GUIDELINES FOR SAMPLING AND ANALYSIS OF PFAS

Under NYSDEC’s Part 375 Remedial Programs

January 2020
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ERRATA SHEET for

Guidelines for Sampling and Analysis of PFAS Under NYSDEC's Part 375 Program
Issued January 17, 2020

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Guidelines for Sampling and Analysis of Per- and Polyfluoroalkyl Substances (PFAS) Under NYSDEC’s Part 375 Remedial Programs

Objective
New York State Department of Environmental Conservation’s Division of Environmental Remediation (DER) performs or oversees sampling of environmental media and subsequent analysis of PFAS as part of remedial programs implemented under 6 NYCRR Part 375. To ensure consistency in sampling, analysis and reporting of PFAS, DER has developed this document to summarize procedures and update previous DER technical guidance pertaining to PFAS.

Applicability
Sampling for PFAS has already been initiated at numerous sites under DER-approved work plans, in accordance with specified procedures. All future work plans should include PFAS sampling and analysis procedures that conform to the guidelines provided herein.

As part of a site investigation or remedial action compliance program, whenever samples of potentially affected media are collected and analyzed for the standard Target Analyte List/Target Compound List (TAL/TCL), PFAS analysis should also be performed. Potentially affected media can include soil, groundwater, surface water, and sediment. Based upon the potential for biota to be affected, biota sampling and analysis for PFAS may also be warranted as determined pursuant to a Fish and Wildlife Impact Analysis. Soil vapor sampling for PFAS is not required.

Field Sampling Procedures
DER-10 specifies technical guidance applicable to DER’s remedial programs. Given the prevalence and use of PFAS, DER has developed “best management practices” specific to sampling for PFAS. As specified in DER-10 Chapter 2, quality assurance procedures are to be submitted with investigation work plans. Typically, these procedures are incorporated into a work plan, or submitted as a stand-alone document (e.g., a Quality Assurance Project Plan). Quality assurance guidelines for PFAS are listed in Appendix A - Quality Assurance Project Plan (QAPP) Guidelines for PFAS.

Field sampling for PFAS performed under DER remedial programs should follow the appropriate procedures outlined for soils, sediments or other solids (Appendix B), non-potable groundwater (Appendix C), surface water (Appendix D), public or private water supply wells (Appendix E), and fish tissue (Appendix F).

QA/QC samples (e.g. duplicates, MS/MSD) should be collected as specified in DER-10, Section 2.3(c). For sampling equipment coming in contact with aqueous samples only, rinsate or equipment blanks should be collected. Equipment blanks should be collected at a minimum frequency of one per day or one per twenty samples, whichever is more frequent.

Data Assessment and Application to Site Cleanup
Until such time as Ambient Water Quality Standards (AWQS) and Soil Cleanup Objectives (SCOs) for PFAS are published, the extent of contaminated media potentially subject to remediation should be determined on a case-by-case basis using the procedures discussed below and the criteria in DER-10.
Water Sample Results

PFAS should be further assessed and considered as a potential contaminant of concern in groundwater or surface water if PFOA or PFOS is detected in any water sample at or above 10 ng/L (ppt). In addition, further assessment of water may be warranted if either of the following screening levels are met:

a. any other individual PFAS (not PFOA or PFOS) is detected in water at or above 100 ng/L; or
b. total concentration of PFAS (including PFOA and PFOS) is detected in water at or above 500 ng/L

If PFAS are identified as a contaminant of concern for a site, they should be assessed as part of the remedy selection process in accordance with Part 375 and DER-10.

Soil Sample Results

The extent of soil contamination for purposes of delineation and remedy selection should be determined by having certain soil samples tested by Synthetic Precipitation Leaching Procedure (SPLP) and the leachate analyzed for PFAS. Soil exhibiting SPLP results above 70 ppt for either PFOA or PFOS (individually or combined) are to be evaluated during the cleanup phase.

Sites in the site management phase should evaluate for PFAS to determine if modification to any components of the SMP is necessary (e.g., monitoring for PFAS, upgrading treatment facilities, or performing an RSO).

Testing for Imported Soil

Soil imported to a site for use in a soil cap, soil cover, or as backfill is to be tested for PFAS in general conformance with DER-10, Section 5.4(e) for the PFAS Analyte List (Appendix F) using the analytical procedures discussed below and the criteria in DER-10 associated with SVOCs.

If PFOA or PFOS is detected in any sample at or above 1 µg/kg, then soil should be tested by SPLP and the leachate analyzed for PFAS. If the SPLP results exceed 10 ppt for either PFOA or PFOS (individually) then the source of backfill should be rejected, unless a site-specific exemption is provided by DER. SPLP leachate criteria is based on the Maximum Contaminant Levels proposed for drinking water by New York State’s Department of Health, this value may be updated based on future Federal or State promulgated regulatory standards. Remedial parties have the option of analyzing samples concurrently for both PFAS in soil and in the SPLP leachate to minimize project delays. Category B deliverables should be submitted for backfill samples, though a DUSR is not required.

Analysis and Reporting

As of January 2020, the United States Environmental Protection Agency (EPA) does not have a validated method for analysis of PFAS for media commonly analyzed under DER remedial programs (non-potable waters, solids). DER has developed the following guidelines to ensure consistency in analysis and reporting of PFAS.

The investigation work plan should describe analysis and reporting procedures, including laboratory analytical procedures for the methods discussed below. As specified in DER-10 Section 2.2, laboratories should provide a full Category B deliverable. In addition, a Data Usability Summary Report (DUSR) should be prepared by an independent, third party data validator. Electronic data submissions should meet the requirements provided at: https://www.dec.ny.gov/chemical/62440.html.

DER has developed a PFAS Analyte List (Appendix F) for remedial programs to understand the nature of contamination at sites. It is expected that reported results for PFAS will include, at a minimum, all the compounds listed. If lab and/or matrix specific issues are encountered for any analytes, the DER project manager, in consultation with the DER chemist, will make case-by-case decisions as to whether certain analytes may be temporarily or permanently discontinued from analysis at each site. As with other contaminants that are analyzed for at a site, the PFAS Analyte List may be refined for future sampling events based on investigative findings.
Routine Analysis

Currently, New York State Department of Health’s Environmental Laboratory Approval Program (ELAP) does not offer certification for PFAS in matrices other than finished drinking water. However, laboratories analyzing environmental samples for PFAS (e.g., soil, sediments, and groundwater) under DER’s Part 375 remedial programs need to hold ELAP certification for PFOA and PFOS in drinking water by EPA Method 537.1 or ISO 25101. Laboratories should adhere to the guidelines and criteria set forth in the DER’s laboratory guidelines for PFAS in non-potable water and solids (Appendix H - Laboratory Guidelines for Analysis of PFAS in Non-Potable Water and Solids). Data review guidelines were developed by DER to ensure data comparability and usability (Appendix H - Data Review Guidelines for Analysis of PFAS in Non-Potable Water and Solids).

LC-MS/MS analysis for PFAS using methodologies based on EPA Method 537.1 is the procedure to use for environmental samples. Isotope dilution techniques should be utilized for the analysis of PFAS in all media. Reporting limits for PFOA and PFOS in aqueous samples should not exceed 2 ng/L. Reporting limits for PFOA and PFOS in solid samples should not exceed 0.5 µg/kg. Reporting limits for all other PFAS in aqueous and solid media should be as close to these limits as possible. If laboratories indicate that they are not able to achieve these reporting limits for the entire PFAS Analyte List, site-specific decisions regarding acceptance of elevated reporting limits for specific PFAS can be made by the DER project manager in consultation with the DER chemist.

Additional Analysis

Additional laboratory methods for analysis of PFAS may be warranted at a site, such as the Synthetic Precipitation Leaching Procedure (SPLP) and Total Oxidizable Precursor Assay (TOP Assay). Commercially methods are also available for biota and air samples.

SPLP is a technique used to determine the mobility of chemicals in liquids, soils and wastes, and may be useful in determining the need for addressing PFAS-containing material as part of the remedy. SPLP by EPA Method 1312 should be used unless otherwise specified by the DER project manager in consultation with the DER chemist.

Impacted materials can be made up of PFAS that are not analyzable by routine analytical methodology. A TOP Assay can be utilized to conceptualize the amount and type of oxidizable PFAS which could be liberated in the environment, which approximates the maximum concentration of perfluoroalkyl substances that could be generated if all polyfluoroalkyl substances were oxidized. For example, some polyfluoroalkyl substances may degrade or transform to form perfluoroalkyl substances (such as PFOA or PFOS), resulting in an increase in perfluoroalkyl substance concentrations as contaminated groundwater moves away from a source. The TOP Assay converts, through oxidation, polyfluoroalkyl substances (precursors) into perfluoroalkyl substances that can be detected by routine analytical methodology.

Please note that TOP Assay analysis of highly-contaminated samples, such as those from an AFFF (aqueous film-forming foam) site, can result in incomplete oxidation of the samples and an underestimation of the total perfluoroalkyl substances.

Commercial laboratories have adopted methods which allow for the quantification of targeted PFAS in air and biota. The EPA’s Office of Research and Development (ORD) is currently developing methods which allow for air emissions characterization of PFAS, including both targeted and non-targeted analysis of PFAS. Consult with the DER project manager and the DER chemist for assistance on analyzing biota/tissue and air samples.
Appendix A - Quality Assurance Project Plan (QAPP) Guidelines for PFAS

The following guidelines (general and PFAS-specific) can be used to assist with the development of a QAPP for projects within DER involving sampling and analysis of PFAS.

General Guidelines in Accordance with DER-10

- Document/work plan section title – Quality Assurance Project Plan
- Summarize project scope, goals, and objectives
- Provide project organization including names and resumes of the project manager, Quality Assurance Officer (QAO), field staff, and Data Validator
  - The QAO should not have another position on the project, such as project or task manager, that involves project productivity or profitability as a job performance criterion
- List the ELAP-approved lab(s) to be used for analysis of samples
- Include a site map showing sample locations
- Provide detailed sampling procedures for each matrix
- Include Data Quality Usability Objectives
- List equipment decontamination procedures
- Include an “Analytical Methods/Quality Assurance Summary Table” specifying:
  - Matrix type
  - Number or frequency of samples to be collected per matrix
  - Number of field and trip blanks per matrix
  - Analytical parameters to be measured per matrix
  - Analytical methods to be used per matrix with minimum reporting limits
  - Number and type of matrix spike and matrix spike duplicate samples to be collected
  - Number and type of duplicate samples to be collected
  - Sample preservation to be used per analytical method and sample matrix
  - Sample container volume and type to be used per analytical method and sample matrix
  - Sample holding time to be used per analytical method and sample matrix
- Specify Category B laboratory data deliverables and preparation of a DUSR

Specific Guidelines for PFAS

- Include in the text that sampling for PFAS will take place
- Include in the text that PFAS will be analyzed by LC-MS/MS for PFAS using methodologies based on EPA Method 537.1
- Include the list of PFAS compounds to be analyzed (*PFAS Analyte List*)
- Include the laboratory SOP for PFAS analysis
- List the minimum method-achievable Reporting Limits for PFAS
  - Reporting Limits should be less than or equal to:
    - Aqueous – 2 ng/L (ppt)
    - Solids – 0.5 µg/kg (ppb)
- Include the laboratory Method Detection Limits for the PFAS compounds to be analyzed
- Laboratory should have ELAP certification for PFOA and PFOS in drinking water by EPA Method 537.1, EPA Method 533, or ISO 25101
- Include detailed sampling procedures
  - Precautions to be taken
  - Pump and equipment types
  - Decontamination procedures
  - Approved materials only to be used
- Specify that regular ice only will be used for sample shipment
- Specify that equipment blanks should be collected at a minimum frequency of 1 per day per matrix
Appendix B - Sampling Protocols for PFAS in Soils, Sediments and Solids

General

The objective of this protocol is to give general guidelines for the collection of soil, sediment and other solid samples for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson-pdf/sgpsect5.pdf), with the following limitations.

Laboratory Analysis and Containers

Samples collected using this protocol are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1.

The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

Equipment

Acceptable materials for sampling include: stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation’s Division of Environmental Remediation.

No sampling equipment components or sample containers should come in to contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including sample bottle cap liners with a PTFE layer.

A list of acceptable equipment is provided below, but other equipment may be considered appropriate based on sampling conditions.

- stainless steel spoon
- stainless steel bowl
- steel hand auger or shovel without any coatings

Equipment Decontamination

Standard two step decontamination using detergent (Alconox is acceptable) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification. Previous results of “non-detect” for PFAS from the UCMR3 water supply testing program are acceptable as verification.

Sampling Techniques

Sampling is often conducted in areas where a vegetative turf has been established. In these cases, a pre-cleaned trowel or shovel should be used to carefully remove the turf so that it may be replaced at the conclusion of sampling. Surface soil samples (e.g. 0 to 6 inches below surface) should then be collected using a pre-cleaned, stainless steel spoon. Shallow subsurface soil samples (e.g. 6 to ~36 inches below surface) may be collected by digging a hole using a pre-cleaned hand auger or shovel. When the desired subsurface depth is reached, a pre-cleaned hand auger or spoon shall be used to obtain the sample.

When the sample is obtained, it should be deposited into a stainless steel bowl for mixing prior to filling the sample containers. The soil should be placed directly into the bowl and mixed thoroughly by rolling the material into the middle until the material is homogenized. At this point the material within the bowl can be placed into the laboratory provided container.
Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

Quality Assurance/Quality Control

• Immediately place samples in a cooler maintained at 4 ± 2º Celsius using ice
• Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
• Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
• Request appropriate data deliverable (Category B) and an electronic data deliverable

Documentation

A soil log or sample log shall document the location of the sample/borehole, depth of the sample, sampling equipment, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate. Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appropriate rain gear (PVC, polyurethane, or rubber rain gear are acceptable), bug spray, and sunscreen should be used that does not contain PFAS. Well washed cotton coveralls may be used as an alternative to bug spray and/or sunscreen.

PPE that contains PFAS is acceptable when site conditions warrant additional protection for the samplers and no other materials can be used to be protective. Documentation of such use should be provided in the field notes.
Appendix C - Sampling Protocols for PFAS in Monitoring Wells

General

The objective of this protocol is to give general guidelines for the collection of groundwater samples for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

Laboratory Analysis and Container

Samples collected using this protocol are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1.

The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

Equipment

Acceptable materials for sampling include: stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation’s Division of Environmental Remediation.

No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including plumbers tape and sample bottle cap liners with a PTFE layer.

A list of acceptable equipment is provided below, but other equipment may be considered appropriate based on sampling conditions.

- stainless steel inertia pump with HDPE tubing
- peristaltic pump equipped with HDPE tubing and silicone tubing
- stainless steel bailer with stainless steel ball
- bladder pump (identified as PFAS-free) with HDPE tubing

Equipment Decontamination

Standard two step decontamination using detergent (Alconox is acceptable) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification.

Sampling Techniques

Monitoring wells should be purged in accordance with the sampling procedure (standard/volume purge or low flow purge) identified in the site work plan, which will determine the appropriate time to collect the sample. If sampling using standard purge techniques, additional purging may be needed to reduce turbidity levels, so samples contain a limited amount of sediment within the sample containers. Sample containers that contain sediment may cause issues at the laboratory, which may result in elevated reporting limits and other issues during the sample preparation that can compromise data usability. Sampling personnel should don new nitrile gloves prior to sample collection due to the potential to contact PFAS containing items (not related to the sampling equipment) during the purging activities.
Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at 4 ± 2°C Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- Collect one equipment blank every day that sampling is conducted and minimum 1 equipment blank per 20 samples. The equipment blank shall test the new and decontaminated sampling equipment utilized to obtain a sample for residual PFAS contamination. This sample is obtained by using laboratory provided PFAS-free water and passing the water over or through the sampling device and into laboratory provided sample containers
- Additional equipment blank samples may be collected to assess other equipment that is utilized at the monitoring well
- Request appropriate data deliverable (Category B) and an electronic data deliverable

Documentation

A purge log shall document the location of the sample, sampling equipment, groundwater parameters, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate. Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appropriate rain gear (PVC, polyurethane, or rubber rain gear are acceptable), bug spray, and sunscreen should be used that does not contain PFAS. Well washed cotton coveralls may be used as an alternative to bug spray and/or sunscreen.

PPE that contains PFAS is acceptable when site conditions warrant additional protection for the samplers and no other materials can be used to be protective. Documentation of such use should be provided in the field notes.
Appendix D - Sampling Protocols for PFAS in Surface Water

General
The objective of this protocol is to give general guidelines for the collection of surface water samples for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

Laboratory Analysis and Container
Samples collected using this protocol are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1.

The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

Equipment
Acceptable materials for sampling include: stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation’s Division of Environmental Remediation.

No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including sample bottle cap liners with a PTFE layer.

A list of acceptable equipment is provided below, but other equipment may be considered appropriate based on sampling conditions.

- stainless steel cup

Equipment Decontamination
Standard two step decontamination using detergent (Alconox is acceptable) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification.

Sampling Techniques
Where conditions permit, (e.g. creek or pond) sampling devices (e.g. stainless steel cup) should be rinsed with site medium to be sampled prior to collection of the sample. At this point the sample can be collected and poured into the sample container.

If site conditions permit, samples can be collected directly into the laboratory container.

Sample Identification and Logging
A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).
Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at 4 ± 2°C Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- Collect one equipment blank every day that sampling is conducted and minimum 1 equipment blank per 20 samples. The equipment blank shall test the new and decontaminated sampling equipment utilized to obtain a sample for residual PFAS contamination. This sample is obtained by using laboratory provided PFAS-free water and passing the water over or through the sampling device and into laboratory provided sample containers
- Request appropriate data deliverable (Category B) and an electronic data deliverable

Documentation

A sample log shall document the location of the sample, sampling equipment, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate. Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appropriate rain gear (PVC, polyurethane, or rubber rain gear are acceptable), bug spray, and sunscreen should be used that does not contain PFAS. Well washed cotton coveralls may be used as an alternative to bug spray and/or sunscreen.

PPE that contains PFAS is acceptable when site conditions warrant additional protection for the samplers and no other materials can be used to be protective. Documentation of such use should be provided in the field notes.
Appendix E - Sampling Protocols for PFAS in Private Water Supply Wells

General

The objective of this protocol is to give general guidelines for the collection of water samples from private water supply wells (with a functioning pump) for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

Laboratory Analysis and Container

Drinking water samples collected using this protocol are intended to be analyzed for PFAS by ISO Method 25101. The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

Equipment

Acceptable materials for sampling include: stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation’s Division of Environmental Remediation.

No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials (e.g. plumbers tape), including sample bottle cap liners with a PTFE layer.

Equipment Decontamination

Standard two step decontamination using detergent (Alconox is acceptable) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification.

Sampling Techniques

Locate and assess the pressure tank and determine if any filter units are present within the building. Establish the sample location as close to the well pump as possible, which is typically the spigot at the pressure tank. Ensure sampling equipment is kept clean during sampling as access to the pressure tank spigot, which is likely located close to the ground, may be obstructed and may hinder sample collection.

Prior to sampling, a faucet downstream of the pressure tank (e.g., wash room sink) should be run until the well pump comes on and a decrease in water temperature is noted which indicates that the water is coming from the well. If the homeowner is amenable, staff should run the water longer to purge the well (15+ minutes) to provide a sample representative of the water in the formation rather than standing water in the well and piping system including the pressure tank. At this point a new pair of nitrile gloves should be donned and the sample can be collected from the sample point at the pressure tank.

Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).
Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at 4 ± 2º Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- If equipment was used, collect one equipment blank every day that sampling is conducted and minimum 1 equipment blank per 20 samples. The equipment blank shall test the new and decontaminated sampling equipment utilized to obtain a sample for residual PFAS contamination. This sample is obtained by using laboratory provided PFAS-free water and passing the water over or through the sampling device and into laboratory provided sample containers
- Request appropriate data deliverable (Category B) and an electronic data deliverable

Documentation

A sample log shall document the location of the private well, sample point location, owner contact information, sampling equipment, purge duration, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate and available (e.g. well construction, pump type and location, yield, installation date). Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.
Appendix F - Sampling Protocols for PFAS in Fish

This appendix contains a copy of the latest guidelines developed by the Division of Fish and Wildlife (DFW) entitled “General Fish Handling Procedures for Contaminant Analysis” (Ver. 8).

**Procedure Name:** General Fish Handling Procedures for Contaminant Analysis

**Number:** FW-005

**Purpose:** This procedure describes data collection, fish processing and delivery of fish collected for contaminant monitoring. It contains the chain of custody and collection record forms that should be used for the collections.

**Organization:** Environmental Monitoring Section  
Bureau of Ecosystem Health  
Division of Fish and Wildlife (DFW)  
New York State Department of Environmental Conservation (NYSDEC)  
625 Broadway  
Albany, New York 12233-4756

**Version:** 8

**Previous Version Date:** 21 March 2018

**Summary of Changes to this Version:** Updated bureau name to Bureau of Ecosystem Health. Added direction to list the names of all field crew on the collection record. Minor formatting changes on chain of custody and collection records.

**Originator or Revised by:** Wayne Richter, Jesse Becker

**Date:** 26 April 2019

**Quality Assurance Officer and Approval Date:** Jesse Becker, 26 April 2019
NEW YORK STATE
DEPARTMENT OF ENVIRONMENTAL CONSERVATION

GENERAL FISH HANDLING PROCEDURES FOR CONTAMINANT ANALYSES

A. Original copies of all continuity of evidence (i.e., Chain of Custody) and collection record forms must accompany delivery of fish to the lab. A copy shall be directed to the Project Leader or as appropriate, Wayne Richter. All necessary forms will be supplied by the Bureau of Ecosystem Health. Because some samples may be used in legal cases, it is critical that each section is filled out completely. Each Chain of Custody form has three main sections:

1. The top box is to be filled out and signed by the person responsible for the fish collection (e.g., crew leader, field biologist, researcher). This person is responsible for delivery of the samples to DEC facilities or personnel (e.g., regional office or biologist).

2. The second section is to be filled out and signed by the person responsible for the collections while being stored at DEC, before delivery to the analytical lab. This may be the same person as in (1), but it is still required that they complete the section. Also important is the range of identification numbers (i.e., tag numbers) included in the sample batch.

3. Finally, the bottom box is to record any transfers between DEC personnel and facilities. Each subsequent transfer should be identified, signed, and dated, until laboratory personnel take possession of the fish.

B. The following data are required on each Fish Collection Record form:

1. Project and Site Name.

2. DEC Region.

3. All personnel (and affiliation) involved in the collection.

4. Method of collection (gill net, hook and line, etc.)

5. Preservation Method.

C. The following data are to be taken on each fish collected and recorded on the Fish Collection Record form:

1. Tag number - Each specimen is to be individually jaw tagged at time of collection with a unique number. Make sure the tag is turned out so that the number can be read without opening the bag. Use tags in sequential order. For small fish or composite samples place the tag inside the bag with the samples. The Bureau of Ecosystem Health can supply the tags.

2. Species identification (please be explicit enough to enable assigning genus and species). Group fish by species when processing.

3. Date collected.

4. Sample location (waterway and nearest prominent identifiable landmark).

5. Total length (nearest mm or smallest sub-unit on measuring instrument) and weight (nearest g or
smallest sub-unit of weight on weighing instrument). Take all measures as soon as possible with calibrated, protected instruments (e.g. from wind and upsets) and prior to freezing.

6. Sex - fish may be cut enough to allow sexing or other internal investigation, but do not eviscerate. Make any incision on the right side of the belly flap or exactly down the midline so that a left-side fillet can be removed.

D. General data collection recommendations:

1. It is helpful to use an ID or tag number that will be unique. It is best to use metal striped bass or other uniquely numbered metal tags. If uniquely numbered tags are unavailable, values based on the region, water body and year are likely to be unique: for example, R7CAY11001 for Region 7, Cayuga Lake, 2011, fish 1. If the fish are just numbered 1 through 20, we have to give them new numbers for our database, making it more difficult to trace your fish to their analytical results and creating an additional possibility for errors.

2. Process and record fish of the same species sequentially. Recording mistakes are less likely when all fish from a species are processed together. Starting with the bigger fish species helps avoid missing an individual.

3. If using Bureau of Ecosystem Health supplied tags or other numbered tags, use tags in sequence so that fish are recorded with sequential Tag Numbers. This makes data entry and login at the lab and use of the data in the future easier and reduces keypunch errors.

4. Record length and weight as soon as possible after collection and before freezing. Other data are recorded in the field upon collection. An age determination of each fish is optional, but if done, it is recorded in the appropriate “Age” column.

5. For composite samples of small fish, record the number of fish in the composite in the Remarks column. Record the length and weight of each individual in a composite. All fish in a composite sample should be of the same species and members of a composite should be visually matched for size.

6. Please submit photocopies of topographic maps or good quality navigation charts indicating sampling locations. GPS coordinates can be entered in the Location column of the collection record form in addition to or instead for providing a map. These records are of immense help to us (and hopefully you) in providing documented location records which are not dependent on memory and/or the same collection crew. In addition, they may be helpful for contaminant source trackdown and remediation/control efforts of the Department.

7. When recording data on fish measurements, it will help to ensure correct data recording for the data recorder to call back the numbers to the person making the measurements.

E. Each fish is to be placed in its own individual plastic bag. For small fish to be analyzed as a composite, put all of the fish for one composite in the same bag but use a separate bag for each composite. It is important to individually bag the fish to avoid difficulties or cross contamination when processing the fish for chemical analysis. Be sure to include the fish’s tag number inside the bag, preferably attached to the fish with the tag number turned out so it can be read. Tie or otherwise secure the bag closed. The Bureau of Ecosystem Health will supply the bags. If necessary, food grade bags may be procured from a suitable vendor (e.g., grocery store). It is preferable to redundantly label each bag with a manila tag tied between the knot and the body of the bag. This tag should be labeled with the project name, collection location, tag number, collection date, and fish species. If scales are collected, the scale envelope should be labeled with
Groups of fish, by species, are to be placed in one large plastic bag per sampling location. The Bureau of Ecosystem Health will supply the larger bags. Label the site bag with a manila tag tied between the knot and the body of the bag. The tag should contain: project, collection location, collection date, species and tag number ranges. Having this information on the manila tag enables lab staff to know what is in the bag without opening it.

Do not eviscerate, fillet or otherwise dissect the fish unless specifically asked to. If evisceration or dissection is specified, the fish must be cut along the exact midline or on the right side so that the left side fillet can be removed intact at the laboratory. If filleting is specified, the procedure for taking a standard fillet (SOP PREPLAB 4) must be followed, including removing scales.

Special procedures for PFAS: Unlike legacy contaminants such as PCBs, which are rarely found in day to day life, PFAS are widely used and frequently encountered. Practices that avoid sample contamination are therefore necessary. While no standard practices have been established for fish, procedures for water quality sampling can provide guidance. The following practices should be used for collections when fish are to be analyzed for PFAS:

- No materials containing Teflon.
- No Post-it notes.
- No ice packs; only water ice or dry ice.
- Any gloves worn must be powder free nitrile.
- No Gore-Tex or similar materials (Gore-Tex is a PFC with PFOA used in its manufacture).
- No stain repellent or waterproof treated clothing; these are likely to contain PFCs.
- Avoid plastic materials, other than HDPE, including clipboards and waterproof notebooks.
- Wash hands after handling any food containers or packages as these may contain PFCs.
- Keep pre-wrapped food containers and wrappers isolated from fish handling.
- Wear clothing washed at least six times since purchase.
- Wear clothing washed without fabric softener.
- Staff should avoid cosmetics, moisturizers, hand creams and similar products on the day of sampling as many of these products contain PFCs (Fujii et al. 2013). Sunscreen or insect repellent should not contain ingredients with “fluor” in their name. Apply any sunscreen or insect repellent well downwind from all materials. Hands must be washed after touching any of these products.

All fish must be kept at a temperature under 45°F (<8° C) immediately following data processing. As soon as possible, freeze at -20°C ± 5°C. Due to occasional freezer failures, daily freezer temperature logs are required. The freezer should be locked or otherwise secured to maintain chain of custody.

In most cases, samples should be delivered to the Analytical Services Unit at the Hale Creek field station. Coordinate delivery with field station staff and send copies of the collection records, continuity of evidence forms and freezer temperature logs to the field station. For samples to be analyzed elsewhere, non-routine collections or other questions, contact Wayne Richter, Bureau of Ecosystem Health, NYSDEC, 625 Broadway, Albany, New York 12233-4756, 518-402-8974, or the project leader about sample transfer. Samples will then be directed to the analytical facility and personnel noted on specific project descriptions.

A recommended equipment list is at the end of this document.
NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION  
DIVISION OF FISH AND WILDLIFE  
FISH COLLECTION RECORD

Project and Site Name ___________________________________________  DEC Region __________

Collections made by (include all crew) _____________________________________________

Sampling Method: ☐ Electrofishing  ☐ Gill netting  ☐ Trap netting  ☐ Trawling  ☐ Seining  ☐ Angling  ☐ Other ________________________________

Preservation Method: ☐ Freezing  ☐ Other ___________________________  Notes (SWFDB survey number): __________________________

<table>
<thead>
<tr>
<th>FOR LAB USE ONLY - LAB ENTRY NO.</th>
<th>COLLECTION OR TAG NO.</th>
<th>SPECIES</th>
<th>DATE TAKEN</th>
<th>LOCATION</th>
<th>AGE</th>
<th>SEX &amp;/OR REPROD. CONDIT</th>
<th>LENGTH ( )</th>
<th>WEIGHT ( )</th>
<th>REMARKS</th>
</tr>
</thead>
<tbody>
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</tbody>
</table>

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
CHAIN OF CUSTODY

I, ______________________, of ______________________, collected the
(Print Name) (Print Business Address)
following on ______________________, 20__ from ______________________
(Date) (Water Body)
in the vicinity of __________________________________________
(Landmark, Village, Road, etc.)
Town of ______________________, in ______________________ County.

Item(s) __________________________________________________

Said sample(s) were in my possession and handled according to standard procedures provided to me prior to
collection. The sample(s) were placed in the custody of a representative of the New York State Department of
Environmental Conservation on ______________________, 20__.

__________________________ ______________________
Signature Date

I, ______________________, received the above mentioned sample(s) on the date specified
and assigned identification number(s) ______________________ to the sample(s). I
have recorded pertinent data for the sample(s) on the attached collection records. The sample(s) remained in
my custody until subsequently transferred, prepared or shipped at times and on dates as attested to below.

__________________________ ______________________
Signature Date

<table>
<thead>
<tr>
<th>SECOND RECIPIENT (Print Name)</th>
<th>TIME &amp; DATE</th>
<th>PURPOSE OF TRANSFER</th>
</tr>
</thead>
<tbody>
<tr>
<td>SIGNATURE</td>
<td>UNIT</td>
<td></td>
</tr>
<tr>
<td>THIRD RECIPIENT (Print Name)</td>
<td>TIME &amp; DATE</td>
<td>PURPOSE OF TRANSFER</td>
</tr>
<tr>
<td>SIGNATURE</td>
<td>UNIT</td>
<td></td>
</tr>
<tr>
<td>FOURTH RECIPIENT (Print Name)</td>
<td>TIME &amp; DATE</td>
<td>PURPOSE OF TRANSFER</td>
</tr>
<tr>
<td>SIGNATURE</td>
<td>UNIT</td>
<td></td>
</tr>
<tr>
<td>RECEIVED IN LABORATORY BY (Print Name)</td>
<td>TIME &amp; DATE</td>
<td>REMARKS</td>
</tr>
<tr>
<td>SIGNATURE</td>
<td>UNIT</td>
<td></td>
</tr>
<tr>
<td>LOGGED IN BY (Print Name)</td>
<td>TIME &amp; DATE</td>
<td>ACCESSION NUMBERS</td>
</tr>
<tr>
<td>SIGNATURE</td>
<td>UNIT</td>
<td></td>
</tr>
</tbody>
</table>

richter: revised 21 April 2014; becker: 23 March 2017, 26 April, 2019
HANDLING INSTRUCTIONS

On day of collection, collector(s) name(s), address(es), date, geographic location of capture (attach a copy of topographic map or navigation chart), species, number kept of each species, and description of capture vicinity (proper noun, if possible) along with name of Town and County must be indicated on reverse.

Retain organisms in manila tagged plastic bags to avoid mixing capture locations. Note appropriate information on each bag tag.

Keep samples as cool as possible. Put on ice if fish cannot be frozen within 12 hours. If fish are held more than 24 hours without freezing, they will not be retained or analyzed.

Initial recipient (either DEC or designated agent) of samples from collector(s) is responsible for obtaining and recording information on the collection record forms which will accompany the chain of custody. This person will seal the container using packing tape and writing his signature, the time and the date across the tape onto the container with indelible marker. Any time a seal is broken, for whatever purpose, the incident must be recorded on the Chain of Custody (reason, time, and date) in the purpose of transfer block. Container then is resealed using new tape and rewriting signature, with time and date.

NOTICE OF WARRANTY

By signature to the chain of custody (reverse), the signatory warrants that the information provided is truthful and accurate to the best of his/her ability. The signatory affirms that he/she is willing to testify to those facts provided and the circumstances surrounding the same. Nothing in this warranty or chain of custody negates responsibility nor liability of the signatories for the truthfulness and accuracy of the statements provided.
EQUIPMENT LIST

Scale or balance of appropriate capacity for the fish to be collected.

Fish measuring board.

Plastic bags of an appropriate size for the fish to be collected and for site bags.

Individually numbered metal tags for fish.

Manila tags to label bags.

Small envelopes, approximately 2” x 3.5”, if fish scales are to be collected.

Knife for removing scales.

Chain of custody and fish collection forms.

Clipboard.

Pens or markers.

Paper towels.

Dish soap and brush.

Bucket.

Cooler.

Ice.

Duct tape.
# Appendix G – PFAS Analyte List

<table>
<thead>
<tr>
<th>Group</th>
<th>Chemical Name</th>
<th>Abbreviation</th>
<th>CAS Number</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Perfluoroalkyl sulfonates</strong></td>
<td>Perfluorobutanesulfonic acid</td>
<td>PFBS</td>
<td>375-73-5</td>
</tr>
<tr>
<td></td>
<td>Perfluorohexanesulfonic acid</td>
<td>PFHxS</td>
<td>355-46-4</td>
</tr>
<tr>
<td></td>
<td>Perfluoroheptanesulfonic acid</td>
<td>PFHpS</td>
<td>375-92-8</td>
</tr>
<tr>
<td></td>
<td>Perfluoroctanesulfonic acid</td>
<td>PFOS</td>
<td>1763-23-1</td>
</tr>
<tr>
<td></td>
<td>Perfluorodecanesulfonic acid</td>
<td>PFDS</td>
<td>335-77-3</td>
</tr>
<tr>
<td><strong>Perfluoroalkyl carboxylates</strong></td>
<td>Perfluorobutanoic acid</td>
<td>PFBA</td>
<td>375-22-4</td>
</tr>
<tr>
<td></td>
<td>Perfluoropentanoic acid</td>
<td>PFPeA</td>
<td>2706-90-3</td>
</tr>
<tr>
<td></td>
<td>Perfluorohexanoic acid</td>
<td>PFHxA</td>
<td>307-24-4</td>
</tr>
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<td></td>
<td>Perfluoroheptanoic acid</td>
<td>PFHpA</td>
<td>375-85-9</td>
</tr>
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<td></td>
<td>Perfluoroctanoic acid</td>
<td>PFOA</td>
<td>335-67-1</td>
</tr>
<tr>
<td></td>
<td>Perfluorononanoic acid</td>
<td>PFNA</td>
<td>375-95-1</td>
</tr>
<tr>
<td></td>
<td>Perfluorodecanolic acid</td>
<td>PFDA</td>
<td>335-76-2</td>
</tr>
<tr>
<td></td>
<td>Perfluoroundecanoic acid</td>
<td>PFUA/PFUdA</td>
<td>2058-94-8</td>
</tr>
<tr>
<td></td>
<td>Perfluorododecanolic acid</td>
<td>PFDoA</td>
<td>307-55-1</td>
</tr>
<tr>
<td></td>
<td>Perfluorotridecanolic acid</td>
<td>PFTriA/PFTrD</td>
<td>72629-94-8</td>
</tr>
<tr>
<td></td>
<td>Perfluorotetradecanoic acid</td>
<td>PFTA/PFTeDA</td>
<td>376-06-7</td>
</tr>
<tr>
<td><strong>Fluorinated Telomer Sulfonates</strong></td>
<td>6:2 Fluorotelomer sulfone</td>
<td>6:2 FTS</td>
<td>27619-97-2</td>
</tr>
<tr>
<td></td>
<td>8:2 Fluorotelomer sulfone</td>
<td>8:2 FTS</td>
<td>39108-34-4</td>
</tr>
<tr>
<td><strong>Perfluoroctanesulfonamides</strong></td>
<td>Perfluoroctanesulfonamide</td>
<td>FOSA</td>
<td>754-91-6</td>
</tr>
<tr>
<td><strong>Perfluoroctanesulfonamidoacetic acids</strong></td>
<td>N-methyl perfluoroctanesulfonamidoacetic acid</td>
<td>N-MeFOSAA</td>
<td>2355-31-9</td>
</tr>
<tr>
<td></td>
<td>N-ethyl perfluoroctanesulfonamidoacetic acid</td>
<td>N-EtFOSAA</td>
<td>2991-50-6</td>
</tr>
</tbody>
</table>
Appendix H - Laboratory Guidelines for Analysis of PFAS in Non-Potable Water and Solids

General

New York State Department of Environmental Conservation’s Division of Environmental Remediation (DER) developed the following guidelines for laboratories analyzing environmental samples for PFAS under DER programs. If laboratories cannot adhere to the following guidelines, they should contact DER’s Quality Assurance Officer, Dana Maikels, at dana.maikels@dec.ny.gov prior to analysis of samples.

Isotope Dilution

Isotope dilution techniques should be utilized for the analysis of PFAS in all media.

Extraction

For water samples, the entire sample bottle should be extracted, and the sample bottle rinsed with appropriate solvent to remove any residual PFAS.

For samples with high particulates, the samples should be handled in one of the following ways:

1. Spike the entire sample bottle with isotope dilution analytes (IDAs) prior to any sample manipulation. The sample can be passed through the SPE and if it clogs, record the volume that passed through.
2. If the sample contains too much sediment to attempt passing it through the SPE cartridge, the sample should be spiked with isotope dilution analytes, centrifuged and decanted.
3. If higher reporting limits are acceptable for the project, the sample can be diluted by taking a representative aliquot of the sample. If isotope dilution analytes will be diluted out of the sample, they can be added after the dilution. The sample should be homogenized prior to taking an aliquot.

If alternate sample extraction procedures are used, please contact the DER remedial program chemist prior to employing. Any deviations in sample preparation procedures should be clearly noted in the case narrative.

Signal to Noise Ratio

For all target analyte ions used for quantification, signal to noise ratio should be 3:1 or greater.

Blanks

There should be no detections in the method blanks above the reporting limits.

Ion Transitions

The ion transitions listed below should be used for the following PFAS:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Transition</th>
</tr>
</thead>
<tbody>
<tr>
<td>PFOA</td>
<td>413 &gt; 369</td>
</tr>
<tr>
<td>PFOS</td>
<td>499 &gt; 80</td>
</tr>
<tr>
<td>PFHxS</td>
<td>399 &gt; 80</td>
</tr>
<tr>
<td>PFBS</td>
<td>299 &gt; 80</td>
</tr>
<tr>
<td>6:2 FTS</td>
<td>427 &gt; 407</td>
</tr>
<tr>
<td>8:2 FTS</td>
<td>527 &gt; 507</td>
</tr>
<tr>
<td>N-EtFOSAA</td>
<td>584 &gt; 419</td>
</tr>
<tr>
<td>N-MeFOSAA</td>
<td>570 &gt; 419</td>
</tr>
</tbody>
</table>
Branched and Linear Isomers

Standards containing both branched and linear isomers should be used when standards are commercially available. Currently, quantitative standards are available for PFHxS, PFOS, NMeFOSAA, and NEtFOSAA. As more standards become available, they should be incorporated into the method. All isomer peaks present in the standard should be integrated and the areas summed. Samples should be integrated in the same manner as the standards.

Since a quantitative standard does not exist for branched isomers of PFOA, the instrument should be calibrated using just the linear isomer and a technical (qualitative) PFOA standard should be used to identify the retention time of the branched PFOA isomers in the sample. The total response of PFOA branched and linear isomers should be integrated in the samples and quantitated using the calibration curve of the linear standard.

Secondary Ion Transition Monitoring

Quantifier and qualifier ions should be monitored for all target analytes (PFBA and PFPeA are exceptions). The ratio of quantifier ion response to qualifier ion response should be calculated for each target analyte and the ratio compared to standards. Lab derived criteria should be used to determine if the ratios are acceptable.

Reporting

Detections below the reporting limit should be reported and qualified with a J qualifier.

The acid form of PFAS analytes should be reported. If the salt form of the PFAS was used as a stock standard, the measured mass should be corrected to report the acid form of the analyte.
Appendix H - Data Review Guidelines for Analysis of PFAS in Non-Potable Water and Solids

General

These guidelines are intended to be used for the validation of PFAS analytical results for projects within the Division of Environmental Remediation (DER) as well as aid in the preparation of a data usability summary report. Data reviewers should understand the methodology and techniques utilized in the analysis. Consultation with the end user of the data may be necessary to assist in determining data usability based on the data quality objectives in the Quality Assurance Project Plan. A familiarity with the laboratory’s Standard Operating Procedure may also be needed to fully evaluate the data. If you have any questions, please contact DER’s Quality Assurance Officer, Dana Maikels, at dana.maikels@dec.ny.gov.

Preservation and Holding Time

Samples should be preserved with ice to a temperature of less than 6°C upon arrival at the lab. The holding time is 14 days to extraction for aqueous and solid samples. The time from extraction to analysis for aqueous samples is 28 days and 40 days for solids.

<table>
<thead>
<tr>
<th>Temperature greatly exceeds 6°C upon arrival at the lab*</th>
<th>Use professional judgement to qualify detects and non-detects as estimated or rejected</th>
</tr>
</thead>
<tbody>
<tr>
<td>Holding time exceeding 28 days to extraction</td>
<td>Use professional judgement to qualify detects and non-detects as estimated or rejected if holding time is grossly exceeded</td>
</tr>
</tbody>
</table>

*Samples that are delivered to the lab immediately after sampling may not meet the thermal preservation guidelines. Samples are considered acceptable if they arrive on ice or an attempt to chill the samples is observed.

Initial Calibration

The initial calibration should contain a minimum of five standards for linear fit and six standards for a quadratic fit. The relative standard deviation (RSD) for a quadratic fit calibration should be less than 20%. Linear fit calibration curves should have an $R^2$ value greater than 0.990.

The low-level calibration standard should be within 50% - 150% of the true value, and the mid-level calibration standard within 70% - 130% of the true value.

<table>
<thead>
<tr>
<th>%RSD &gt;20%</th>
<th>J flag detects and UJ non detects</th>
</tr>
</thead>
<tbody>
<tr>
<td>$R^2 &gt;0.990$</td>
<td>J flag detects and UJ non detects</td>
</tr>
<tr>
<td>Low-level calibration check &lt;50% or &gt;150%</td>
<td>J flag detects and UJ non detects</td>
</tr>
<tr>
<td>Mid-level calibration check &lt;70% or &gt;130%</td>
<td>J flag detects and UJ non detects</td>
</tr>
</tbody>
</table>

Initial Calibration Verification

An initial calibration verification (ICV) standard should be from a second source (if available). The ICV should be at the same concentration as the mid-level standard of the calibration curve.

<table>
<thead>
<tr>
<th>ICV recovery &lt;70% or &gt;130%</th>
<th>J flag detects and non-detects</th>
</tr>
</thead>
</table>
Continuing Calibration Verification

Continuing calibration verification (CCV) checks should be analyzed at a frequency of one per ten field samples. If CCV recovery is very low, where detection of the analyte could be in question, ensure a low level CCV was analyzed and use to determine data quality.

<table>
<thead>
<tr>
<th>CCV recovery &lt;70 or &gt;130%</th>
<th>J flag results</th>
</tr>
</thead>
</table>

Blanks

There should be no detections in the method blanks above the reporting limits. Equipment blanks, field blanks, rinse blanks etc. should be evaluated in the same manner as method blanks. Use the most contaminated blank to evaluate the sample results.

<table>
<thead>
<tr>
<th>Blank Result</th>
<th>Sample Result</th>
<th>Qualification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Any detection</td>
<td>&lt;Reporting limit</td>
<td>Qualify as ND at reporting limit</td>
</tr>
<tr>
<td>Any detection</td>
<td>&gt;Reporting Limit and &gt;10x the blank result</td>
<td>No qualification</td>
</tr>
<tr>
<td>&gt;Reporting limit</td>
<td>&gt;Reporting limit and &lt;10x blank result</td>
<td>J+ biased high</td>
</tr>
</tbody>
</table>

Field Duplicates

A blind field duplicate should be collected at rate of one per twenty samples. The relative percent difference (RPD) should be less than 30% for analyte concentrations greater than two times the reporting limit. Use the higher result for final reporting.

<table>
<thead>
<tr>
<th>RPD &gt;30%</th>
<th>Apply J qualifier to parent sample</th>
</tr>
</thead>
</table>

Lab Control Spike

Lab control spikes should be analyzed with each extraction batch or one for every twenty samples. In the absence of lab derived criteria, use 70% - 130% recovery criteria to evaluate the data.

<table>
<thead>
<tr>
<th>Recovery &lt;70% or &gt;130% (lab derived criteria can also be used)</th>
<th>Apply J qualifier to detects and UJ qualifier to non detects</th>
</tr>
</thead>
</table>

Matrix Spike/Matrix Spike Duplicate

One matrix spike and matrix spike duplicate should be collected at a rate of one per twenty samples. Use professional judgement to reject results based on out of control MS/MSD recoveries.

<table>
<thead>
<tr>
<th>Recovery &lt;70% or &gt;130% (lab derived criteria can also be used)</th>
<th>Apply J qualifier to detects and UJ qualifier to non detects of parent sample only</th>
</tr>
</thead>
<tbody>
<tr>
<td>RPD &gt;30%</td>
<td>Apply J qualifier to detects and UJ qualifier to non detects of parent sample only</td>
</tr>
</tbody>
</table>
Extracted Internal Standards (Isotope Dilution Analytes)

Problematic analytes (e.g. PFBA, PFPeA, fluorotelomer sulfonates) can have wider recoveries without qualification. Qualify corresponding native compounds with a J flag if outside of the range.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>Recovery &lt;50% or &gt;150%</td>
<td>Apply J qualifier</td>
</tr>
<tr>
<td>Recovery &lt;25% or &gt;150% for poor responding analytes</td>
<td>Apply J qualifier</td>
</tr>
<tr>
<td>Isotope Dilution Analyte (IDA) Recovery &lt;10%</td>
<td>Reject results</td>
</tr>
</tbody>
</table>

Secondary Ion Transition Monitoring

Quantifier and qualifier ions should be monitored for all target analytes (PFBA and PFPeA are exceptions). The ratio of quantifier ion response to qualifier ion response should be calculated from the standards for each target analyte. Lab derived criteria should be used to determine if the ratios are acceptable. If the ratios fall outside of the laboratory criteria, qualify results as an estimated maximum concentration.

Signal to Noise Ratio

The signal to noise ratio for the quantifier ion should be at least 3:1. If the ratio is less than 3:1, the peak is discernable from the baseline noise and symmetrical, the result can be reported. If the peak appears to be baseline noise and/or the shape is irregular, qualify the result as tentatively identified.

Branched and Linear Isomers

Observed branched isomers in the sample that do not have a qualitative or quantitative standard should be noted and the analyte should be qualified as biased low in the final data review summary report. Note: The branched isomer peak should also be present in the secondary ion transition.

Reporting Limits

If project-specific reporting limits were not met, please indicate that in the report along with the reason (e.g. over dilution, dilution for non-target analytes, high sediment in aqueous samples).

Peak Integrations

Target analyte peaks should be integrated properly and consistently when compared to standards. Ensure branched isomer peaks are included for PFAS where standards are available. Inconsistencies should be brought to the attention of the laboratory or identified in the data review summary report.