:(Signature) DATE TIME AM PM

RELINQUISHED BY:(Signature) DATE TIME AM PM RECEIVED FOR LABORATORY BY: DATE TIME AM PM

LAB RESERVES THE RIGHT TO RETURN UNUSED PORTIONS OF NON-AQUEOUS SAMPLES TO CLIENT

LAB COMMENTS

CONDITIONS UPON RECEIPT (check one):

____ Iced: Wet/Blue ____ Ambient ____ °C Upon Receipt ____ N/A

MATRIX CODES:

DW-DRINKING WATER
RW-REAGENT WATER
GW-GROUND WATER
EW-EXPOSURE WATER
SW-SURFACE WATER
PW-POOL WATER
WW-WASTE WATER

TURN-AROUND TIME (TAT) - SURCHARGES

$W - Standard Written: (15 working days) 0%
RV* = Rush Verbal: (5 working days) 50%
RW* - Rush Written: (5 working days) 75%

IV* - Immediate Verbal: (3 working days) 100%
IW* -Immediate Written: (3 working days) 125%
$P* - Weekend, Holiday CALL
$TAT* - Less than 48 hours CALL

Samples received unannounced with less than 48 hours holding time remaining may be subject to additional charges.

* Please call, expedited service not available for all testing

Sample analysis will be provided according to the standard EEA/Water Services Terms, which are available upon request. Any other terms proposed by Customer are deemed material alterations and are rejected unless expressly agree to in writing by EEA.

06-LO-F0435 Issue 4.0 Effective Date: 2014-05-01

NOTE: See the attached hard copy or the file *Appendix G – Eurofins Eaton Analytical, LLC Chain-of-Custody Sheet.pdf*

Appendix H – U.S. EPA Method 537.1 for PFAS Analysis

EPA Document #: EPA/600/R-18/352

METHOD 537.1 DETERMINATION OF SELECTED PER- AND POLYFLUORINATED ALKYL SUBSTANCES IN DRINKING WATER BY SOLID PHASE EXTRACTION AND LIQUID CHROMATOGRAPHY/TANDEM MASS SPECTROMETRY (LC/MS/MS)

Version 1.0
November 2018

J.A. Shoemaker and D.R. Tettenhorst, Office of Research and Development

J.A. Shoemaker (Office of Research and Development), P.E. Grimmett (Office of Research and Development), B.K. Boutin (National Council on Aging), Method 537, Rev 1.1 (2009)

NATIONAL EXPOSURE RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U. S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

NOTE: See the attached hard copy or the file *Appendix H – USEPA Method 537.1 for PFAS Analysis.pdf*

Appendix I – Eurofins Eaton Analytical, LLC SOP for PFAS Extraction and Analysis

eurofins	Always check on-line for validity.	Level:
Document number: **LCMS-SOP26645**	**LCMS-EPA 537.1 Determination of Selected Per - and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and LC/MS/MS**	**Standard Operating Procedure**
Old Reference:		
Version: **5**		Organisation level: **6-Unit**
Approved by: **UBIR, UCC9, UTZI, UYLI** Effective Date **13-MAY-2020**	Document users: **6_LCMS, 6_SP**	Responsible: **6_LCMS**

EUROFINS EATON ANALYTICAL, LLC
Standard Operating Procedure
EPA 537.1
Confidential

NOTE: See the attached hard copy or the file *Appendix I – Eurofins Eaton Analytical, LLC SOP for PFAS Extraction and Analysis.pdf*

Appendix J – Method 537.1 Table 12 – Initial Demonstration of Capability Quality Control Requirements

Method Reference	Requirement	Specification and Frequency	Acceptance Criteria
Sect. 9.2.2	Initial Demonstration of Low System Background	Analyze LRB prior to any other IDC steps.	Demonstrate that all method analytes are below 1/3 the MRL and that possible interferences from extraction media do not prevent the identification and quantification of method analytes.
Sect. 9.2.3	Initial Demonstration of Precision (IDP)	Analyze four to seven replicate LFBs fortified near the midrange calibration concentration.	%RSD must be <20%
Sect. 9.2.4	Initial Demonstration of Accuracy (IDA)	Calculate average recovery for replicates used in IDP.	Mean recovery ± 30% of true value
Sect. 9.2.5	Initial Demonstration of Peak Asymmetry Factor	Calculate the peak asymmetry factor using the equation in Section 9.3.9 for the first two eluting chromatographic peaks in a mid-level CAL standard.	Peak asymmetry factor of 0.8 - 1.5
Sect. 9.2.6	Minimum Reporting Limit (MRL) Confirmation	Fortify, extract and analyze seven replicate LFBs at the proposed MRL concentration. Calculate the Mean and the Half Range (HR). Confirm that the upper and lower limits for the Prediction Interval of Result (Upper PIR, and Lower PIR, Sect. 9.2.6.2) meet the recovery criteria.	Upper PIR ≤ 150% Lower PIR ≥ 50%
Sect. 9.2.7 and 9.3.10	Quality Control Sample (QCS)	Analyze a standard from a second source, as part of IDC.	Results must be within 70-130% of true value.
Sect. 9.2.8	Detection Limit (DL) Determination (optional)	Over a period of three days, prepare a minimum of seven replicate LFBs fortified at a concentration estimated to be near the DL. Analyze the replicates through all steps of the analysis. Calculate the DL using the equation in Sect. 9.2.8.1.	Data from DL replicates are not required to meet method precision and accuracy criteria. If the DL replicates are fortified at a low enough concentration, it is likely that they will not meet precision and accuracy criteria.

NOTE: Table 12 is intended as an abbreviated summary of QC requirements provided as a convenience to the method user. Because the information has been abbreviated to fit the table format, there may be issues that need additional clarification, or areas where important additional information from the method text is needed. In all cases, the full text of the QC in Section 9 supersedes any missing or conflicting information in this table.

Appendix K – Method 537.1 Table 13 – Ongoing Quality Control Requirements (Summary)

Method Reference	Requirement	Specification and Frequency	Acceptance Criteria
Sect. 8.1 - Sect. 8.5	Sample Holding Time	14 days with appropriate preservation and storage as described in Sections 8.1-8.5.	Sample results are valid only if samples are extracted within the sample holding time.
Sect. 8.5	Extract Holding Time	28 days when stored at room temperature in polypropylene centrifuge tubes.	Extract results are valid only if extracts are analyzed within the extract holding time.
Sect. 9.3.1	Laboratory Reagent Blank (LRB)	One LRB with each extraction batch of up to 20 samples.	Demonstrate that all method analytes are below 1/3 the MRL, and confirm that possible interferences do not prevent quantification of method analytes. If targets exceed 1/3 the MRL or if interferences are present, results for these subject analytes in the extraction batch are invalid.
Sect. 9.3.3	Laboratory Fortified Blank (LFB)	One LFB is required for each extraction batch of up to 20 Field Samples. Rotate the fortified concentrations between low, medium and high amounts.	Results of LFB analyses must be 70-130% of the true value for each method analyte for all fortified concentrations except the lowest CAL point. Results of the LFBs corresponding to the lowest CAL point for each method analyte must be 50-150% of the true value.
Sect. 9.3.4	Internal Standard (IS)	Internal standards, $^{13}C_2$-PFOA (IS#1), $^{13}C_4$-PFOS (IS#2), and d_3-NMeFOSAA (IS#3), are added to all standards and sample extracts, including QC samples. Compare IS areas to the average IS area in the initial calibration and to the most recent CCC.	Peak area counts for all ISs in all injections must be within ± 50% of the average peak area calculated during the initial calibration and 70-140% from the most recent CCC. If ISs do not meet this criterion, corresponding target results are invalid.
Sect. 9.3.5	Surrogate Standards (SUR)	Surrogate standards, $^{13}C_2$-PFHxA, $^{13}C_3$-HFPO-DA, $^{13}C_2$-PFDA, and d_5-NEtFOSAA, are added to all CAL standards and samples, including QC samples. Calculate SUR recoveries.	SUR recoveries must be 70-130% of the true value. If a SUR fails this criterion, report all results for sample as suspect/SUR recovery.