

The PFAS Quality Assurance Project Plan, included below, was developed in 2022 for DEQ's special monitoring study to provide reconnaissance information on the occurrence, distribution, and concentrations of PFAS in the rivers and streams of Virginia. While some of the information below is specific to DEQ monitoring processes, the general monitoring process outlined in the QAPP, the PFAS sampling guidelines (Appendix A), and the PFAS sample shipping protocols (Appendix D) may be relevant and useful to public and private entities establishing PFAS monitoring plans.

Questions on monitoring processes outlined in the PFAS special study QAPP can be directed to [pfas@deq.virginia.gov](mailto:pfas@deq.virginia.gov).

## Quality Assurance Project Plan:



Reconnaissance of Per- and Polyfluoroalkyl Substances (PFAS) in Rivers and Streams of Virginia

Special Study Number: 220359

Commonwealth of Virginia  
Departmental of Environmental Quality  
Richmond, Virginia

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**1.1. Title and Approval Sheet:**

TITLE	NAME	DATE	APPROVAL
DEQ Office of Ecology Director	Bryant Thomas	8/29/2022	Bryant Thomas
VA DEQ Project Manager	Andrew Garey	12/7/2022	Andrew L. Garey
USGS Project Manager	Doug Chambers	10/16/2023	
VADEQ Water Quality Monitoring QA Coordinator	Reid Downer	8/29/2022	
SGS AXYS Laboratory Quality Assurance Manager	Rhonda Stoddard	10/16/2023	

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**1.3. Distribution List**

<b>TITLE</b>	<b>NAME</b>
Office of Ecology Water Quality Monitoring & Assessment Program Director	Bryant Thomas
Project Manager	Andrew Garey
VADEQ Water Quality Monitoring Quality Assurance Officer	Reid Downer
USGS Project Manager	Doug Chambers
SGS AXYS Laboratory Quality Assurance Manager	Rhonda Stoddard
VADEQ Sampling Team/Field Crew Leaders	Jason Hill Emma Jones

#### 1.4. Project Organization and Responsibility

**Table 1.** VADEQ Project organization and responsibility.

Role	Personnel
<u>Project Management</u> - Supervises conductance of the project in accordance with this quality assurance project plan and DEQ WQM Standard Operating Procedures (SOP) Manual, where applicable (DEQ 2020).	Andrew Garey
<u>Project Implementation</u> - Manages day to day operation of the program. Manages contract with the laboratory.	Jason Hill Emma Jones Roger Stewart Doug Chambers
<u>Project Design</u> - Project plan design and study preparation.	Andrew Garey Doug Chambers Jason Hill Emma Jones
<u>Sampling team</u> - Conduct all office and field related duties directly affecting sample collection and handling.	Central Office, and Regional Office staff USGS water monitoring staff
<u>Project Quality Assurance Officers</u> - Evaluates QA/QC procedures in the field and makes recommendations for corrective action.	Emma Jones Jason Hill
<u>Agency Quality Assurance Officer</u> - Develops the QAPP. Provides additional data review, audits and other QA/QC assistance as needed.	Reid Downer
<u>Partner Liaison Officers</u> - Coordinates activities between VADEQ and USGS and between the VADEQ contract laboratory. This includes scheduling delivery of needed supplies and equipment from USGS to VADEQ offices, sample collection and sample shipment or delivery based on laboratory capabilities.	Roger Stewart Emma Jones Andrew Garey
<u>Data Review and Reporting</u> - Reviews data and writes summary and annual reports.	Andrew Garey Doug Chambers
<u>SGS AXYS Laboratory Quality Assurance Manager</u>	Rhonda Stoddard

## 1.5. Problem Definition/Background

Per- and polyfluoroalkyl substances (PFAS) are a group of chemical compounds that have been produced for a wide variety of uses in consumer products and industrial applications. The use of PFAS chemicals and products containing PFAS is widespread and common. The components of PFAS compounds are long-lasting and slow to break down. As such, they are persistent in the environment and have been detected in water, air, and soil. A number of PFAS compounds have been detected in the blood of humans and animals, where they persist and bioaccumulate. The USEPA has issued drinking water health advisories for perfluorooctanoic acid (PFOA) and perfluorooctane-sulfonic acid (PFOS), two of the thousands of PFAS chemicals (USEPA 2016a, USEPA 2016b). PFOA and PFOS were linked to adverse health effects in animals in laboratory trials, and numerous studies have suggested that some PFAS compounds may lead to adverse health effects in humans. PFAS has been detected in organisms, sediment and water in Virginia (e.g. recent detection in waters of in the Middle Chickahominy River Watershed); however, comprehensive data are lacking and, therefore, the occurrence and concentrations of PFAS in Virginia rivers and streams is currently largely unknown (reviewed by Xia 2022).

### 1.5.1. Purpose of Study

This project will provide reconnaissance information on the occurrence, distribution, and concentrations of PFAS in the rivers and streams of Virginia. Sampling will occur at both targeted and probabilistic (i.e. random) sites across the state as well as at existing USGS sampling stations in Virginia. Targeted sites were selected to represent waterbodies in areas where PFAS compounds may be likely to occur due to their proximity to suspected sources, which includes production or use in industrial facilities, dispersal of fire-fighting chemicals at airports and/or military installations, or in waste (i.e. landfills). Sites from the DEQ Freshwater Probabilistic Monitoring (ProbMon) program were selected randomly to provide broad representativeness to streams across the state. A subset of new, wadeable ProbMon sites will be sampled for PFAS. USGS Chesapeake Bay River Input Monitoring Network (RIM) and Non-tidal Network (NTN) sites are established sampling sites with ongoing routine (monthly) and storm-targeted sampling across the Chesapeake Bay Watershed in Virginia. The RIM and NTN data will provide information on the short-term (i.e. within one year), temporal variability in PFAS concentrations, and on the effects of storm flow inputs and elevated discharge. Collectively, the targeted, ProbMon, RIM and NTN datasets will provide statewide more comprehensive information on the status of PFAS in Virginia, which will guide future studies and monitoring programs.

## 1.6. Work Schedule

The activities will occur over a period of one sampling season in 2022. Table 2 describes the project work schedule.

**Table 2.** Work Schedule

Task	Description	Schedule
1	QAPP Development/Approval	April/May 2022
2	Establish stations, determine sample collection logistics	January-March 2022
3	Collect PFAS samples.	Spring 2022
4	Laboratory processing and analysis of samples as received.	Begin Spring 2022
5	Data validation	Begin Summer 2022



6	USGS published data release	Fall 2022
7	DEQ summary analysis/report	Fall-Winter 2022

## 1.7. Quality Assurance Objectives

Data from PFAS sampling in this study will be used to evaluate the current baseline distribution and concentration of PFAS in surface waters, which will be used to inform future DEQ PFAS monitoring and studies in the state. Both field and laboratory personnel will work to achieve the highest possible level of confidence in the quality of study results by using established procedures to ensure the accuracy, precision, representativeness, comparability, and completeness of the data. For a list of the target analytes and their respective reporting levels, please refer to Table 5 below.

Accuracy, precision, detection limits, and completeness for the chemical analyses are addressed in established technical procedures utilized by the contract laboratories. Preventive maintenance and calibration is performed on all sampling equipment per manufacturer's instructions.

## 1.8. Personnel Training Requirement

### 1.8.1. Field Staff

Proper training of field personnel represents a critical aspect of quality control. Field technicians are trained to conduct a wide variety of activities using standardized protocols to ensure comparability in data collection among crews and across geographic areas. Field crews receive training relative to the operation and maintenance of the field equipment used throughout the duration of the field program. In addition to training for regular DEQ water quality monitoring, field crews collecting PFAS samples have completed a training session on PFAS-specific sampling protocols.

Field equipment operation can be found in Section 2.3 below and the Safety Plan is located in Appendix C.

## 1.9. Documentation and Records

Documentation of field and laboratory data is to be stored in the Comprehensive Environmental Data System (CEDS) Water Quality Module. USGS will serve as the primary liaison with the contract laboratory. For samples collected by USGS and those collected by VADEQ, USGS will coordinate submission of field and laboratory documentation to the contract laboratory. USGS will initially receive and review laboratory documentation submitted to them from the contract laboratory (i.e. analytical results and supporting documents). After review of this laboratory documentation, USGS will submit it to VADEQ, initially as provisional, and later as final, depending on the level of review and completion of the Quality Control (QC) and Quality Assurance (QA) process.

### 1.9.1. Field Documentation

Field documentation will consist of field notes, data sheets, chain of custody forms, sample labeling, and shipping forms. For detailed information on field documentation, see the PFAS Sampling Guidelines (Appendix A) and shipping protocols (Appendix D). For details on general sampling guidelines, see the latest version of Virginia DEQ's [WQM SOP](#) manual (VADEQ, 2020) and the USGS [National Field Manual](#) for the Collection of Water-Quality Data (USGS, variously dated).

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### **1.9.2. Laboratory Documentation**

Laboratory documentation will include:

1. Electronic copies of results, to include certificates of analysis, and printed copies of documentation when specifically requested.
2. Chain of Custody (COC) records.
3. Any other data associated with the measurement process when specifically requested.

### **1.9.3. Audit Reports**

Technical System Audits (TSA) will be conducted as needed by the field team coordinators during field activities or by the VADEQ QA Officer (or designee) during field and laboratory activities. The auditors will prepare a report that summarizes the observations and findings of each of these audits. As needed, the audit reports will be supplemented by a corrective action plan, to be implemented as soon as feasible, to correct each observation or finding of erroneous procedures.

### **1.9.4. Data Validation Reports**

After initial review of data on analytical results submitted by the laboratory, USGS will provide them as provisional to VADEQ. These provisional data are intended for internal review by USGS and VADEQ only. After comprehensive review of the data by both agencies and any changes made for quality assurance purposes, USGS will submit a final dataset to VADEQ and will release the data publicly. Unlike provisional data, these final data are approved by USGS for circulation, publication and for informing environmental management decisions. Data validation flags will be applied to those sample results that fall outside of quality control limits.

The contract laboratory's Quality Assurance Officer, at the request of VADEQ, will identify biases inherent in the data, including assessment of laboratory performance, and overall precision, accuracy, representativeness and completeness. The data validation report will address whether the quality of the flagged data affects the ability to use the data as intended. As needed, the data validation reports will be supplemented by a corrective action plan, to be implemented as soon as feasible, to correct each observation or finding of erroneous procedures.

## **2. MEASUREMENT / DATA ACQUISITION**

### **2.1. Experimental Design**

Sampling will occur at three types of sites (See Appendix B for full list):

1. DEQ ProbMon sites: These are randomly selected sites from the DEQ Freshwater Probabilistic Monitoring program. Sites are selected to broadly represent freshwater streams and rivers in Virginia. A single sample will be collected from 40 unique, wadeable ProbMon sites across Virginia. Each sampling season a few selected ProbMon sites cannot be sampled for a variety of reasons (e.g. access permission is denied, the water body is dry). Therefore, a subset of 42 new (i.e. not revisits) wadeable ProbMon sites will be randomly selected and identified as PFAS sampling sites. Selection will occur such that PFAS sites assigned to the staff of each VADEQ region are in proportion to the total number of ProbMon sites within the region (Table 3). See Appendix B for a complete list of DEQ ProbMon Sites (Table 3). If regions do not reach the numbers listed in Table A-2 at ProbMon sites due to lack of access, wadeable trend monitoring
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sites (i.e. regularly revisited sites) will serve as alternative sites until the requisite number of samples is reached. Project implementation staff (Table 1) will coordinate with regional field staff to adjust the sample totals allocated to each region so that a total of 40 samples are collected, each at a unique ProbMon or trend monitoring site.

2. DEQ Targeted sties: 14 pre-selected sites will each be sampled twice for PFAS by DEQ Monitoring staff (See Tables 3 and A-1).

**Table 3:** DEQ Freshwater ProbMon and targeted PFAS samples to be collected, by DEQ region. \*Note: the 42 ProbMon sites and samples shown represents an overdraw of 2 reserve sampling locations. A total of 40 ProbMon sites will be visited and 40 samples will be collected.

DEQ Region	ProbMon Sites			Targeted Sites		
	Sites*	Samples*	Blanks	Sites	Samples	Blanks
Blue Ridge	12	12	2	3	6	1
Northern	5	5	1	4	8	2
Piedmont	7	7	2	3	6	1
Tidewater	10	10	2	4	0	0
Southwest	3	3	1	0	8	2
Valley	5	5	2	0	0	0
<b>Total</b>	<b>42</b>	<b>42</b>	<b>10 (24%)</b>	<b>14</b>	<b>28</b>	<b>6 (21%)</b>

3. USGS RIM and NTN sties: 18 sites will be sampled by USGS monitoring staff (see Table A-3). Sampling will occur along with ongoing routine (monthly) and storm-targeted site visits. PFAS samples will be collected at routine sampling events every other month beginning in February, 2022 (February, April, June). Two storm-targeted samples will be collected at each site when streamflow is at least twice the recent normal flow by June 30, 2022. All USGS samples will be collected in accordance with the USGS National Field Manual for the Collection of Water Quality Data (USGS, variously dated). Field blanks will be collected at a rate of approximately 20% of samples.

## 2.2. PFAS Sampling Locations

See Appendix B for lists of sampling locations and locality information. The subset of 42 ProbMon sites will be selected from the full list of ProbMon sites. See Section 2.1 for detailed information on site selection. Project implementation staff (Table 1) will coordinate with regional field staff to adjust the sample totals allocated to each region so that a total of 40 samples are collected, each at a unique ProbMon or trend monitoring site.

## 2.3. Water Quality Sampling Methods

DEQ water monitors will follow sampling methodology from the PFAS Sampling Guidelines SOP document (Appendix A) in non-tidal streams. Wadeable ProbMon sites will all consist of direct grab samples. Targeted PFAS sample locations include a variety of waterbodies, including free-flowing streams, lakes, and tidal waters. Sample collection may entail direct grab samples, midstream bridge

grab samples, or other techniques aimed to obtain a representative sample from the waterbody. Virginia DEQ monitors will follow the sampling methodology outlined in the 2022 DEQ Water monitoring Strategy (VADEQ, 2022A) at targeted lake sites and the Virginia Chesapeake Bay Program tidal SOP (VADEQ, 2022B) at targeted tidal sites. USGS sampling will consist of depth-integrated composite samples and will follow procedures described in the USGS National Field Manual for the Collection of Water Quality Data (USGS, variously dated) and the PFAS Sampling Guidelines SOP (Appendix A). PFAS samples will be collected first if multiple water quality samples are to be collected during site visits to reduce the chance of contamination. See sample bottle information in Table 4.

**Table 4.** Sample containers and preservatives

Matrix	Parameters	Volume	Container	Preservation
Water	PFAS congeners (See Appendix C for full list)	500 ml	HDPE, with linerless HDPE or polypropylene caps	Ice (0 - 6°C)

### 2.3.1. Sample Handling and Processing

Field crews will use clean, double-gloved hands (full-arm vinyl gloves with nitrile gloves on top) to handle samples according to the PFAS Sampling Guidelines (Appendix A). Samples will be double bagged in plastic zipper-sealed bags. Sample labels will be affixed to the inner bag and not directly to the sample bottle. Once bagged, samples will immediately be placed in a cooler with sufficient wet ice to store the samples at a temperature between 0 - 6° C. PFAS samples will be sampled from Monday-Wednesday each week and will be shipped to the lab on Wednesdays to ensure the lab can receive and properly store them in a timely manner. All samples will remain in coolers with double-bagged wet ice throughout the shipping process. See Appendix D for detailed information on shipping protocols.

Once received, the laboratory must confirm that the sample temperature is 0 - 6° C. The laboratory will then store the samples at  $\leq -20$  °C until sample preparation. The contract laboratory will follow the protocols outlined for EPA analytical method 1633 (USEPA 2021).

### 2.3.2. Sample Transport

PFAS water samples will be stored between 0 - 6° C until shipped. SGS AXYS Analytical Services is providing shipping service with Federal Express. Detailed shipping instructions along with the shipping address and recipients can be found in Appendix D.

### 2.3.3. Sample Numbering System

Standard VADEQ sample identification procedures will be applied to each sample, including associated QC samples that are collected. Each stream station ID and sample date and time will be used as sample identification. Sampling identification procedures are found in the latest version of DEQ's WQM SOP manual (VADEQ, 2020).

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#### **2.3.4. Chain of Custody (COC) forms**

Completed COC forms will be required for all PFAS samples to be analyzed. At a minimum, the COC form will contain:

1. Unique sample identification
2. Sample collection date and time
3. Sample type (matrix)
4. Analysis requested (EPA draft method 1633)
5. Records of sample transfers as applicable – personnel receiving samples and date/time of transfer
6. Any remarks and observations relating to the sample.

The original COC form will accompany the samples to the SGS AXYS Analytical Services Ltd. Laboratory. The COC forms will remain with the samples at all times until the samples are delivered to the carrier.

A copy of the SGS AXYS COC form used in this study is found in Appendix E.

#### **2.3.5. Sample Packing and Shipping**

PFAS samples are to be shipped to SGS AXYS Analytical Services Ltd. laboratory for analysis. Sample packing and shipping procedures are designed to ensure that the samples and the COC forms will arrive at the lab intact and together. For specific instructions refer to Appendix D. Samples are double-bagged in zipper sealed bags, wrapped in bubblewrap, and placed in the cooler with enough bagged ice to completely fill the cooler. COC forms are placed in zipper-sealed bag and placed inside the cooler. Coolers are sealed with packing tape before shipping. It is assumed that samples in tape sealed ice chests are secure when being transported by carrier. The carrier is not required to sign the COC form. The receiving lab has a sample custodian who examines the samples for correct documents, proper preservation and sign off as recipient on the COC form. The samples will be securely maintained at the appropriate temperature until ready for analysis.

#### **2.3.6. Laboratory Sample Custody**

Laboratory sample custody will be performed in accordance with the lab's Quality Assurance manual.

### **2.4. Analytical method**

Analyses of samples will follow EPA draft method 1633 (USEPA 2021) for 40 PFAS compounds by SGS AXYS, a certified contract laboratory. Table 5 lists the analytical methods, holding times, and reporting limits. See Appendix C for a full list of PFAS analytes and method detection limits (MDL).

**Table 5.** Summary of Method Reference, Maximum Holding Time and Detection Limits

<b>Parameter</b>	<b>Reference</b>	<b>Holding Time</b>	<b>Min Detection Limit</b>
PFAS congener analytes (See Appendix C for full list of 40 PFAS analytes)	EPA Draft Method 1633	28 days at 0-6 °C 90 days at ≤ -20 °C	0.117 - 5.942 ng/l (See Appendix C for parameter-specific MDLs)

### **2.5. Quality Control**

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### **2.5.1. PFAS Quality Control Samples**

#### **2.5.1.1. PFAS Blank Samples**

Field blank samples will be collected to assess possible contamination in the sampling and sample handling processes. More than 20% of site visits will include a field blank sample. Field blanks will be collected by filling a clean sample container with certified clean laboratory-grade water.

#### **2.5.1.2. Laboratory Quality Control**

All laboratory QC samples will be analyzed in accordance with established standard laboratory methods, procedures and QA SOPs, and include an evaluation of accuracy, precision, representativeness and comparability.

### **2.6. Data Management**

Project data will include computer and handwritten entries. Field observations, measurements and records such as sample collection and shipping information will be recorded and retained on hardcopy forms. Data generated for those parameters analyzed by SGS AXYS will be provided within a laboratory report generated by the responsible laboratory personnel. Following validation and approval as described above, the report is sent electronically to VADEQ and USGS in accordance with the Scope of Work. All analytical results will be reviewed and approved by qualified VADEQ and USGS staff prior to release by USGS as final and will be published as a peer-reviewed, citable, and publicly-available USGS Data Release on sciencebase.gov. All USGS-collected sample results will also be loaded into the publicly-accessible USGS National Water Information System (NWIS; [waterdata.usgs.gov](http://waterdata.usgs.gov)).

## **3. Oversight and Assessment**

### **3.1. Assessments and Response Actions**

#### **3.1.1. Technical System Audits (TSA)**

TSA of field activities will be conducted by the field team coordinators and/or the VADEQ QA Officer. This will include:

1. Determining the availability and proper use of field equipment
2. Staff adherence to project-controlling documents for sample collection, identification, handling, and transport; proper collection and handling of QC samples.

TSA of laboratory operations will be performed by the contract laboratory QA Manager. Laboratory TSA tasks will include the review of sample handling procedures, internal sample tracking, SOPs, analytical data documentation, QA/QC protocols, and data reporting.

## **4. Data Validation and Usage**

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#### 4.1. Data Review

To ensure the project objectives were met, individually and collectively, and to estimate the effect of any QA/QC procedural deviations on the ability to use the data, each of the following areas will be reviewed by the VADEQ Project Manager and VADEQ QA Officer:

1. Sampling collection procedures
2. Sample handling
3. Analytical procedures
4. Quality control verification of field blanks

#### 4.2. Validation and Verification Methods

Data validation will be performed, reviewed, and interpreted by the VADEQ Project Manager and VADEQ QA Officer. The analytical laboratory report will be reviewed for compliance with the applicable method and for the quality of the data reported. The VADEQ QA Officer will identify sample biases by reviewing overall precision and accuracy of analysis, data representativeness and completeness. The Laboratory QA Officer will note data validation flags in the form of Remark Codes, to those sample results that fall outside of the QC acceptance criteria.

#### 4.3. Data Evaluation and Usage

After the project is complete, the Project Manager is responsible for collecting all the data, analyzing the results and preparing a summary report. See section 2.6 for information on data management and storage.

##### 4.3.3 Data Usage

The data generated will be used by DEQ to evaluate PFAS concentrations across the state. The data collected may establish the need for future special studies, based on the findings. Once the data are released as final by USGS and made publicly available, the data may be used by members of the public. Interested parties should contact the VADEQ QA Officer or Project Manager for information on the proper use and limitations of the data.

## 5.0 REFERENCES

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- USEPA 2021. Draft Method 1633; Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS. August 2021. Washington, DC.
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**APPENDIX A:**

**PFAS Sampling Guidelines (SOP)**

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## PFAS Sampling guidelines

The collection of water samples to be analyzed for per- and polyfluoroalkyl substances (PFAS) does not significantly differ from the collection of samples for other analytes. However, the collection of PFAS samples differs from sampling most common analytes largely in the level of attention given to eliminating as many potential sources of sample contamination as possible. The analytical methods routinely employed have limits of quantification in the sub parts-per-billion range, increasing the risk of biasing samples with inclusion of PFAS substances that are extraneous to the sampled medium. This is compounded by the near ubiquity of PFAS substances, they are found in a startling range of industrial and consumer products.

Reducing the likelihood of sample contamination can be accomplished through the following:

- Appropriate sampling technique
- Selecting of sampling equipment
- Sample equipment cleaning
- Field gear selection
- Sample handling

By carefully examining the potential sources of PFAS that may be present in the sampling process and eliminating or reducing them, the associated sources of bias can be mitigated. Additionally, collection and processing of quality-assurance samples, primarily blanks, can be used to characterize and quantify bias in PFAS sampling.

### Appropriate sampling technique

Selecting the appropriate sampling technique is primarily driven by project needs and data-quality objectives. Secondly, site conditions can dictate which sampling techniques are used. Given the project needs, data-quality objectives, and site conditions that exist for the VDEQ PFAS survey, a combination of grab-sample and depth- and width-integrated sampling techniques will be used. Grab samples can be collected for smaller, well-mixed stream locations. As stream size increases and/or streams become less well mixed, depth- and width-integrated sampling techniques will aid in collecting a representative sample. DEQ monitors will collect direct grab samples at ProbMon and targeted sites, while USGS will collect depth- and width-integrated samples where appropriate. Each technique is discussed in the following section.

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### Grab samples

Grab (or dip) samples are quick, easy, and require little equipment. This method is used when isokinetic samplers are impractical, such as when streams are too shallow or velocities are below sampler minimum requirements, or when project needs can be met using this technique. The error introduced by this method can be significant if suspended constituents are not evenly distributed in the stream.

#### Steps for collecting a grab sample (DEQ sampling at ProbMon and targeted sites):

In non-tidal, lotic systems, grab samples should be taken at the point along a perpendicular cross-section where the centroid of flow is located. The centroid of flow is the point in the stream where the greatest volume of water is moving. This is typically the deepest and fastest section on the cross-section. In lakes and tidal systems, sample locations may be set at the same locations chosen for the other water quality measurements (e.g. dissolved oxygen, nutrients). Samples should be collected from 0.3m below the water surface per agency Standard Operating procedures (VADEQ 2020).

Prepare provided sample bottles. Double bag bottles in zipper lock plastic bags.

Carefully wade to in an upstream direction to a spot just downstream of the sampling location in the centroid of flow.

Put on gloves. First put on elbow-length vinyl gloves, then standard nitrile gloves.

Remove first sample bottle from zipper-lock bag. Remove cap and submerge bottle. Fill bottle. Avoid disturbing the bottom, either by wading or with the bottle. Recap bottle. Place bottle in zipper-lock bag.

Repeat process for second bottle.

Keep samples at approximately 4° C.

#### Depth- and width-integrated samples (USGS sampling at RIM and NTN sites):

Samples collected using depth- and width-integrated sampling techniques and using isokinetic samplers better represent stream water quality, especially in regard to suspended matter. PFAS compounds can sorb to suspended particulate matter and can form micelles at high concentrations, mimicking suspended particulate matter, therefore using isokinetic samplers and depth- and width-integrating techniques will result in more representative samples than other sampling approaches.

As VDEQ staff are trained in the collection of depth- and width-integrated samples using the equal-width increment method (EWI), the following instructions for PFAS sampling will focus on areas that differ from routine sampling.

Steps for collecting a depth- and width-integrated sample:

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Identify the cross section to be sampled. Divide the stream width into sections of equal width and identify the mid-section sampling point of each section. The USGS rule of thumbs is a minimum of 10 sections, agency or program requirements may differ.

Prepare the sampler for collection. Ensure that the sampler has no components consisting of a PFAS compound such as PTFE (polytetrafluoroethylene or Teflon). Rinse sampler three times with native water.

Wear gloves, elbow-length vinyl gloves, then standard nitrile gloves, during sample collection and processing.

Use “clean-hands/dirty-hands” protocols. Clean-hands touches only sample bottle, churn carrier, and churn. Dirty-hands touches the sampler, sampler suspension (wading rod, bridge crane).

Samples from individual vertical sections should be composited in a HDPE 8-L churn. The churn should be double bagged in PE bags and placed in a plastic “churn carrier” (10-gallon plastic garbage can with lid). The churn carrier should only be opened to add samples from individual verticals or for rinses.

After sampling cross section is complete move the churn in churn carrier to a suitable location (mobile water-quality lab, laboratory, etc.) for processing.

Decant PFAS sample first. Fill each of two lab-provided bottles.

Keep samples at approximately 4° C.

### Selection of sampling equipment used in PFAS sampling

The equipment needed for collecting grab samples is simple, the lab-provided bottles. Sample containers are 500 ml high density polyethylene (HDPE) bottles, with linerless HDPE or polypropylene caps.

The equipment required for depth- and width-integrated samples is more complex. The standard isokinetic samplers used for EWI samples are the DH-81, the DH-85, and the D-95. All three of these samplers use interchangeable sample bottles, nozzles, nozzle holders, and these three components are available in a variety of materials, including PFAS compounds. For sampling PFAS compounds, it is critical to ensure that all components are made of plastics such as HDPE or Delrin. Avoid components made of Teflon, PTFE, TFE, or PFA. Carefully inspect these sampler components to exclude parts made with PFAS compounds.

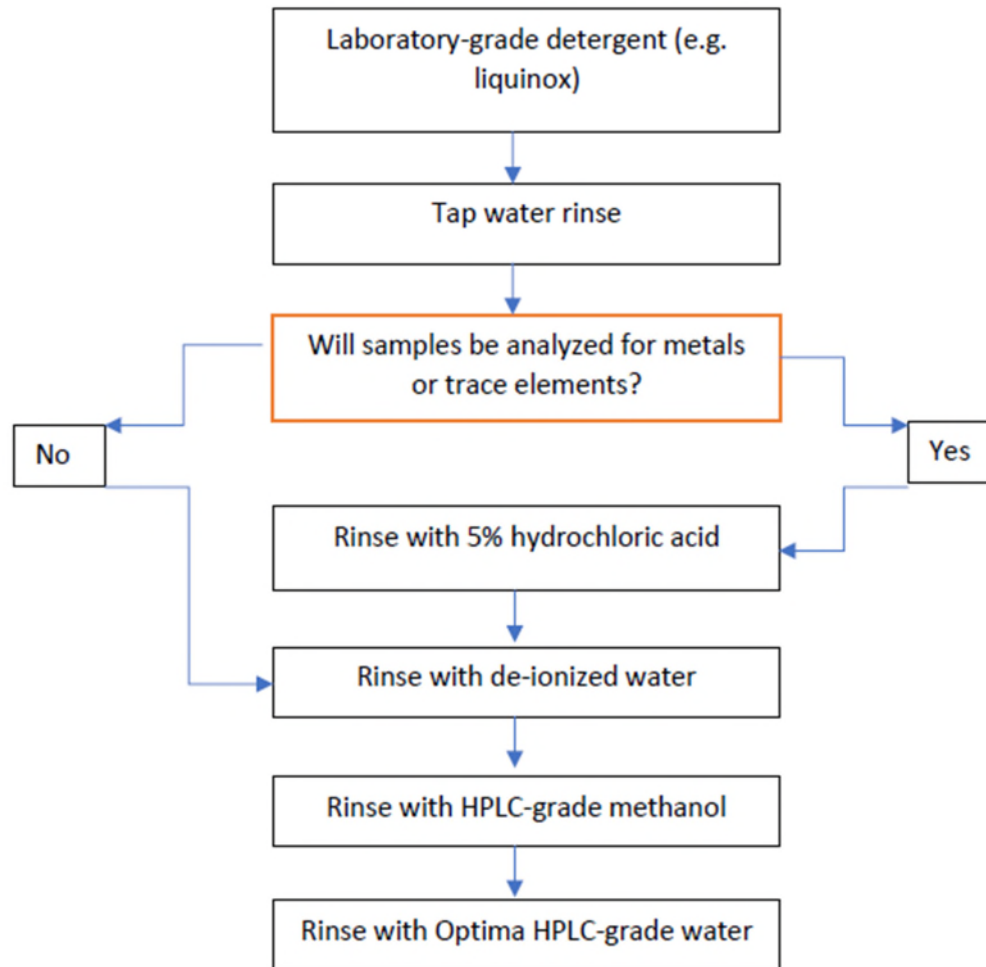
### Sampling equipment cleaning

Cleaning equipment that will be used to collect PFAS samples is similar to that used for sampling nutrients and suspended sediment, but with an additional cleaning step at the end of the process. The final steps of cleaning for PFAS sampling are a rinse with HPLC-grade methanol

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followed by rinsing with Optima HPLC-grade water. The general workflow for cleaning is illustrated below (Figure 1). Neutralize waste acid according to agency SOP. Collect waste methanol and properly dispose in accordance with agency policies.

Following cleaning, bottles, nozzle holders, and nozzles should be placed in polyethylene zipper-lock bags until use. Cleaned churns should be double bagged in polyethylene bags and placed in a clean churn carrier. Following cleaning, bottles, nozzle holders, and nozzles should be placed in polyethylene zipper-lock bags until use. Cleaned churns should be double bagged in polyethylene bags and placed in a clean churn carrier.



**Figure 1:** PFAS sampling equipment cleaning workflow

## Field Gear Selection

As mentioned previously, PFAS compounds have been incorporated in a broad range of both industrial and consumer products. The presence of PFAS compounds in commonly used products are a potential source of sample contamination and therefore a potential source of sample bias. Products to consider as potential sources of PFAS compounds include clothing, personal-care products, inks, coated papers, and food wrappers.

The risk of sample contamination from many of these sources can be mitigated through prudent lab and field practices. Chief among these is neither eating nor drinking while actively engaged in sampling. Eating or drinking should not occur in the sampling area or in the sample processing area. And sampling crews should wash their hands and get new gloves prior to resuming sampling or processing duties.

PFAS compounds have been used in the manufacture of many clothing items, especially breathable, waterproof items such as rainwear and waders. And many clothing items are treated with PFAS compounds to increase stain resistance. Therefore, it is recommended that the following items be avoided:

- Waterproof, breathable garments
  - Brands such as GoreTex
  - Breathable waders
  - Breathable rainwear
- New, unwashed clothing
- Clothing laundered or treated with:
  - Fabric softeners
  - Fabric protectors, including UV protection
  - Insect resistant chemicals
  - Water, dirt, and/or stain resistant chemicals

Allowable items include:

- Well-laundered synthetic or 100% cotton clothing, with most recent launderings not using fabric softeners
  - Rainwear and waders made of or with:
    - Polyurethane
    - Polyvinyl chloride (PVC)
    - Wax-coated fabrics
- .....

- Rubber / Neoprene
- Uncoated Tyvek®

Other commonly used field supplies that may include PFAS compounds are pens and waterproof paper. Items to be avoided include:

- Any PFAS-treated paper or notebook
- PostIt notes

Allowable items include:

- Loose paper (non-waterproof, nonrecycled)
- Rite in the Rain® notebooks
- Aluminum, polypropylene, or Masonite field clipboards
- Ballpoint pens, pencils, and Fine or Ultra-Fine Point Sharpie® markers

Personal-care products such as insect repellent and sunscreen can be used during sampling. However, care must be used. Insect repellents and sunscreen must be applied in an area removed from the sampling area, the processing area, and bottle and sampling equipment storage. Hands must be thoroughly washed following application of personal-care products.

## Sample Handling

All samples are to be double bagged and placed on ice and maintained at approximately 4° C. Use ice to keep samples chilled, do not use “blue ice”, “freezePaks” or other ice substitutes. Take care to ensure that samples are sufficiently separated from ice to prevent melt water from contaminating the samples.

Ship samples on ice to AXYS Laboratories overnight at the earliest practical time.



**APPENDIX B:**  
**Sampling locations**

**Table A-1.** Targeted PFAS sampling locations

Station ID	Location	Latitude	Longitude
9-NEW066.90	New River	37.185	-80.55694444
TBD	Roanoke River	37.234052	-80.177668
4AROA198.08	Roanoke River	37.249637	-79.867347
1aSOT001.44	South Run	38.748055	-77.686666
1aACO002.50	Accotink Creek	38.711112	-77.148891
1aCUB001.25	Cub Run	38.94611111	-77.43
TBD	Occoquan River	38.703774	-77.296383
2-CHK079.23	Chickahominy River	37.682	-77.568
TBD	James River	37.45329	-77.435519
2-KSL002.62	Kingsland Creek	37.40777778	-77.43416667
5BWNC010.02	West Neck Creek	36.7875	-76.041389
5BPCT001.79	Pocaty River	36.67333333	-76.1
5BNTW015.56	Northwest River	36.56638889	-76.26944444
7-POQ005.72	Poquoson River	37.13611111	-76.46416667

**Table A-2.** Possible Freshwater ProbMon PFAS sampling locations. PFAS will be sampled at subset of new, wadeable sites.

Station ID	Location	Latitude	Longitude
VA-2072	South Fork Roanoke River-Brake Branch	37.21286377	-80.23719167
VA-2081	Lower Sandy Creek	36.79214194	-79.11243392
VA-2084	Catawba Creek-Town Branch	37.49400328	-79.9207079
VA-2088	Cherrystone Creek	36.81036417	-79.35604369
VA-2091	New River-Bear Spring Branch	37.25281333	-80.61130174
VA-2095	Roanoke River-Beechtree Creek	37.12427586	-79.24420242
VA-2097	Aarons Creek-John H Kerr Reservoir	36.60931116	-78.71222248
VA-2100	Upper Sinking Creek	37.41783456	-80.29031725
VA-2101	Roanoke Creek-Spring Creek	37.11132549	-78.56404868
VA-2104	North Mayo River-Polebridge Creek	36.69790741	-80.16051929
VA-2111	North Otter Creek	37.46257083	-79.46782297
VA-2116	Dan River-Pumpkin Creek	36.57155397	-79.37603772
VA-2127	Pedlar River-Browns Creek	37.61831174	-79.25576057
VA-2129	Turnip Creek	37.0504757	-78.87081705
VA-2132	Jackson River-Falling Spring Creek	37.90037126	-79.98144527
VA-2133	James River-Alabama Creek	37.60736445	-78.81080567
VA-2136	Blackwater River-Madcap Creek	37.054335	-79.90118339
VA-2139	Little River-Brush Creek	37.05482028	-80.33978278
VA-2098	Broad Run-Rocky Branch	38.75051818	-77.57813486

VA-2102	South Anna River-Turkey Creek	37.75345054	-77.72093178
VA-2106	Carter Run	38.79418072	-77.93737406
VA-2114	Cameron Run	38.80398948	-77.08832664
VA-2118	Mattaponi River-Gravel Run	37.8272459	-77.12568448
VA-2131	Hughes River	38.55813783	-78.22768371
VA-2134	Rappahannock River-Rock Run	38.40857371	-77.66098688
VA-2073	James River-Picketts Creek	37.66425218	-78.07772247
VA-2074	Rappahannock River-Brockenbrough Creek	38.02204535	-76.92460064
VA-2077	Rowanty Creek	37.01361978	-77.43728306
VA-2082	Flat Rock Creek	36.92109857	-78.12602475
VA-2086	Lower Little River	37.92663041	-77.69602107
VA-2087	Pamunkey River-Mill Creek	37.54081302	-76.83217846
VA-2090	Rappahannock River-Peedee Creek	38.10982406	-77.00221463
VA-2093	James River-East Branch Tuckahoe Creek	37.55663009	-77.5610367
VA-2099	Piscataway Creek	37.87528489	-76.87034919
VA-2105	James River-Curles Creek	37.37736894	-77.30540408
VA-2109	Winterpock Creek	37.32595419	-77.72692381
VA-2113	South Meherrin River-Finneywood Creek	36.86969845	-78.44283593
VA-2117	Appomattox River-Fishpond Creek	37.40753982	-78.63776062
VA-2121	Herring Creek	37.35477119	-77.16543303
VA-2125	Buckskin Creek	36.95104438	-77.66341124
VA-2130	Reedy Creek	36.73923918	-77.70751671
VA-2137	Willis River-Bonbrook Creek	37.61769493	-78.23165741
VA-2138	Piscataway Creek	37.87715932	-76.99290667
VA-2076	Wolf Creek-Spoon Gap Creek	36.713398	-82.02432353
VA-2079	Nobusiness Creek	37.24012304	-80.92414424
VA-2080	Guest River-Toms Creek	36.91725416	-82.44393915
VA-2092	Middle Fork Holston River-Walker Creek	36.80525778	-81.69234073
VA-2107	New River-Rock Creek	36.62102083	-81.06249721
VA-2108	Lick Creek-Lynn Camp Creek	37.03158184	-81.38448694
VA-2120	Clinch River-Bull Run	36.85053191	-82.31660471
VA-2123	New River-Shorts Creek	36.86374407	-80.85962177
VA-2124	Russell Prater Creek	37.20942371	-82.26350335
VA-2128	North Fork Holston River-Roberts Creek	36.60944454	-82.51146719
VA-2140	North Fork Holston River-Little Creek	36.81558092	-82.10686124
VA-2071	Southwest Branch Back River	37.03326344	-76.36651544
VA-2075	Dismal Swamp-Lake Drummond	36.57705576	-76.40987721
VA-2083	Gargathy Inlet	37.75661494	-75.56966749
VA-2103	Blackwater River-Terrapin Swamp	37.01714591	-76.84458595
VA-2115	Lynnhaven River	36.8363993	-76.05929297
VA-2119	Mill Swamp-Darden Pond	36.64277476	-77.15395393

VA-2135	Nansemond River-Cedar Lake	36.7994519	-76.57404135
VA-2078	North Fork Shenandoah River-Narrow Passage Creek	38.79750352	-78.52245588
VA-2085	Rockfish River-Beaver Creek	37.75066704	-78.6841148
VA-2089	James River-Bear Garden Creek	37.71003567	-78.30905425
VA-2094	Cunningham Creek	37.8587564	-78.32050706
VA-2096	Jackson River-Warm Springs Run	38.12343365	-79.7797251
VA-2110	Mechums River-Stockton Creek	38.02302027	-78.68713067
VA-2112	Long Glade Creek	38.31480601	-79.03628174
VA-2122	Opequon Creek-Turkey Run	39.25348435	-78.05814862
VA-2126	South Fork Shenandoah River-Elk Run-Boone Run	38.44606858	-78.63426628

**Table A-3.** USGS NTN and RIM PFAS sampling locations.

VA DEQ Station ID	USGS STATION ID	Location	Latitude	Longitude
1BMDD00581	1621050	Muddy Creek at Mount Clinton, VA	38.48679	-78.9603075
1BSSF003.56	1631000	S F Shenandoah River at Front Royal, VA	38.914001	-78.2108339
1BSMT004.60	1632900	Smith Creek near New Market, VA	38.69345	-78.6427935
1BNFS010.34	1634000	N F Shenandoah River near Strasburg, VA	38.976776	-78.3361152
1ADIF000.86	1646000	Difficult Run near Great Falls, VA	38.975943	-77.2458144
1AACO014.57	1654000	Accotink Creek near Annandale, VA	38.812891	-77.2283158
1ASQQ006.73	1658500	S F Quantico Creek near Independent Hill, VA	38.587343	-77.4285958
3-RAP030.21	1665500	Rapidan River near Culpeper, VA	38.350408	-77.9749972
3-RPP113.37	1668000	Rappahannock River near Fredericksburg, VA	38.308462	-77.5291527
7-DRN010.48	1669520	Dragon Swamp at Mascot, VA	37.633753	-76.6963468
8-NAR005.42	1671020	North Anna River at Hart Corner near Doswell, VA	37.85014	-77.4277595
8-PMK082.34	1673000	Pamunkey River near Hanover, VA	37.767642	-77.3322018
8-PCT000.76	1674182	Polecat Creek at Route 301 near Penola, VA	37.960333	-77.3434444
8-MPN054.17	1674500	Mattaponi River near Beulahville, VA	37.883889	-77.1652778
2-JMS157.28	2035000	James River at Cartersville, VA	37.67098	-78.0858328
2-JMS113.20	2037500	James River near Richmond, VA	37.563202	-77.5469314
2-APP016.38	2041650	Appomattox River at Matoaca, VA	37.225151	-77.475263
2-CHK035.26	2042500	Chickahominy River near Providence Forge, VA	37.436258	-77.0608027

**APPENDIX C:**  
**Table of PFAS Analytes**

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**Table A-4.** PFAS congeners to be analyzed, method, and minimum detection limits (MDL).

PFAS Analyte	Storet Code	Analytical Method	MDL
Heptafluorobutyric acid	375-22-4	EPA Draft Method 1633	0.33 ng/l
Perfluorovaleric acid	2706-90-3	EPA Draft Method 1633	0.196 ng/l
Perfluoroheptanoic acid	375-85-9	EPA Draft Method 1633	0.221 ng/l
Perfluorooctanoic acid	335-67-1	EPA Draft Method 1633	0.302 ng/l
Perfluorononanoic acid	375-95-1	EPA Draft Method 1633	0.221 ng/l
Perfluorodecanoic acid	335-76-2	EPA Draft Method 1633	0.333 ng/l
Perfluoroundecanoic acid	2058-94-8	EPA Draft Method 1633	0.264 ng/l
Perfluorododecanoic acid	307-55-1	EPA Draft Method 1633	0.379 ng/l
Perfluorotridecanoic acid	72629-94-8	EPA Draft Method 1633	0.238 ng/l
Perfluorotetradecanoic acid	376-06-7	EPA Draft Method 1633	0.264 ng/l
Perfluorobutanesulfonic acid	375-73-5	EPA Draft Method 1633	0.245 ng/l
1-Pentanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,5-undecafluoro-	2706-91-4	EPA Draft Method 1633	0.204 ng/l
Perfluorohexanesulfonic acid	355-46-4	EPA Draft Method 1633	0.217 ng/l
1-Heptanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,7-pentadecafluoro-	375-92-8	EPA Draft Method 1633	0.137 ng/l
Perfluorooctane sulfonic acid	1763-23-1	EPA Draft Method 1633	0.327 ng/l
1-Nonanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,9-nonadecafluoro-	68259-12-1	EPA Draft Method 1633	0.303 ng/l
1-Decanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-heneicosafluoro-	335-77-3	EPA Draft Method 1633	0.334 ng/l
Perfluorododecanesulfonic acid	79780-39-5	EPA Draft Method 1633	0.179 ng/l
Perfluorohexanoic acid	307-24-4	EPA Draft Method 1633	0.318 ng/l
1-Octanesulfonic acid, 3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluoro-	27619-97-2	EPA Draft Method 1633	3.973 ng/l
Fluorotelomer sulfonate 8:2	39108-34-4	EPA Draft Method 1633	1.566 ng/l
Perfluorooctanesulfonamide	754-91-6	EPA Draft Method 1633	0.227 ng/l
N-methylperfluoro-1-octanesulfonamide	31506-32-8	EPA Draft Method 1633	0.196 ng/l
Sulfluramid	4151-50-2	EPA Draft Method 1633	0.585 ng/l
N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	EPA Draft Method 1633	0.586 ng/l
N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	EPA Draft Method 1633	0.324 ng/l
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	EPA Draft Method 1633	1.191 ng/l
N-Ethyl-N-(2-hydroxyethyl)perfluorooctanesulfonamide	1691-99-2	EPA Draft Method 1633	1.022 ng/l
Hexafluoropropylene oxide dimer acid	13252-13-6	EPA Draft Method 1633	0.406 ng/l
Perfluoro(4-methoxybutanoic) acid	863090-89-5	EPA Draft Method 1633	0.117 ng/l
Perfluoro-3-methoxypropanoic acid	377-73-1	EPA Draft Method 1633	0.177 ng/l
Perfluoro-3,6-dioxaheptanoic acid	151772-58-6	EPA Draft Method 1633	1.384 ng/l
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid	756426-58-1	EPA Draft Method 1633	0.871 ng/l
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	EPA Draft Method 1633	0.819 ng/l

Hexanoic acid, 4,4,5,5,6,6,6-heptafluoro-	356-02-5	EPA Draft Method 1633	0.721 ng/l
4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	EPA Draft Method 1633	0.779 ng/l
Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	EPA Draft Method 1633	0.137 ng/l
Octanoic acid, 4,4,5,5,6,6,7,7,8,8,8-undecafluoro-	914637-49-3	EPA Draft Method 1633	5.066 ng/l
Decanoic acid, 4,4,5,5,6,6,7,7,8,8,9,9,10,10,10-pentadecafluoro-	812-70-4	EPA Draft Method 1633	5.942 ng/l
Fluorotelomer sulfonate 4:2	757124-72-4	EPA Draft Method 1633	2.281 ng/l

**APPENDIX D:**  
**PFAS Sample Shipping Protocol**

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**PFAS Shipping Procedure:**

1. Fill out one Contract ASR per sample, e.g. per bottle set (BLANK\_\_Contract ASR.docx). **Blanks require their own ASR.**
  - Fill in the STATION ID, BEGIN DATE, BEGIN TIME, MEDIUM CODE, SAMPLE TYPE, STATION NAME OR FIELD ID, ASR RELINQUISHED BY, DATE, and TIME fields.
  - Begin Date is the sample date. Begin time is the sample time.
  - For regular samples, Medium Code = WS and Sample Type = 9
  - For blank samples, Medium Code = OAQ and Sample Type = 2
  - **Make a copy of all the Contract ASRs and send one to the lab with the sample and email the other to Emma, Jason, Scott, and Drew to track the samples.**
2. Fill out the Commercial Invoice Form (BLANK\_\_Commercial Invoice.docx).
  - **THREE copies of this form need to be included with each cooler.**
3. Fill out the SGS analytical request form (BLANK\_\_SGS analytical request form.docx).
  - Use the provided example (EXAMPLE\_\_SGS analytical request form.pdf) to complete the form.
  - Sign the bottom Relinquished By field. Date and time need to be filled out.
  - **Waybill number provided by FedEx needs to be filled in at the bottom right corner.**
4. Complete the return shipping information on the cooler return form (BLANK\_\_Cooler\_Return.docx).
  - You only need to complete the DEQ Office address to communicate to the lab where to return the cooler.
5. Print the forms specified above and the return shipping information (EXAMPLE\_\_Shipping Label.pdf).  
**DO NOT PRINT DUPLEXED.** Print one sided only.
6. Take your unsealed coolers to FedEx and provide them the example shipping label.
  - **Shipper: name; Company: VA DEQ; Phone Number: ###-###-####; Declared value = Total Value on your Commercial Invoice Form**
  - **The FedEx account number you need to provide, if available**
7. Place all the paperwork (COC Release form, completed ASR forms, Commercial invoice, and Return shipping information) inside the ziplock inside the cooler.
8. Double check all the forms are inside the cooler.
9. Seal your cooler with tape: three wraps around each side of the latch.
10. Send a photo of the FedEx tracking number and associate DEQ StationIDs sampled to Emma, Jason, Scott, and Drew to track the samples



1. Prepare cooler by using a plastic garbage bag to line the cooler.



2. Place samples in larger zip lock bags with DEQ sample labels on the outside of the inner zip lock bags.



3. Bubble wrap the samples to protect them.



4. Fill zip lock bags with wet ice and label bags "ICE."



5. Place "ICE" bags inside cooler lined with garbage bag.



6. Place bubble wrapped samples inside cooler.



7. Place additional "ICE" bags inside cooler surrounding samples.



8. Tie garbage bag.



9. Place sample forms inside gallon zip lock bag inside cooler.



10. Take cooler to FedEx for shipping.

11. Seal cooler with tape three times around latch after completing FedEx shipment procedure (at the store).

12. FedEx Shipping label:

**FedEx International Air Waybill**  
Lettre de transport aérien internationale

**1 From / Please print and press hard / Expéditeur / Écrivez en caractères d'imprimerie et appuyez fermement**

Date: 08/29/2022  
Sender's Name: N° de compte FedEx de l'expéditeur  
Company: Nom de la société  
Address: Adresse  
City: Ville Province: CANADA Postal Code: Code postal  
Email Address: Adresse e-mail  
Internal Billing Reference: Référence pour la facturation interne

**2 To / Destinataire** ☐ Recipient's Delivery / Livraison résidentielle

Recipient's Name: **SAMPLE RECEIPT** Phone: **250-655-5800**  
Company: **SGS AXYS ANALYTICAL SERVICES**  
Address: **2045 MILLS RD W.**  
City: **SIDNEY** State: **BC**  
Country: **CANADA** ZIP/Postal Code: **V8L5X2**  
Email Address: Adresse e-mail  
Recipient's Tax ID Number Required for Customs Purposes: N° fiscal du destinataire aux fins de la douane

**3 Shipment Information / Informations sur l'envoi**

Commodity Description / Description de la marchandise: **DETAIL REQUIRED, PRINT IN ENGLISH / DETAIL REQUIS, EN ANGLAIS SVP**  
Number of Packages: Nombre total de colis: **1** Total Weight: Poids total: **1.00** kg  
Sender's Signature: Signature de l'expéditeur: **[Signature]**

**4 Express Package Service / Service colis express**  
NOTE: Service order has changed. Please select carefully. / NOTE: L'ordre des services a changé. Veuillez sélectionner avec attention.  
☐ FedEx International First ☐ FedEx International Priority ☐ FedEx International Economy

**5 Packaging / Emballage**  
☐ FedEx Envelope Enveloppe FedEx ☐ FedEx Pak Pak FedEx ☐ FedEx Box Boîte FedEx ☐ FedEx Tube Tube FedEx  
☐ FedEx 10kg Box / Boîte 10kg FedEx ☐ FedEx 25kg Box / Boîte 25kg FedEx ☐ Other Autre

**6 Special Handling and Delivery Signature Options / Options de manutention spéciale et de signature de livraison**  
☐ HOLD at FedEx Location RETENIR à la succursale FedEx ☐ SATURDAY Delivery / Livraison le SAMEDI  
☐ Direct Signature / Signature directe ☐ Indirect Signature / Signature indirecte  
☐ Signature of Shipper / Signature du destinataire ☐ Signature of Recipient / Signature du destinataire

**7 Payment / Paiement**  
Bill transportation charges to: Facturer le transport à: ☒ Shipper / Expéditeur ☐ Addressee / Destinataire  
Bill Customs charges to: Facturer les droits de douane à: ☐ Shipper / Expéditeur ☒ Addressee / Destinataire

**8 Required Signature / Signature requise**  
Signature of Shipper: Signature de l'expéditeur: **[Signature]**

**Sender's Copy / Copie expéditeur**

For questions or to ship and track packages, visit [fedex.ca](http://fedex.ca).  
Visitez [fedex.ca](http://fedex.ca) si vous avez des questions, désirez expédier un colis ou faire le suivi de vos envois.  
Or call 1.800.GoFedEx (1.800.463.3339). Terms and conditions of service may vary from country to country. / Ou appelez 1.800.GoFedEx (1.800.463.3339). Les modalités de service peuvent varier selon les pays.

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**APPENDIX E:**

**SGS AXYS Chain of Custody Form**

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## CHAIN OF CUSTODY

2045 Mills Road West TEL: (250) 655-5800 TOLL FREE 1-888-373-0881  
Sidney, British Columbia, Canada V8L 5X2 FAX: (250) 655-5811

SGS AXYS CLIENT #:

REPORT TO:			INVOICE TO:			ANALYSIS REQUESTED				
Company _____			Company _____							
Address _____			Address _____							
_____			_____							
Contact _____			Contact _____							
Phone _____			Phone _____							
FAX _____			FAX _____							
E-mail _____			E-mail _____							
Project Name/Number: _____			Sampler's Name: _____							
Signature: _____										
Client Sample Identification	Matrix	Sampling Date	Sampling Time	Container Type/No.	SGS AXYS Lab Sample ID (Lab use only)					
Relinquished by (Signature)	Date	Time	Received by (Signature)			Courier		Waybill No.		
			Date			Time				
Relinquished by (Signature)	Date	Time	Received by (Signature)			Sample Receipt				
			Date			Time				
Remarks						Temp °C		Cooler		
						Custody Seal #				
						Seal Intact Y / N				
						Sample Tags		Y / N		

**APPENDIX F:**

**USGS Analytical Services Request (ASR) Form**

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[illegible]

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**CHAIN OF CUSTODY RECORD**

ASR: Relinquished by:		Date:		Time:	
ASR: Received by:		Date:		Time:	

**APPENDIX G:**  
**Commercial Invoice Form**

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## COMMERCIAL INVOICE

Date of Exportation:		EXPORTER REFERENCE (i.e., Order #, Invoice #, etc.):					
SHIPPER/EXPORTER (complete name and address):		CONSIGNEE (complete name and address): SGS AXYS Analytical Services Ltd. 2045 Mills Road Sidney, BC V8L 5X2 Canada 250.655.5800 <b>Importer of Record: SGS Axys Analytical</b>					
Country of Export:		REASON FOR SHIPMENT: <b>HS Code 3822.00</b> <b>Analytical Samples for Scientific Testing Purposes only.</b> <b>Not for human consumption.</b> <b>Not for re-sale.</b> <b>No Commercial Value.</b> <b>Soil Permit # P-2021-04057</b>					
Country of Manufacture:							
Country of Ultimate Destination:							
USA							
USA							
Canada							
International Air Waybill (Tracking) No.:							
No. of PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	Qty.	UNIT OF MEASURE		UNIT VALUE	TOTAL VALUE
1	Cooler	500mL HDPE plastic bottles containing Canadian Springs water for Scientific Testing Purposes only. <b>HS CODE 2201.90.00.</b> <b>Not for human consumption.</b> <b>Not for re-sale. No commercial value.</b>		Each		\$5.00	
		500mL HDPE Plastic Bottles Containing Aqueous Samples for Scientific Testing Purposes Only. <b>HS Code 3822.00.</b> <b>Not for Human Consumption. Not for Resale. No Commercial Value</b>		Each		\$5.00	
		250mL HDPE jars containing Sediment Samples for Scientific Testing only. <b>H.S. Code 3822.00.</b> <b>Soil PERMIT # P-2021-04057</b> <b>Not for human consumption. Not for re-sale. No commercial value.</b>		Each		\$5.00	
TOTAL No. of PKGS							TOTAL INVOICE VALUE
1							

COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN.

DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT.

SIGNATURE OF SHIPPER/EXPORTER (Type name and title, and sign)

DATE

**APPENDIX H:**

**SGS AXYS Cooler Return Form**

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PLEASE RETURN COOLER TO:

DEQ Office Address:

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**FedEx Account Number: if available**