Inactive Hazardous Sites Branch

Guidelines for Assessment and Cleanup of Contaminated Sites

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Department of Environmental Quality
Division of Waste Management
Superfund Section
Inactive Hazardous Sites Branch

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Registered Environmental Consultant Program website:
https://deq.nc.gov/about/divisions/waste-management/superfund-section/registered-environment-consultant-program

Updates since September 2022 Version:

1. Section 1.2. Clarified that the groundwater remedial action initiation milestone (2 years from completion of the remedial investigation) applies to risk-based remedies. Initiation is considered to be on the date the REC certifies (approves) the remedial action plan.
2. Section 1.6. Added "The Branch encourages remediating parties conducting independent work to submit data and reports, as they are received to keep the site file up to date."
3. Section 2.1.2. Added "The DEQ Risk Calculator may also be used to calculate cumulative risk for residential/unrestricted use at each sampling point to confirm delineation of soil contamination."
4. Section 2.2. Added the requirement to include copies of all field logs and notes to document the investigation to mirror the requirements in IHSB's administrative agreements.
5. Section 3.1.1.3. "Calculating Cumulative Risk" was added.
6. Section 3.4.1.1. "USEPA Proposed MCLs" for PFAS were provided.
7. Section 3.5 on naturally occurring background levels now states, "The Branch does not allow comparison with published background values alone" and "the 95% upper confidence level calculation is preferred by the Branch."
8. Appendix B. Section B.9 was added to include procedures for "Sampling and Analyses for Fill Imported from Another Property".
9. Appendix C. The USEPA Questions and Answers document, dated July 18, 2022, was replaced with the updated FAQ document, dated March 14, 2023
10. Appendix D. "Sensitive Environment Contacts" were verified or updated.

Updates since July 2021 Version:

1. Section 2.1.1. Added that phases of work already approved by the Branch do not need to be repeated in the REC Program.
2. Section 2.1.2. Changed the receptor survey update interval from 3 years to 5 years to be consistent with the REC Rules and added "depending on changes in plume extent and/or land use".
3. Guidelines for PFAS assessment are included in pertinent sections of the document.
4. Sections 4.2 and 4.3. Clarified that in the REC Program, according to the readopted rules, the contents of a pre-construction report and construction completion report can be included in the remedial action plan or the first progress monitoring report as appropriate.
5. Section 4.5. Added the suggestion to request monitoring well abandonment and no further action status in the Remedial Action Completion Report.
6. Section 5. Added the annual inspection and certification requirements for sites with land-use restrictions.

7. New Appendix C. USEPA PFAS resources and the Interstate Technology & Regulatory Council (ITRC) guidance for PFAS Sampling and Analytical Methods.
8. The former Appendix C is now Appendix D with updated contact information for identifying sensitive environments.

Updates since January 2020 Version:

1. Section 3.1.2. Added the option of using a twenty-times method for determining leachability of soil.
2. Sections 3.5 and 3.6. Separated into unique sections the use of naturally occurring background levels and practical quantitation limits, respectively, as remedial goals.
3. Updated REC rule references throughout according to the readopted rules effective July 1, 2020.
4. Appendix A. Updated the REC Program document certification requirements according to the readopted rules effective July 1, 2020.
5. Appendix B. Added information pertaining to the analysis of 1,4-Dioxane.
6. Appendix C. Updated the contact information for identifying sensitive environments.

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1.0 General Information

1.1 Statutory Authority and Jurisdiction

Links to Program information:
Inactive Hazardous Sites Program

REC Program

Inactive Hazardous Sites Statutes and Rules

The Inactive Hazardous Sites Response Act of 1987 (N.C.G.S. 130A-310 *et. Seq*) was established by the North Carolina General Assembly to address releases to the environment of hazardous substances, as defined in CERCLA/SARA. Parties responsible under law for the releases must assess and clean up these contaminated sites. The Inactive Hazardous Sites Branch (Branch) is responsible for oversight and approval of the assessment and remediation activities conducted by remediating parties and their environmental consultants. These sites include historical and recent accidental releases of hazardous substances and contamination in, or threatening, groundwater. Excluded are discharges associated with DEQ permits, hazardous waste dumping, proper application of agricultural chemicals, federal remediation sites, petroleum releases and sites undergoing remediation by the State's Drycleaning Solvent Cleanup Act (DSCA) Program. The Branch oversees remedial actions, conducts necessary enforcement at high-risk (highest priority) sites, and conducts the work itself at orphaned sites when state funds are available.

The Inactive Hazardous Sites Response Act provides site owners, operators, or responsible parties an opportunity to voluntarily clean up inactive hazardous substance or waste disposal sites with Branch approval. The Branch must dedicate its staff resources toward overseeing assessment and remedial actions at the sites that pose the highest risk to human health and the environment. At sites not designated as the Branch's highest priorities, a party may still proceed with an approved voluntary cleanup by working through the Branch's privatized oversight program known as the Registered Environmental Consultant (REC) Program. Voluntary remediators will be notified by the Branch whether their site is eligible for the REC Program.

1.2 The Registered Environmental Consultant Program

The Branch performs audits of the REC's work for compliance with the REC Rules and protection of public health. The audits also help the Branch improve procedures, guidance documents, and training for RECs.

Due to the large number of contaminated sites, the Branch is unable to respond to all requests for remedial action oversight. To help address this problem, the North Carolina General Assembly amended the Inactive Hazardous Response Act in 1994 and 1995 to establish a program for remediating parties to pursue an approved voluntary cleanup at lower-risk sites. In this privatized program, the remediating party hires an REC to both perform and certify a voluntary cleanup in place of state oversight. A list of approved RECs may be found on the REC Program website. Rules for implementing the REC Program (15A NCAC 13C .0300) specify the minimum qualification requirements for RECs and their Registered Site Managers (RSMs) and the administrative and technical requirements for conducting voluntary remedial actions in the program. All RSMs must familiarize themselves with the REC rules and this guidance to ensure remedial action regulatory compliance. Administrative requirements include REC certification of required document content and of work phase completion, meeting work-phase completion milestones (see Section 1.7) and submitting documents for the public record.

The REC and its RSMs shall recognize their primary obligation is to protect public health, safety, and welfare and the environment in the performance of professional services as an REC and to comply with the standards of professional responsibility specified in REC Rule 15A NCAC 13C .0305.

Only Branch-approved RSMs may manage site cleanups and make certifications on behalf of the REC. An RSM is responsible for the day-to-day oversight of the project. This responsibility may not be delegated to anyone else that is not an approved RSM. Inquiries to the Branch from the REC should come from the RSM. When an REC learns that an RSM intends to change employment, the REC must notify the Branch within 30 days.

Where there are rule violations, the Branch may conduct enforcement actions. The Branch may also revoke its approval of REC oversight of a voluntary remedial action and assume direct oversight if the voluntary remedial action is not being properly implemented.

Additional resources to help with REC Program compliance can be found on the REC Program website. The website includes links to the REC Program rules, certification forms, document content checklists, which can be used as a tool to guide the RSM through the REC Program planning and documentation requirements, and links to other guidance.

The following deadlines are specified in the REC Program Rules based on the effective date of an administrative agreement:

- Remedial investigation completion – 3 years
- Non-groundwater (soil, sediment and surface water) remedial action completion – 8 years
- Groundwater remedial action initiation – 2 years from completion of the remedial investigation. Initiation is considered to be on the date the REC certifies (approves) the remedial action plan (RAP) for risk-based or monitored natural attenuation remedies and on the date remedial equipment has been installed and initial physical or chemical treatment actions have begun for active remedies.

For active remedies, remedial action progress reports shall be submitted quarterly until one year after the construction of the remedy is complete. After the first year of progress reporting, or if the remedy includes no construction component, remedial action progress reports shall be submitted annually until remedial action is complete.

1.3 New Site Notification

The Inactive Hazardous Sites Response Act (N.C.G.S. 130A-310.1(b)) requires that within 90 days of the date on which any owner, operator or responsible party knows or should know of the existence of an inactive hazardous substance or waste disposal site, the owner, operator or responsible party shall submit to the Branch all site data that is known or readily available to the owner, operator, or responsible party. A Site Notification Form and Instructions are available on the Branch's website.

1.4 Procedures for Initiating an Approved Remedial Action

Remediating parties wishing to receive approval for a voluntary cleanup should complete a Site Conditions Questionnaire (Questionnaire) with the aid of their environmental consultant. Branch staff will review the Questionnaire and evaluate the site's risk to determine whether the remediation should be directly overseen by Branch staff or by a REC hired by the remediator.

To participate in a voluntary cleanup program (Branch-staff supervised or REC Program), the remediating party must sign an Administrative Agreement and pay a fee as discussed in the following section. The Administrative Agreement will specify the requirements for site cleanups, including, but not limited to, the work to be performed, reporting requirements, and document certification requirements.

The importance of retaining a qualified and experienced environmental consultant cannot be overemphasized. Any work that would constitute the "practice of engineering" as defined by G.S. 89C shall be performed under the responsible charge of a professional engineer registered in the state of North Carolina. Any work that would constitute the "public practice of geology" as defined by G.S. 89E shall be performed under the responsible charge of a geologist licensed in the state of North Carolina. Select documents, or portions thereof may require the seal and signature of a licensed professional (e.g., a registered engineer for engineering design work and/or a licensed geologist for geologic evaluations).

1.5 Voluntary Remedial Action Fees

To participate in a voluntary cleanup program, the remediating party must pay a fee. The one-time fee for remedial action oversight of higher-risk sites by the Branch is $1,000.

The initial REC Program fee is currently $3,000 to offset the cost of the Branch's administration and auditing of the REC Program. Thereafter, an annual fee is adjusted each year to reflect the actual cost of the audit program and availability of remaining funds. The annual fee is usually lower than the initial fee. The fee to participate in either program must be paid to the Branch before an Administrative Agreement will be executed.

1.5.1 Quick Clean Procedures

An option exists to complete cleanup of mildly contaminated properties where soil is the only medium affected (groundwater meets 15A NCAC 02L standards), the contamination is limited in extent and/or concentration, and the remedy consists of land-use restrictions. A remedy that relies on land-use restrictions requires Branch approval, so a signed Administrative Agreement would be needed for restricted property use scenarios. Refer to the Methods to Quickly Close Soil-Only Sites on the Branch's website.

1.6 Independent Remedial Action

Parties that do not wish to perform an approved voluntary cleanup are not prohibited from performing remedial activities independently. Once the site is remediated to *unrestricted use* cleanup levels for all contaminated media, a remediating party can submit a no-further-action review request to the Branch. Refer to the No Further Action requirements on the Branch's website for procedures and fees. The Branch encourages remediating parties conducting independent work to submit data and reports, as they are received to keep the site file up to date.

Independent cleanups are performed without Branch or REC oversight and approval and are not eligible for more lenient, risk-based cleanup levels or the cap on remedial expenses set out in the statute. Risk-based cleanups of soil and/or groundwater that rely on institutional controls as part of the remedy must be performed under a signed Administrative Agreement.

1.7 Document Submittal and File Access

Sites with Branch oversight (non-REC) will need to submit both paper and electronic copies of work plans and reports. Documents may be e-mailed, but if they are large in size then a file-sharing system may be used.

All REC certified documents must be submitted electronically through the portal on the REC Program website unless otherwise requested by Branch staff. All documents should be submitted as one PDF file that includes any cover letters, appendices, and signed and notarized certifications.

Work plans and reports should be submitted to the Branch as follows:

- Non-REC – In accordance with the schedule in the administration agreement for voluntary cleanup
- REC – Within 30 days of completion (RSM certification)

All Branch files are stored electronically in an online document management system which can be accessed remotely. Instructions for accessing the system can be found here: http://deq.nc.gov/about/divisions/waste-management/superfund-section/sf-file-records.

2.0 Remedial Investigation

At any time during remedial activities, the Branch must be notified within 24-hours of discovery, or as soon as feasible, of the following:

1. Contamination that has migrated beyond the source property
2. Imminent Hazards
   a. uncontrolled access by children to the contaminated media
   b. contamination detected in water supply wells, or
   c. site contaminant vapors detected within structures
3. Threat to a sensitive environment

The notification can be by phone, email, or in a report. If notification is written, it should be expressed clearly up-front and not in the body of a document. Any delays should be shown to be unavoidable.

The remedial investigation (RI) involves planning the investigation, gathering information and analytical data, and reporting the findings in a RI report. At least two phases of work are generally necessary to complete the investigation. The purpose of the first phase is to identify all known or suspected releases of hazardous substances to the environment, characterize the nature of such releases, and collect sufficient sampling data to compile a list of contaminants of concern. Additional phases delineate the extent of contamination in all media, and evaluate the potential for imminent hazards such as exposure to contaminated soil, water supplies or indoor air.

Per-and polyfluoroalkyl substances (PFAS) are a complex group of chemicals. PFAS are found in the environment worldwide due to their widespread use in products such as firefighting foams, textiles, food packaging, non-stick coatings, and waterproof fabrics. Because sampling and analytical methods, fate and transport properties and toxicity data are evolving rapidly, the remedial investigation of sites with known or potential PFAS should aim for a comprehensive characterization of ***all known and potential*** PFAS sources and analytes. The lack of documentation of PFAS use is not sufficient to rule out a site for further investigation because PFAS chemicals are generally not listed on Safety Data Sheets or other product inserts. See the Interstate Technology & Regulatory Council (ITRC) PFAS sampling and analytical methods fact sheet and supplemental information on PFAS assessment from USEPA in Appendix C.

For REC sites, a detailed, certified progress update discussing site activities and progress toward meeting the deadlines set out in 15A NCAC 13C.0302(h) must be submitted at least annually.

2.1 Remedial Investigation Work Plans

The initial RI work plan should present all available information on disposal history, regulatory history and site characteristics, and outline methods for gathering data to identify contaminant source areas and characterize the waste and extent of contaminants in soils, ground water, surface water and other contaminated media. The plan should also provide for collecting data to understand the geology and hydrogeology of the area. These data are then used to develop a site conceptual model that allows design of an effective remedy. A work plan should be prepared for each work phase. The number of phases needed depends on the size and complexity of the contaminated site. All documents must be properly certified according to the requirements in the Administrative Agreement (for work conducted under state oversight) or according to the REC rules (for work conducted under REC oversight). See Appendix A for the document certification requirements.

If part or all of the remedial investigation was completed prior to the execution of an Administrative Agreement for voluntary cleanup, a summary of the work and the results of the remedial investigation should be prepared. Work does not have to be repeated unless the Branch or the consultant finds QA/QC issues or data gaps in the previous work. The summary should list all pre-existing RI work plans and reports for the site and ensure that they have been provided to the Branch. **Phases of work previously approved by the Branch do not need to be repeated, just certified as complete.**

2.1.1 Identification of Contaminants and Areas of Concern (First Phase) Work Plan

In this initial phase of work, all areas known or suspected of being contaminated should be investigated. Known or suspected contaminated areas are those where there is some indication of a release based on the following:

1. Existing laboratory data;

2. Observable conditions indicative of contamination, such as staining, odors, or evidence of damage to, or leakage from, a storage facility or vessel;
3. Records of on-site spills or disposal; and
4. Other evidence actually known to the environmental consultant or the remediating party.

The following information should be documented in a RI work plan to guide sample collection and analyses according to the site's setting and environmental history.

1. Site location information including street address, longitude and latitude, and site and surrounding property land use;
2. A summary of the nature of all identified on-site hazardous substance releases, including disposal or spills;
3. Through a discussion with employees and review of records, a summary of all management practices employed at the site for hazardous wastes and wastes that may have contained hazardous substances including: a list of types and amounts of waste generated, treatment and storage methods, and ultimate disposition of wastes; a description of the facility's past and current RCRA status; and the location and condition of all identified vessels currently or previously used to store any chemical products, hazardous substances or wastes;
4. United States Geological Survey topographic maps sufficient to display topography within a one-mile radius of the site;
5. An accurate and detailed site map including: scale, north arrow, locations of property boundaries, buildings, structures, perennial and non-perennial surface water features, drainage ditches, dense vegetation, known and suspected spill or disposal areas, sumps chemical or waste, storage vessels, existing on-site wells, septic systems, and storm water conduit and ponds;
6. A description of local geologic and hydrogeologic conditions;
7. A chronological listing of all previous owners and each period of ownership since the property was originally developed from pristine land;
8. Operational history with aerial photographs and Sanborn Fire Insurance maps to support land-use history as needed;
9. A list of all hazardous substances which have been used or stored at the site, and approximate amounts and dates of use or storage as revealed by available written documentation and interviews with a representative number of former and current employees or occupants possessing relevant information;
10. Site environmental permit history, including copies of all federal, state, and local environmental permits, past and present, issued to the remediating party or within its custody or control;
11. A summary of all previous and ongoing environmental investigations and environmental regulatory involvement with the site, and copies of all associated reports and laboratory data in public records, or within the custody or control of the remediating party so the work does not have to be repeated;

12. Proposed procedures to evaluate the risk of contaminant migration into structures via the vapor pathway and to wells, springs, surface water supply intakes and to sensitive environments;
13. Intended procedures for characterizing site geologic and hydrogeologic conditions and identifying each contamination source as to each affected environmental medium, including any plan for special assessment such as a geophysical survey;
14. Intended field methods, locations, depths of, and justification for, all sample collection points for all media sampled, including monitoring well locations and anticipated screened intervals;
15. Proposed field and laboratory procedures for quality assurance/quality control. PFAS sampling should be done carefully to prevent sample contamination and should include the collection of blanks. The ITRC online document for PFAS sample collection is provided in Appendix C;
16. Proposed analytical parameters and analytical methods for all samples (see Appendix B);
17. Equipment and personnel decontamination procedures;
18. A description of measures that assure the health and safety of nearby residential and business communities by demonstrating that they will not be adversely affected by activities related to the remedial investigation;
19. Signatures and seals from the appropriate professionals, if necessary (e.g., licensed geologist, registered professional engineer, etc.). A single document may require the signature and seal of more than one professional;
20. Additional appropriate document certification statements or forms (See Appendix A); and
21. A schedule for completing site activities and submission of reports.

The REC Program rules do not authorize an RSM to practice outside his/her area of professional expertise in any phase of work. If an RSM has relied on the advice of other professionals with relevant expertise, the document must be signed and sealed by the appropriate professionals.

2.1.2 Delineation of Extent of Contamination (Subsequent RI Work Phase)

Even if the site property is, and/or will be, designated as industrial-use only, the extent of contamination must be delineated to the unrestricted-use goals to identify where land use controls must be placed.

Upon completion of the initial phase of the remedial investigation, the contaminants of concern for the site should be known. Subsequent phases of, and work plans for, the remedial investigation are generally designed to (i) delineate the lateral and vertical extent of contamination in each area of concern for all contaminated media (soil, groundwater, sediment, surface water, and vapor), (ii) identify potential exposure pathways and receptors that may currently be, or may become, exposed to the contamination, (iii) collect sufficient sampling data to support a cleanup-level determination, and (iv) characterize site conditions sufficiently to conduct a feasibility study of remedial alternatives and to support a proposed remedy. Appropriate document certification statements or forms (see Appendix A) should be included in all RI work plans.

The sampling and analysis procedures for delineating the extent of contamination should follow the specifications in Appendix B. The unrestricted-use remediation goals referenced in this document (See Chapter 3.0) *must* be used as delineation endpoints for soil, groundwater, and surface water during the remedial investigation. However, at some sites, local natural background concentrations (metals only) and anthropogenic background concentrations (PAHs, PCB and/or Dioxins) or sample reporting limits (using the analytical methods specified in Section B.6.12) may serve as delineation endpoints, provided that the laboratory's reporting limits are not elevated more than 10 times the laboratory's MDLs, and/or published average MDLs for the particular method/analyte. The DEQ Risk Calculator may also be used to calculate cumulative risk for residential/unrestricted use at each sampling point to confirm delineation of soil contamination.

Information contained in prior reports submitted to DEQ may be summarized or referenced. Any data or reports not already provided to DEQ should be attached. The following information should be included in subsequent RI work phase work plans:

1. Items 14-21 from Section 2.1.1.
2. An inventory and map of all identifiable wells, springs, and surface-water intakes used as sources of potable water (this excludes all wells used solely for the purposes of monitoring groundwater quality) within 1,500 feet of the contaminant perimeter as defined by 15A NCAC 02L standards or interim maximum allowable levels (IMACs) set by DEQ for ground water, or if the extent of contamination is not defined, within 1,500 feet of the property boundary. Contaminants such as 1,4-Dioxane and several short-chained PFAS are very water soluble and tend to travel at the speed of groundwater. Concentrations of these chemicals, time since the release, groundwater flow velocity, local topography, and likelihood of aerial deposition (e.g., previous presence of a smokestack or fume hood at facility) should be considered when determining the distance from the property that may impact receptors.

   As the remedial investigation and remedial action proceeds, conditions may change, so the potable water survey for a site should be updated approximately every five years or more frequently, depending on changes in plume extent and/or land use, to determine whether any threats to water supply wells or sensitive receptors exist.

   Note: This information is required unless documentation is available to demonstrate that groundwater is not and will not become contaminated from site sources or contaminant migration has stabilized.
3. A structural vapor intrusion evaluation if volatile organic contamination is located within 100 feet of an occupied or potentially occupied building. Refer to the DWM Vapor Intrusion Guidance document for procedures. The DEQ Risk Calculator should be used to evaluate structural vapor intrusion risk as groundwater, soil gas, and indoor air data are gathered.
4. An evaluation of the source property and adjacent properties for the existence of environmentally sensitive areas. A visual evaluation of the source property and all adjacent properties within 1,500 feet from the extent of contamination should be conducted to identify and document the existence of any of the areas listed below. In addition to a visual site reconnaissance, the state and federal agencies noted below should

be contacted to identify sensitive, protected, or recreational areas within 1,500 feet from the extent of contamination. Associated contact information is listed in Appendix D.

- Wetlands
- Natural areas that could be attractive to terrestrial ecological receptors
- Areas of stressed vegetation or stressed wildlife
- Sensitive areas identified under the National Estuary Program
- Rare species (state and federal threatened and endangered)
- Sensitive aquatic habitats
- Federal land designated for the protection of natural ecosystems
- State-designated areas for protection or maintenance of aquatic life
- Terrestrial areas utilized for breeding by large or dense aggregations of animals
- Spawning areas critical for the maintenance of fish/shellfish species within river, lake or coastal tidal waters
- Migratory pathways and feeding areas critical for maintenance of anadromous fish species within river reaches or areas in lakes or coastal tidal waters in which such fish spend extended periods of time
- Areas important to maintenance of unique natural communities
- National and State historical sites
- Areas identified under coastal protection legislation
- Coastal barriers or units of a coastal barrier resources system
- Designated State natural areas
- Federal or State designated wild and scenic rivers
- Designated and proposed federal wilderness and natural areas
- National and State preserves and forests
- National or State wildlife refuges
- Marine sanctuaries
- State lands designated for wildlife or game management
- National and State seashore, lakeshore and river recreational areas
- National and State parks or monuments

Knowledge of the presence of any sensitive environments is necessary to determine if any special sampling (such as aquatic toxicity testing) is necessary for an ecological risk assessment and to plan remedial actions to avoid damage to these areas where possible. Whether active site remediation may do more harm to an ecosystem than leaving residual contamination in place should be considered. The Branch should be contacted for further instructions regarding conducting ecological screening evaluations and risk assessments or consult existing DWM guidance here: 2003 Eco Risk Guidelines. Procedures for conducting additional assessment can be described in a work plan.

Distance to known or potential threatened receptors should be reviewed as the remedial investigation proceeds. The primary receptors of concern include potable water supplies, structures within 100 feet of volatile contamination, surface waters, and sensitive ecological environments.

2.2 Remedial Investigation Reports

Following the remedial investigation, reports should be submitted to the Branch. Reports prepared by RECs should be clearly written and well organized for the public to understand. The investigation reports should document the findings of the site investigation in sufficient detail to delineate the contamination in all media to unrestricted use levels or regional background levels, identify potential sensitive receptors, support the final cleanup-level determination, and assist with conducting a feasibility study of remedial alternatives. Any special studies that may be conducted including an ecological evaluation, a vapor intrusion study, or a geophysical survey for buried material would be part of a remedial investigation and the findings should be included in the remedial investigation report. If a single investigative phase can complete contaminant delineation for a site, the report should state that it serves as a final RI report.

If the remedial investigation is complete and no remedial goals have been exceeded for any of the media (i.e., soil, groundwater, sediment, surface water, vapor, etc.) or cumulative risk for soil and vapor is acceptable for residential use such that remedial action is not necessary, a combined Remedial Investigation and Remedial Action Completion Report should be prepared. Such reports prepared under the REC Program or state-lead oversight must be certified according to the document certification requirements in Appendix A.

RI reports should be organized in sections and at minimum, they should include the following information:

1. A narrative description of how the investigation was conducted, including a discussion of any variances from the approved work plan(s);
2. A description of groundwater monitoring well design and installation procedures, including drilling methods used, completed drilling logs, "as built" drawings of all monitoring wells, well construction techniques and materials, geologic logs, and copies of all well installation permits;
3. A map, drawn to scale, showing all soil sample and monitoring well locations in relation to known disposal areas or other sources of contamination. Monitoring wells should be surveyed to a known benchmark and groundwater elevations to a known datum. Soil sample locations should be surveyed to a known benchmark or flagged with a secure marker until after the remedial action is completed;

   Note: As provided in G.S. 89C-2, it is unlawful for any person to practice land surveying in North Carolina, as defined in G.S. 89C, unless such person has been duly registered as a registered land surveyor.

4. A description of all field and laboratory quality control and quality assurance procedures followed during the remedial investigation (see Appendix B, Section B.9, and the protocol for PFAS sampling in Appendix C);
5. A description of procedures used to manage drill cuttings, purge water and decontamination water;
6. A summary of site geologic conditions, including a description of soils and vadose zone characteristics;
7. A description of site hydrogeologic conditions (if groundwater is, or may become, contaminated), including current uses of groundwater, notable aquifer characteristics, a water table elevation contour map with groundwater flow patterns depicted, and tabulated groundwater elevation data;
8. Tabulated analytical results for all sampling (including sampling dates and soil sampling depths) and copies of all laboratory reports including quality assurance/quality control documentation;

   Note: Where a GC/MS library search is conducted, a summary of the nature of any tentatively identified compounds (TICs) eliminated from further analyses and reporting should be provided in the report, including reasons for discounting the constituent as a site contaminant. Refer to Section B.6.1.1.2 in Appendix B for further information on evaluating TICs**.**
9. Where contaminants exceed cleanup levels for groundwater or surface water, or risk for residential use is exceeded for soil or sediment using the DEQ Risk Calculator, contaminant delineation maps for each primary contaminant of concern, including scale and sampling points with contaminant concentrations;
10. If contaminant concentrations exceed unrestricted use remediation standards in groundwater or risk for residential use in soil at depths greater than five feet below ground surface, cross sections including scale and sampling points with contaminant concentrations;
11. A description of procedures and the results of any special assessments such as geophysical surveys, soil gas surveys, test pit excavations, or if volatile contaminants are present at the site, structural vapor intrusion evaluations (please refer to the DWM Vapor Intrusion Guidance);
12. Results of the water supply and sensitive environment receptor survey outlined in Section 2.1.1 of this guidance, if not provided in a previous document;
13. Copies of all field logs and notes, and color copies of site photographs, if collected, that provide documentation of the investigation;
14. The signature and seal of licensed professionals (e.g., licensed geologist, registered professional engineer, etc., if such work is included). A single document may require the signature and seal of more than one professional; and
15. Appropriate certification statements or forms (see Appendix A).

3.0 Remediation Goals and Standards

Remediation goals and standards are established in a manner consistent with CERCLA/SARA and the National Contingency Plan, as required by N.C.G.S. 130A-310.3. With Branch approval, site-specific, risk-based groundwater remediation goals may be pursued for eligible sites under N.C.G.S. 130A 310.65 through 310.77.

3.1 Soil Remediation Goals

Soil contamination must be remediated to (1) levels that are protective of human health through direct contact and (2) levels that do not leach to groundwater and cause exceedance of the North Carolina groundwater quality standards under 15A NCAC 02L, IMACs, or a site-specific groundwater standard for a risk-based cleanup per N.C.G.S. 130A 310.65 through 310.77.

Preliminary Soil Remediation Goals (PSRGs) are provided as part of the Inactive Hazardous Site Guidance Documents on the Branch's website and on the DEQ Risk-Based Remediation website under Risk Evaluation Resources. PSRGs are updated twice per year when USEPA's updated Regional Screening Levels are released. The current PSRG Table should be consulted at the onset of remedial action since toxicity values may change or new chemicals may be added, particularly in the case of emerging compounds such as PFAS. Final soil remediation goals should be based on acceptable risk for the intended property use (restricted or unrestricted) and can be determined using the DEQ Risk Calculator available for download from the Risk Evaluation Resources page of the Risk-Based Remediation website.

3.1.1 Human-Health-Based Direct Contact Soil Remediation Goals

In addition to direct exposure to contaminated soils through dermal contact, ingestion and inhalation, the following conditions must be considered when determining the final cleanup levels for soils:

1. Could the property become agricultural (crops, livestock, etc.), with possible uptake of contaminants by plants and livestock?
2. Could surface waters, wetlands, or other sensitive ecological receptors be affected by contaminated soils?
3. Can soil contamination cause a structural vapor intrusion risk to future structures?

3.1.1.1 Unrestricted-Use Goals

The "human health-based" PSRGs established for unrestricted (residential) land use considers both adult and child (1 to 6 years of age) exposure to contaminated soil. These residential or unrestricted use PSRGs or calculated values based on cumulative risk are to be used when no limit on site use is desired.

3.1.1.2 Restricted Use Goals

Preliminary commercial/industrial contaminant remediation goals are available in the PSRG table and can be used as final remedial goals. Less conservative cleanup levels can also be based on cumulative risk for a non-residential property use and can be determined using the DEQ Risk Calculator. Any remedy that does not meet residential/unrestricted use will require the recordation of Branch-approved land-use restrictions as part of a RAP. Engineered controls, such as permanent barriers, supported by land use restrictions may allow higher concentrations to remain in place if contaminants do not leach to groundwater. Note that the extent of contamination must be delineated using unrestricted use goals even if restricted use remediation goals are planned.

3.1.1.3 Calculating Cumulative Risk

If a human health PSRG is exceeded, all detected contaminants in each sample or representative sitewide maximum concentrations should be entered into the DEQ Risk Calculator to calculate cumulative risk. Soil remediation goals can also be developed using the DEQ Risk Calculator. The Branch prefers that site decisions be based on risk rather than screening levels.

3.1.1.4 Averaging Soil Contaminant Concentrations

Average contaminant concentrations in soil may be used to compare with the health based PSRGs for both unrestricted and restricted land use. Representative concentrations used in the DEQ Risk Calculator should be a 95% upper confidence level calculation and will require review by the Branch. Averaging cannot be used to demonstrate that soil concentrations are protective of groundwater. Averaging should only be conducted in areas of consistent use and generally uniform release of contaminants (e.g., former waste lagoons, spray fields, orchards, etc.) All of the following conditions apply to the use of such averaging:

- Only sample points within 1/4-acre sectors may be averaged for comparison to unrestricted-use levels. For restricted industrial use (land use restrictions approved as part of the RAP), averaging over larger areas can be performed if the access and use across the area is consistent. Remote areas and areas of less frequent access may not be included in the industrial restricted-use averaging.
- No single sample point may exceed ten times the site-specific adjusted cleanup level for all contaminants except lead. For lead, no single sample point used in an average may exceed 1000 ppm for unrestricted-use and no more than three times the site-specific cleanup level for restricted-use.
- The quarter-acre zone may be a circle or a square or triangle of generally equal sides. One dimension of the zone’s perimeter may not be disproportionately longer than another. An exception would be a greenway corridor.
- Samples must generally be evenly spaced over the zone of averaging.
- Only samples of the same vertical horizon may be averaged (0-6 inches for surface samples and no more than 5-foot vertical spread for subsurface samples.
- Only actual sample data may be used for all points included in the average and not published averages for background concentrations.
- The laboratory practical quantitation limit must be used for points where concentrations are at or below laboratory reporting limits. Sample data should not have elevated reporting limits (see Background Adjustments on the previous page).
- Composite sample results may be included in an average but must be weighted proportionally to the area they represent. For example, if one composite sample in an area represents ½ of the area and 5 others represent 1/10 of the area each, then the

concentration of the first composite should be multiplied by 5, added to the sum of the other concentrations and then divided by 10 to compute the average concentration.

- For characterizing soil concentrations over a 1/4-acre area, a sampling grid with 50-foot grid node spacing should be established. The average concentration for each compound within a 1/4-acre area is presumed to represent the entire 1/4-acre area. If the average concentration for any compound exceeds unrestricted-use remedial goals, the 1/4-acre area would require cleanup or land use restrictions. For very large areas (e.g., a 500-acre orchard), an alternative is to collect samples in multiple 1/4-acre sectors within the overall contaminated area that represent the range of environmental conditions present (i.e., various geologic and geographic conditions such as slope vs. valley, wetter vs. drier, etc.). Grids with a 50-foot node spacing should be established across these representative areas. The highest 1/4-acre average from all the areas tested would be presumed to reflect the overall area. This approach requires the area to be consistent in use and accessibility and requires land use restrictions as part of the remedy.
- For unique circumstances, contact the Inactive Hazardous Sites Branch for further guidance.

3.1.2 Protection of Groundwater Soil Remediation Goals

In addition to meeting health-based remediation goals, soil must be remediated to levels that protect groundwater. Soils that leach contaminants in excess of the groundwater remediation goals will require further remediation. Groundwater remediation goals are either the lower of the15A NCAC 02L standards or federal maximum contaminant levels (MCLs), IMACs, or natural background concentrations for unrestricted groundwater use, or site-specific risk-based cleanup levels (for restricted groundwater use). Multiple methods are available to evaluate the leachability of contaminated soil.

1. The Branch provides Protection of Groundwater screening levels for individual contaminants in the PSRG table. These protection of groundwater soil screening levels may be used as final remedial goals, but the values are calculated using conservative assumptions and default values that do not fit most site conditions, so one of the other methods may be more useful.
2. Site-specific aquifer data (porosity, bulk density, and organic carbon content) can be used in place of the default values in the equation at the end of the PSRG table or in the DEQ Risk Calculator. Only the parameters noted should be modified and only site-specific data should be used. All calculations and data must be provided in the RI report.
3. If groundwater data from the area of concern show that the contaminants do not exceed (1) the target groundwater concentration or are below quantitation limits for those contaminants without numeric standards and (2) the release occurred fifteen or more years ago, the protection of groundwater is considered to have been met. Note that quantitation limits may not exceed the standards or otherwise must be the lowest achievable limits.
4. Collect and analyze soil samples in the areas of highest contaminant concentration using the Synthetic Precipitation Leaching Procedure (SPLP) or Toxicity Characterization

Leaching Procedure (TCLP). TCLP is a procedure that uses organic acids to simulate typical landfill conditions. For this reason, SPLP may be a more appropriate procedure because it is more representative of leaching under natural rainfall conditions. If contaminant concentrations in the soil leachate exceed their respective groundwater remediation goals, those soils require remediation.

5. Determine a soil protection of groundwater remediation goal by plotting sample pairs of total and leachable (using SPLP or TCLP) concentrations and finding the total concentration that corresponds to a leachable concentration at the 15A NCAC 02L standard or IMAC. Several soil samples need to be collected from various locations within the area of concern representing the higher and lower concentrations.
6. If the soil contaminant concentrations (in mg/kg) for both metals and organics (totals analysis) do not exceed values of twenty times the corresponding groundwater remediation goals (in ug/L), then the leachability criterion is considered met. The 20 times multiplier represents the dilution of the soil sample during the SPLP and TCLP extraction.

3.2 Sediment Remediation Goals

For intermittent streams, the procedures that are used to establish remedial goals for soil should be used as described in Section 3.1. Remediation goals for sediment in perennial streams and other surface waters must meet all the following:

1. The health-based soil remediation goals listed in the PSRG table (or the upstream "background" concentrations if higher);
2. Levels that ensure contaminated sediment will not cause exceedance of the remediation goals for groundwater and surface water; and
3. Levels that ensure protection of aquatic receptors. Maximum sediment contaminant concentrations must be compared to USEPA Region 4 Eco Risk Screening Levels for sediment.

To demonstrate compliance with the Branch's preliminary sediment remediation goals for the protection of aquatic receptors, the laboratory must achieve sample quantitation limits less than or equal to the USEPA Ecological Screening Level. If this is not possible, it needs to be stated in the case narrative that the quantitation limits are the lowest that can be achieved using USEPA-approved methods.

If site ecological screening levels for sediments are exceeded, the remediating party needs to contact the Branch with a request for the Branch to determine the need for further ecological evaluation. The request should provide the following information:

1. A statement that the contaminated surface water body is perennial or intermittent;
2. A topographic map with roads and surface water features clearly identified;
3. A map drawn to scale with locations of all sampling points;
4. A summary table containing maximum contaminant concentrations, upstream contaminant concentrations, USEPA aquatic screening levels and sample quantitation

limits. All contaminant concentrations that exceed screening levels should be clearly identified and highlighted. Also, concentrations that have no screening level should be clearly identified and highlighted;

5. A discussion of potential mobility of contaminated sediment and potential for contaminants to leach into surface water;
6. The names and classifications of all downstream surface water bodies *if* they are potential recipients of contaminated surface water or sediment;
7. The identity of adjacent or downstream wetlands that could be affected; and
8. An estimate of the width and depth of the contaminated surface water body.

3.3 Surface Water Remediation Standards

Remediation goals for surface water are the NC DEQ Division of Water Resources 15A NCAC 02B standards or confirmed upstream background concentrations. However, if surface water contamination is causing sediments to exceed cleanup criteria, remediation of surface water will be necessary to eliminate this effect. Violation of the 15A NCAC 02B standards may be evaluated based on the number of surface water samples, frequency of sampling, and magnitude of contaminant concentrations detected rather than just one sampling event.

3.4 Groundwater Remediation Standards

3.4.1 Unrestricted Use

The unrestricted-use groundwater remediation levels are the permanent and interim groundwater standards established under 15A NCAC 02L. If groundwater is or may be used for potable purposes in any area where the groundwater contaminant plume is currently located or may be located in the future, the remediation goal would be the lower of the 15 NCAC 02L standard, IMAC, or federal MCL. For contaminants without 15A NCAC 02L standards or IMACs, the remediating party should contact the Branch.

> *Note:* Remediation to below the practical quantitation limits or site-specific natural background levels (for metals only) is not required.

3.4.1.1 USEPA Proposed Standards for PFAS

Recent studies have linked PFAS exposure to health effects on the liver, the kidney, the immune system, developmental effects, and cancer. Ingestion of even low levels of some PFAS can cause some of these health effects. In March 2023, USEPA proposed regulatory standards for the following six PFAS in public drinking water supplies. The final regulation is anticipated to be issued by the end of 2023. Units are in parts per trillion (ppt) or nanograms per liter (ng/L) when discussing PFAS in groundwater.

PFOA	4.0 ppt, proposed,
PFOS	4.0 ppt, proposed, and

A mixture of HFPO-DA (GenX Chemicals), PFBS, PFNA, PFHxS at a Hazard Index (HI) of 1.0. Note that a concentration above of any of the limits provided below will automatically exceed a HI of 1.

GenX – 10 ppt

PFBS – 2000 ppt

PFNA – 10 ppt

PFHxS – 9 ppt

Hazard Index = [GenX]/10 + [PFBS]/2000 + [PFNA]/10 + [PFHxS]/9 (all in ppt)

The USEPA also recommended the following laboratory minimum reporting limits (MRLs) to ensure that labs are able to measure these PFAS at sufficiently low levels:

PFOA – 4 ppt

PFOS – 4 ppt

GenX (HFPO-DA) – 5 ppt

PFBS – 3 ppt

PFNA – 4 ppt

PFHxS – 3 ppt

Even though health advisory levels currently exist for only four PFAS, measuring for all analytes using USEPA Method 537.1 or USEPA Method 1633 is recommended to (1) identify the specific PFAS present, (2) acquire a more comprehensive set of data for discerning unique PFAS releases and their sources, and (3) to prepare for future health advisory levels or regulatory standards for PFAS. The USEPA is currently conducting toxicity assessments for other legacy PFAS and has reserved the option to include those PFAS in the enforceable MCL that is projected to become final in Fall 2023. USEPA also plans to fulfill additional health protective goals in a rapid timeframe through 2024 as outlined in the USEPA PFAS Strategic Roadmap that can be found on the USEPA website and in Appendix C.

3.4.2 Restricted Use

Cleanup to risk-based levels higher than the state's 15A NCAC 02L groundwater quality standards or IMACs, or EPA health advisory levels may be approved for qualifying sites with stable or predictable groundwater contamination and permission from all affected property owners. N.C.G.S. 130A-310.65 through 310.77 specifies the fees and procedures for pursuing a risk-based remedy for groundwater. Sites with extensive groundwater monitoring data that demonstrate stable or predictable plume conditions are ideal candidates. Visit the DEQ Risk-Based Remediation website for the Administrative Procedures, the DEQ Risk Calculator, and the Technical Guidance document for preparing a risk-based RAP.

Note: Due to Branch review, approval and maintenance of land-use restrictions, there are additional fees associated with groundwater risk-based cleanups, as specified in N.C.G.S. 130A-310.76.

3.5 Naturally Occurring Background Levels

At some sites, local naturally occurring background concentrations (metals only) and/or anthropogenic background concentrations (PAHs, PCB and/or Dioxins) can contribute to the site's total contaminant concentrations and risk. Background contaminants can either be naturally occurring substances that are present in the environment in forms that have not been influenced by human activity, or anthropogenic substances that are present due to human activities not specifically related to the site. Background concentrations can serve as cleanup levels for those contaminants.

Sufficient sampling should take place to statistically quantify these potential contributions to site in a legally defensible manner. The Branch does not allow comparison with published background values alone. Statistics play a major role in establishing background concentration levels, and methods vary widely in their degree of complexity, but the 95% upper confidence level calculation is preferred by the Branch The methodology used to eliminate naturally occurring compounds or determine cleanup levels for site contaminants that could also be naturally occurring should be well-documented.

3.6 Practical Quantitation Limits

According to 15A NCAC 02L .0202, where the standard for a substance either does not exist or is less than the practical quantitation limit, the practical quantitation limit becomes the unrestricted-use cleanup level (when the analytical methods specified in Section B.6.1.2 are used), provided that the laboratory's quantitation limits are not elevated more than 10 times laboratory's the Method Detection Limit (MDL) or published MDL for a contaminant. Contact the Branch if a contaminant does not have a 15A NCAC 02L standard or a PSRG.

4.0 Remedial Action

Branch concurrence is required for any remedy proposed in the REC Program that would:
1) be conducted entirely on site and for which a permit waiver is desired under G.S. 130A-310.3(e);
(2) include institutional controls for restricted use of contaminated areas or media; or
(3) exceed the cost set forth in G.S. 130A-310.9(a)

Once the remedial investigation has been completed, a Remedial Action Plan (RAP) must be prepared and submitted whenever contamination exceeds standards or acceptable risk. Prior to approval, the RAP must be made available for public comment for at least thirty days.

The public notice of the proposed RAP must be mailed after the Branch has approved the mailing list for and content of the public notice. The Branch may request that the remediating party provide additional copies of the RAP for distribution to the local health director, register of deeds office, and the public library closest to the site as part of the public notice. If the Branch determines that there is significant public interest in a site, the Branch may hold a public meeting or public hearing. Any substantive comments from the public must be evaluated, and the RAP revised as necessary before the RAP is approved. A RAP should not be implemented until permission is received in writing from the Branch, or it is approved and certified by the REC.

> *Note:* Remedial actions that involve the emission or discharge of hazardous substances to the atmosphere should be conducted in a manner that provides for the protection of human health and the environment, and complies with any applicable permits, approvals, laws or other rules or regulations.

4.1 Remedial Action Plan

If the remedial action was already in progress prior to an Administrative Agreement and continuation of the same remedy is approved by the Branch, any information previously prepared and submitted can instead be described and referenced in the RAP.

The RAP should describe the following information:

1. A discussion of the remedial investigation results including media contaminated, contaminants of concern, and the areal and vertical extent of contamination;
2. A brief statement of objectives for the remedial action;
3. An evaluation of available remedial alternatives using the following feasibility study criteria:
    a. protection of human health and the environment, including attainment of cleanup levels;
    b. compliance with applicable federal, state and local regulations;
    c. long-term effectiveness and permanence;
    d. reduction of toxicity, mobility and volume;
    e. short-term effectiveness, i.e., effectiveness at minimizing the impact of the site remedial action on the environment and the local community;
    f. implementability, i.e., technical and logistical feasibility, including an estimate of time required for completion;
    g. cost; and
    h. community acceptance.
4. A detailed description and conceptual design of the proposed remedy, for each contaminated medium including an evaluation of the potential for the remedy to affect sensitive environments identified;
5. A demonstration that the proposed remedy is supported by the remedial alternative feasibility study conducted pursuant to item 3 above;

6. A description of all activities necessary to implement the proposed method(s) of remedial action in compliance with applicable laws and regulations and in a manner, such that cleanup standards are met. These activities include, but are not limited to, well installation and abandonment, sampling, run-on/run-off control, discharge of treated waste streams, and management of investigation and remedial action derived wastes;
7. A description of any proposed treatability studies and additional site characterization needed to support the final design;
8. A description of procedures and a schedule for additional site characterization, treatability studies, final design, construction, operation and maintenance, system monitoring and performance evaluation, and progress reporting;
9. A description of the criteria for remedial action completion, including procedures for post-remedial and confirmatory sampling;

   Note: The RAP should include a work plan for monitoring and evaluating the remedy's performance and any lateral or vertical expansion of the extent of the contaminant and associated risk to receptors. The work plan should also describe post-remediation confirmation sampling. Confirmation sampling results should be submitted in Remedial Action Completion Reports. Branch guidance on confirmation sampling and analysis is provided in Appendix B;
10. A health and safety plan that assures that the health and safety of **nearby residential and business communities** will not be adversely affected by exposure to site contaminants and activities related to the remedial action. The plan should conform to all local, state, and federal regulations for health and safety;
11. Equipment and personnel decontamination procedures;
12. A schedule for completion of the remedial design, remedial action construction and implementation and periodic sampling and reporting;
13. All professional work must be signed and sealed by the appropriate professionals (e.g., licensed geologist, registered professional engineer, etc.). A single document may require the signature and seal of more than one professional; and
14. Appropriate certification statements or forms (see Appendix A).

4.2 Remedial Action Preconstruction Reports

A Preconstruction Report should be prepared and submitted in accordance with the terms of the Administrative Agreement prior to the beginning of construction activities for all remedies involving construction or equipment installation. This includes but is not limited to remedies such as injection treatments, capping or excavation. These reports should include the following elements:

1. The results of all treatability studies and additional site characterization work completed since the remedial investigation;
2. Final engineering design report, including a narrative description of process design, final plans and specifications, and an updated project schedule;
3. Copies of final registrations, permits and approvals; and
4. Appropriate certification statements or forms (see Appendix A).

To streamline the REC Program reporting requirements, contents of the Remedial Action Preconstruction Reports are now to be included in the RAP or the first remedial action progress report.

4.3 Remedial Action Construction Completion Reports

When a remedy involving activities such as excavation, construction of borings, injection treatments or other active actions, a remedial action Construction Completion Report should be prepared and submitted. This report should include the following:

1. “As-built" plans and specifications;
2. A summary of major variances from the final design plans;
3. A summary of any problems encountered during construction; and
4. Appropriate certification statements or forms (see Appendix A).

To streamline the REC Program reporting requirements, contents of the Remedial Action Construction Completion Reports should be included in the first remedial action progress report.

4.4 Remedial Action Progress Reports

Remedial Action Progress Reports should be prepared and submitted in accordance with the terms of the Administrative Agreement. In general, quarterly Remedial Action Progress Reports are necessary for remedial actions greater than three months in duration. Groundwater Remedial Action Progress Reports may be prepared on an annual basis after the first full year of remedial action and the completion of four quarterly monitoring events. Remedial Action Progress Reports should contain at least the following:

1. A summary of operation and maintenance activities, observations and a discussion of major problems encountered;
2. Performance evaluation results, i.e., tabulated and graphical presentations of monitoring data and a comparison of remedial action performance to design goals;
3. A description of all field and laboratory quality control and quality assurance procedures followed during any sampling and analysis;
4. Copies of all laboratory reports including quality assurance/quality control documentation;
5. A map, drawn to scale, showing all monitoring locations; and

6. Appropriate certification statements or forms (see Appendix A).

4.5 Remedial Action Completion Report (Final Progress Report)

The Remedial Action Completion Report (final progress report) should be submitted in accordance with the terms of the Administrative Agreement. This report should include the information required under Section 4.4, above, and the following:

1. A summary of remedial action operating experience and effectiveness in meeting design goals, based on all performance monitoring data and progress reporting to date;
2. A discussion of criteria for remedial action completion, and a demonstration, supported by confirmatory sampling data, that such criteria have been satisfied;
3. Proof that all monitoring wells have been properly abandoned, if abandonment is approved in writing by Branch staff.
4. Request for a No Further Action letter if site closure is desired.
5. Appropriate certification statements or forms (see Appendix A).

5.0 No Further Action Determinations

After satisfactorily completing a voluntary remedial action, the work required under the Administrative Agreement is considered to be complete, and the Administrative Agreement is terminated. The site will then be assigned "No Further Action" status in the Inactive Hazardous Sites inventory. This change of inventory status does not preclude any future state action if new evidence of contamination is discovered at a later date. In accordance with N.C.G.S. 130A-310.7(c), any party wishing to receive a written "No Further Action" determination from the Branch must provide the request in writing.

Remediating parties that have completed an independent cleanup that meets unrestricted use remedial goals may also request a no-further-action review of the work. A fee is required for Branch review of independent cleanups. The fee schedule is generally based on the duration and volume of contamination and the remedial action completed and can be found on the Branch's website here: https://www.deq.nc.gov/about/divisions/waste-management/superfund-section/inactive-hazardous-sites-program/ihs-no-further-actions.

Owners of properties with land-use restrictions will be required to submit to the Branch annual certifications affirming that the institutional controls remain recorded at the Register of Deeds office and activities at the site are in compliance with the land use restrictions. Current and future property owners, operators and other responsible parties are required under N.C.G.S. 130A-310.3(f) to enforce the LURs and are expected to take action immediately upon discovery of a violation of the LURs. Failure to do so could cause automatic revocation of Branch concurrence of the remedial action or a no-further-action status. Certification forms can be downloaded from the Branch website.

Appendix A: Document Certification Requirements

Whether remedial action is conducted with direct Branch oversight or the Branch's Registered Environmental consultant program, certifications by the person overseeing the work are required to ensure the integrity of the work.

The specific certification requirements for both State-lead and REC work conducted in the Branch are outlined in Tables A-1 and A-2 below. All certifications must contain the notarized signature of the appropriate representative responsible for the remedial activities.

REC Certification Forms

REC work plans and reports must include notarized document certification forms from the remediating party and the RSM.

The specific certification forms cannot be reproduced in any way.

Separate REC work phase completion (WPC) forms must be used to certify the completion of work phases in accordance with .0306(b)(5) and (b)(6).

The signatures must be properly notarized using only the notary text shown on the forms.

The remediating party must sign and have notarized their document content certification **prior** to the RSM's certification.

Table A-1. State-Lead and REC Document Certification Statements and Forms.

Documents and Other Submissions	REC Document Certification Form	REC Work-Phase Completion Form*	State-Lead Certification Statements
Annual Progress Update Reports	DC-I and DC-II		The following statement individually signed and notarized by the RP and the consultant: “I certify that, to the best of my knowledge, after thorough investigation, the information contained in or accompanying this certification is true, accurate, and complete.” If the document includes the relevant professional work, include: Any work that would constitute the “practice of engineering” as defined by G.S. 89C shall be performed under the responsible charge of, and signed and sealed by, a professional engineer registered in the state of North Carolina. Any work that would constitute the “public practice of geology” as defined by G.S. 89E shall be performed under the responsible charge of, and signed and sealed by, a geologist licensed in North Carolina.
Remedial Investigation Work Plan	DC-I and DC-II		
Remedial Investigation Report	DC-I and DC-II	WPC-II	
Remedial Action Plan	DC-I and DC-II	WPC-III	
Groundwater Remedial Action Initiation	DC-I and DC-II	WPC-V	
Progress Monitoring Reports	DC-I and DC-II		
Remedial Action Completion Report	DC-I and DC-II	WPC-VI, WPC-VII, or WPC-VIII	
Project Schedules, Data Summaries, Interpretations, Calculations	DC-I and DC-II		Not applicable

* WPC-I and WPC-IV are no longer used according to the re-adopted REC Rules effective July 1, 2020.

Table A-2. REC Program Work-Phase Completion (WPC) Form*

WPC Form	Title
II	Remedial Investigation Completion Certification
III	Proposed Remedial Action Plan Completion Certification
V	Groundwater Remedial Action Initiation
VI	Remedial Action Completion Certification
VII	Remedial Action Completion Certification “for Remedy with Land Use Restrictions”
VIII	Combined Remedial Investigation & Remedial Action Completion Certification “for No Action Remedy”

* WPC-I and WPC-IV are no longer used according to the re-adopted REC Rules effective July 1, 2020.

Appendix B: Sampling and Analyses

Environmental sample collection and analyses should only be performed by persons who are qualified by education, training, and experience. Procedures and methodologies employed for the collection and analysis of soil, sediment, water, vapor, air, and waste samples should follow the methods published by the United States Environmental Protection Agency (USEPA), the American Society for Testing and Materials (ASTM), the American Public Health Association (APHA), the National Institute for Occupational Safety and Health (NIOSH), the American Water Works Association (AWWA), or other organizations with expertise in the development of standardized analytical testing methods.

> *Note:* Due to the extensive use of PFAS and their presence at trace levels in most environmental media, all aspects of sampling and analysis require a unique protocol and high level of care to avoid cross-contamination with everyday packaging, body lotions, and biasing sampling equipment. Please follow the Interstate Technology & Regulatory Council Sampling and Analytical Methods in Appendix C.

B.1 Soil Sample Collection

B.1.1 Phase I Sampling to Identify Contaminants

The purpose of the Phase I soil investigation is to identify all releases of hazardous substances to site soils, to characterize the chemical nature of such releases, and to collect sufficient sampling data to establish remediation goals.

Known or suspected spills and disposal areas must be identified by researching waste management records, vintage maps, aerial photographs; and other information and by conducting employee interviews. All areas known, suspected or having been contaminated by hazardous substances must be investigated. Areas known or suspected of being contaminated are those areas where there is some evidence (such as, but not limited to, allegations or indications of spills, visual observations, field instrument readings, laboratory data, and chemical odor) of a release of hazardous substances or of materials that contain or may contain hazardous substances. The necessary sampling strategy depends on whether or not there is visible evidence of contamination.

All soil sampling and boring locations should be documented using properly calibrated GPS equipment (in decimal degrees to 5 decimal places). Note that some data (e.g., monitoring well elevations and locations) will require survey-grade precision.

B.1.1.1 Visible Evidence of Contamination

At least one grab soil sample should be collected centrally from the most visibly contaminated location and horizon in each area of hazardous substance release or possible release.

B.1.1.2 No visible Evidence of Contamination

Surface Release

1. If no visible evidence exists in an area of a suspected *surface release* of contaminants, sampling should be conducted by first establishing a grid with grid line intersections (nodes) spaced no farther than 50 feet apart. Samples collected for all analyses except volatile organics (VOCs) should be collected from ground surface (0 to 6 inches) at each grid node. Samples collected for volatile organic analysis should be collected at a depth of 6 to 12 inches below ground surface. Compositing to reduce the total number of samples for non-VOC analyses may be conducted for qualitative purposes as follows:

   ≤ 62,500 square feet: No more than four adjacent grid node (250 ft. x 250 ft.) samples may be composited.

   > 62,500 square feet: A greater number of adjacent grid node samples may be composited, but a minimum of five resulting composite samples should be submitted for laboratory analysis.

   Samples collected for volatile organic analysis should be unmixed grab samples.

   Samples for VOC analyses should be collected at each node as unmixed grab samples without compositing. If the area exceeds 15,000 square feet, a minimum of five samples should be collected from locations that are evenly distributed across the area of suspected contamination. Field screening methods may be used to select the locations of these unmixed samples. For areas greater than 62,500 square feet, at least five additional samples should be collected by compositing grab samples from at least 25% of the nodes which generally reflect an even distribution across the area. These composited samples will be used for qualitative purposes only.

   Note: For extremely large sites (sites several acres in size), contact the Branch to discuss site specific conditions.

2. In addition to 1 above, if the actual contaminants released are unknown, mobile contaminants or contaminants that have been detected in groundwater at the site, a soil boring should be advanced to the water table. The boring should be centrally located in the area of concern and adequately sampled at intervals from ground surface to the water table. Examples of sampling intervals include 0 to 6 inches below ground surface, every five feet thereafter to just above the water table. Note that saturated samples may be indicative of groundwater contamination and not soil contamination. Additional sampling depths should also be chosen based on visual and field-screening evidence.

Subsurface Release

1. The results of the historical research should be used to plan geophysical surveys and test trenching. Geophysical surveys should be conducted by scanning areas of concern on parallel and perpendicular traverses spaced no further than 30 feet apart. Closer spacing may be necessary when using a metal detector. Grids should be established in all areas that yield anomalous readings during the scanning phase. Grid nodes should be spaced no greater than 10 feet apart. Readings should be recorded at each grid node and mapped. If areas are excluded from the survey due to instrument interference, the remediating party should

provide a written justification for exclusion along with a map delineating the features causing the interference with the Remedial Investigation Report.

2. Once the subsurface disposal area has been identified, it should be sampled according to whether there is visible evidence of contamination or no visible evidence as described above. If the suspected subsurface disposal area cannot be located using the methods described above, a soil boring should be advanced through the suspected disposal area in accordance with the procedures for surface releases above.

B.1.1.3 Waste Material Sampling

Waste materials (e.g., fly ash, sludge, concrete, wood, etc.) that are known or suspected to contain hazardous substances that may cause an exposure hazard and contaminate other media should be evaluated using the same procedures as if it were contaminated soil. Laboratory analyses are necessary to determine if the contaminants in the waste materials exceed the Branch's remedial goals.

B.1.2 Subsequent Sampling to Delineate Extent of Soil Contamination

Delineating the extent of soil contamination requires sampling all ditches, culverts or other drainage features that may have received runoff from known-contaminated areas. Field screening methods, such as soil gas testing and immunoassay test kits, may be used to help define the extent of contamination. If these methods are used, soil samples should also be collected at the expected vertical and lateral boundaries of each contaminated area and sent to the laboratory for confirmation.

B.2 Groundwater Sample Collection

B.2.1 Phase I Sampling to Identify Contaminants

In many cases, insufficient information on the nature of hazardous substance releases at the site will make it necessary to perform the Phase I groundwater field work after the Phase I soil results are received and evaluated. If the water table is within five feet of the ground surface, the contaminants are known to extend to within a five-foot depth of the water table, or a soil leaching test (like the synthetic precipitation leaching procedure) suggests that mobile soil contaminants (such as VOCs and leachable metals) could leach to groundwater above the 15A NCAC 02L standards, groundwater in the uppermost aquifer should be sampled. At least one well should be installed centrally *within each area of an identified soil release*. Where contaminants are believed to be "floaters" due to their density and solubility in water, well screens should be positioned across the water table. Where contaminants are believed to be "sinkers," the well screen should be positioned at depth or just above the bedrock surface.

If the remediating party decides not to install a well within an area due to grossly contaminated conditions or concern for rupturing buried vessels, a minimum of three wells must then be installed immediately surrounding the suspect area. Once groundwater flow patterns are clearly defined, a well will be necessary on the hydraulically down-gradient perimeter of the area of concern. Depending on the size of the area and nature of the release, additional monitoring wells may be necessary once the source is removed or remediated.

A professional land surveyor, registered in North Carolina, must survey all monitoring well locations from a USGS known datum, and groundwater elevation data should be collected. At least one sample must be collected from each monitoring well and analyzed according to Section B.6.

B.2.2 Subsequent Sampling to Delineate Extent of Contamination

If hazardous substances are present in groundwater, additional groundwater assessment will be required to delineate their lateral and vertical extent. Sufficient data are needed to understand groundwater flow direction and pathways in the aquifer(s). The lateral and vertical extent of groundwater contaminant plumes must be defined to the 15A NCAC 02L standards or IMACs.

Groundwater elevation data should be collected during each sampling event. If subsequent water table elevation data indicate a significant change in the direction of groundwater flow, additional wells will be necessary to adequately evaluate groundwater contamination.

B.3 Surface Water and Sediment Sample Collection

B.3.1 Phase I Sampling to Identify Contaminants

Surface water assessment will be necessary if there is a potential for contaminants to migrate to surface water via surface runoff or through a discharge of contaminated groundwater. If a surface water assessment is necessary, water and sediment samples should be collected at the probable point of entry. In addition, at least one water and one sediment sample must be collected immediately upstream and downstream of the site.

For surface waters that are very shallow (less than six inches deep) or turbulent, highly turbid samples may be collected in a separate collection container and then decanted into the sample container. Samples for organic analysis must be decanted into the sample container immediately. Samples for metals analysis may be allowed to settle for a few minutes prior to decanting. All collection containers must be made of the same materials as the sample container. They must be pre-cleaned and handled in the same manner.

These samples need only be analyzed for contaminants previously detected in other media at the site unless a non-permitted direct discharge of a hazardous substance to surface water has occurred. If such a discharge has occurred, samples should be analyzed for the Phase I analyses described in Section B.6.1.

B.3.2 Subsequent Sampling to Delineate Extent of Contamination

If contamination is detected in any downstream sample above concentrations detected in upstream samples, additional surface water/sediment assessment will be needed. The purpose of the next phase(s) of surface water/sediment investigation is to define the downstream extent of contamination to concentrations less than or equal to the 15A NCAC 02B standards for surface water and the residential PSRGs for soil.

B.4 Background Sample Collection

B.4.1 Natural Soil Metals Concentrations

Site-specific background soil samples should be collected to establish natural metals concentrations if metals are a contaminant of concern at the site. Samples should be located away from roadways, railways, parking areas and other potential sources of contamination.

Because natural metals concentrations are highly variable, the Branch recommends collecting a minimum of five background soil samples. Background soil samples should be collected from depths and soil types that are representative of contaminated soils but should not be collected from topsoil (0-6 inches). Statistical methods for establishing representative background concentrations may be used. The 95% upper confidence level (UCL) calculation is preferred by the Branch. Sample concentrations that are obvious outliers should not be used to establish background concentrations.

B.4.2 Area-wide Soil Anthropogenic Background

Background soil samples should also be collected at any site having known or suspected Dioxin, PAH, and/or PCB contamination in order to establish area-wide anthropogenic background levels. These samples should be collected at various distances from the site. If the results indicate over a large area, no increase in concentration toward the site and after any obvious outliers are removed, the upper end of the range of concentrations detected (or preferably the 95% UCL) can be used as the anthropogenic level.

B.4.3 Natural Groundwater Metals Concentrations

If metals exceed groundwater remediation goals for the site, groundwater samples should be collected upgradient of any on-site sources of contamination to established natural background conditions. Use of published values alone is not allowed.

B.4.4 Normal Application of Agricultural Chemicals

Arsenic and 1,2-dichloropropane are sometimes found in groundwater due to normal application of pesticides. Other pesticides, if applied properly, are not as commonly found in groundwater due to their lower solubility. Many former agricultural properties, however, will have levels of pesticides or nitrates remaining in soils due to natural application. Federal and state laws generally exempt concentrations associated with normal application of agricultural chemicals. Overuse and improper use do not qualify for that exemption. If the presence of agricultural chemicals can be shown to be due to normal application, their remediation is not required by the IHSRA and by most state and federal remediation law.

B.4.5. Upstream Surface Water Background Concentrations

If surface water assessment is necessary, background (upstream) surface water and sediment samples should be collected to establish natural or anthropogenic background conditions. Samples should be collected upstream of any on-site sources of contamination. If contamination is found upstream of the site in concentrations greater than the downstream concentrations, downstream delineation may not be necessary.

B.5 Investigation-Derived Waste

Investigation-derived waste or IDW (may include drill cuttings and muds, sampling materials, purge water, soil and residuals from testing) generated as part of assessment activities may be discharged or stored in the area of contamination and are not subject to RCRA permitting as long as the material: (1) stays on site and remains in the contaminated area, (2) is secured, (3) does not increase the spread of contamination or concentrations in a particular medium, (4) does not cause mobilization of contaminants, and (5) does not introduce contamination to uncontaminated soil (causing an increase in contaminant concentrations). In residential and public use areas, IDW will require off-property management unless it meets unrestricted use levels and disposal permission has been granted by the property owner. IDW cannot be transferred and discharged to another area of concern.

B.6 Sample Analyses

Environmental consultants should provide the laboratory with copies of Sections B.6 and B.7 of this Guidance document to ensure that appropriate analyte lists are used in the analysis of samples.

B.6.1 Phase I Analyses to Identify Contaminants

B.6.1.1 Analytical Parameters

In most cases the parameters listed below must be included in the first phases of testing each contaminated medium. As most of the sites managed by the Branch are pre-regulatory, non-permitted discharges, little information is available on the nature of the discharge. Where property uses, activities and chemical usage at a property are clearly known and limited, some of the analyses listed below can be excluded. Each Phase I sample should be analyzed for the following unless there is documentation indicating that a specific analysis is not necessary:

1. Hazardous substance list metals (totals analysis) including antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, mercury, nickel, selenium, silver, thallium, vanadium and zinc. Hexavalent chromium is the most toxic of the chromium species. Hexavalent chromium testing is only needed for soils if total non-speciated chromium soil concentrations in the Phase I samples exceed the site-specific natural background concentrations and the hexavalent chromium soil remedial goal. For groundwater, hexavalent chromium analyses is not needed as the 15 A NCAC 02L groundwater standard is for total chromium.

   If coal ash is known or suspected to have been discharged at the site, the following additional toxic, non-hazardous substance metals should be included in the testing of groundwater: boron, molybdenum, strontium.

2. All of the volatile and semi-volatile compounds listed on the most current USEPA Contract Laboratory Program (CLP) Target Compound List using analytical methods specified in Section B.6.1.2 with a library search (using the National Institute of Standards and Technology mass spectral library) to produce a list of tentatively identified compounds (TICs). The library search should identify TICs for the largest 10 peaks in each analytical fraction (VOCs and SVOCs) that have reasonable agreement with reference spectra (i.e., relative intensities of major ions agree within ± 20%). The list of identified TICs should not include laboratory control sample compounds, surrogates, matrix spike compounds, internal standards, system monitoring compounds or target compounds. The library search for TICs during the first assessment phase should be done on samples from the “worst case” location in each area of concern or if the “worst case” location is not known, then on a representative number of samples across the area.

   Any TICs that are hazardous substances, that have reasonable agreement with reference spectra, and are detected in more than one sample in an area of concern should be included in all subsequent analytical work unless the compound is a laboratory contaminant, naturally occurring, or documented from an anthropogenic source. Check with the laboratory on possible procedures to quantify the TICs so that cleanup levels can be determined. A summary of the nature of any TICs eliminated from future analysis and reporting should be provided in the Remedial Investigation Report, including reasons for discounting the constituent.

3. 1,4-Dioxane if chlorinated solvents such as 1,1,1-trichloroethane (TCA) or trichloroethylene (TCE) are present, or if it is a suspected contaminant of concern. It is often associated with certain chlorinated solvents because of its widespread use as a stabilizer. It is also a by-product present in many goods and is used as a purifying agent in the manufacture of pharmaceuticals.

4. Pesticides, PCBs, dioxins, cyanide, formaldehyde, nitrates, nitrites, ammonia, phosphorus, and any other CERCLA hazardous substances or pollutants not mentioned here if suspected to have been discharged at the property.

    a. If cyanide is a known or suspected contaminant of concern, cyanide should be analyzed using total cyanide methods.

    b. If PCBs are a known or suspected contaminant of concern in soils, Phase I soil samples should be collected in the area of highest concentration and analyzed using both total PCB analysis and congener-specific analysis. The congener analysis should specify the dioxin-like PCB congeners. The sum of the dioxin-like PCB congener concentrations should be subtracted from the Total PCB analytical result. The resulting concentration must then be compared to the Branch's allowable concentrations for non-dioxin like PCBs. If concentrations detected are less than soil remedial goals for both the individual dioxin-like congeners and for the total non-dioxin like congeners, no additional PCB sampling is required. If concentrations exceed applicable remedial goals, more gross delineation can be performed using total PCB analyses and then the perimeter of the extent of contamination samples run for the dioxin-like congeners found at the site. In areas where PCBs are detected, soil samples should also be collected and analyzed for VOCs because they are commonly present as carriers for PCBs. Final confirmation samples must also be analyzed using congener specific analyses. Aroclor analyses should not be used unless trying to fingerprint a manufacturer of PCB fluid.

       If soils are found to exceed PSRGs, an evaluation of anthropogenic background concentrations may be warranted. Groundwater samples should be analyzed for *Total* PCBs and the results compared to the 15A NCAC 02L standards.

5. Per-and polyfluoroalkyl substances (PFAS), including all analytes in USEPA Method 537.1 or Method 1633, if PFAS are suspected to have been discharged at the property and a drinking water source is potentially impacted by the site based on groundwater movement or aerial deposition (such as from a smokestack). More information regarding the list of USEPA-industry sectors likely to have used PFAS and how to comprehensively characterize sites affected by PFAS can be found in the following document available in Appendix C: USEPA Federal Facilities Superfund Program RPM Bulletin 2021-05 *Considerations for PFAS Conceptual Site Models (CSMs) in the Uniform Federal Policy (UFP) – Quality Assurance Project Plan (QAPP)* Parts 1 and 2.

B.6.1.2 Analytical Methods

The analytical methods used should be the *most recent* versions of the analytical methods tabulated below. For SW-846 Methods, the latest edition of SW-846, including any subsequent updates which have been incorporated into the edition, must be used. Sampling must be planned so that required holding times for analytical methods are met. The laboratory's reporting limits should be at or below remedial goals.

Soil and Sediment Samples

Volatile Organic Compounds[1]	SW-846 Method 8260
1,4-Dioxane[2]	SW-846 Method 8270
Semi-volatile Organic Compounds[1]	SW-846 Method 8270
Metals[3] (excluding hexavalent chromium), Pesticides, PCB congeners, total PCBs, Dioxins, Cyanide, Formaldehyde and any other analytes not covered by above methods	USEPA method or method published in *Standard Methods for the Examination of Water and Wastewater* having detection limits below unrestricted use remedial goals or having the lowest detection limit. For PCB congeners use USEPA Method 1668.
Hexavalent chromium (if total chromium exceeds the site-specific natural background concentrations and the remedial goal for hexavalent chromium)	SW-846 Method 3060A[4] alkaline digestion coupled with a USEPA method or method published in *Standard Methods for the Examination of Water and Wastewater* having detection limits below unrestricted use remedial goals or otherwise having the lowest detection limit.
PFAS compounds[8]	USEPA Method 1633 ASTM D7968-17a

Water Samples (including groundwater, surface water and TCLP/SPLP leachate)

Volatile Organic Compounds[1]	SW 846 Method 8260
1,4-Dioxane[2]	SW-846 Method 8270 SIM using d8 isotope analysis.
Semi-volatile Organic Compounds[1]	SW-846 Method 8270
Metals[3, 5], Pesticides, PCBs, Dioxins, Cyanide, Formaldehyde and any other analytes not covered by above methods	USEPA method or method published in *Standard Methods for the Examination of Water and Wastewater* having the lowest detection limits or having detection limits below the 15A NCAC 02L standards or IMACs
Hexavalent chromium (if total Cr exceeds 2 times the site-specific natural background concentrations and the applicable remedial goal for hexavalent Cr, and Cr is a known or suspected contaminant at the site)[6, 7]	USEPA Method 218.7 or Method 218.6 as modified by USEPA Region IV.
PFAS compounds[8]	USEPA Method 1633 USEPA Method 537.1 Modified for drinking water

1. *Analyses must include the USEPA Target Compound List plus a library search as described in Section B.6.1.1(2).*
2. *Samples for 1,4-Dioxane analysis should not be collected with passive- or diffusive-bag samplers. Also, avoid use of Alconox and Liquinox for precleaning of sampling equipment and containers.*

Methods 8260 and 8260 SIM is not recommended due to interference issues between 1,4-dioxane and some chlorinated solvents, particularly TCE and 1,2-DCE.

3. *SW-846 Method 6010 does not have detection limits below the unrestricted use standards/15A NCAC 02L standards for all of the hazardous substance list metals. Therefore, ICP-MS should be used when conducting first phase metals scans. For metals, ICP-MS is preferred over ICP due to lower quantitation limits. However, ICP should be used for certain metals where interference issues exist.*
4. *SW-846 Method 3060A extraction for soil and sediment samples allows for a 30-day holding time prior to extraction.*
5. *Rapid analyses of samples are recommended to lessen the contact time with the acid preservative. Filtration of groundwater and surface water samples before digestion is not permitted. Highly turbid water samples for metals analysis should be collected in accordance with Section B.3.1.*
6. *Hexavalent chromium analysis is not needed for groundwater samples as the 15A NCAC 02L standard for total chromium is based on the more toxic hexavalent chromium species. This level must be met for all chromium in groundwater. The listed methods can be used for surface water and for ecological assessment purposes.*
7. *Samples collected for hexavalent chromium analyses must be field filtered within 15 minutes of collection. Each sample must be collected in a separate pre-preserved container from those for other metals analyses. Method 218.7 or Method 218.6 as modified by USEPA Region IV should be used. Method 218.7 requires low turbidity and allows for a 14-day holding time. USEPA Region IV has developed a modification to Method 218.6 that allows for a 28-day holding time. Bottles must be pre-preserved as specified in the modification to the Method. Laboratories should contact the USEPA in Region IV for the methodology. Otherwise, any USEPA Method or Standard Method may be used. However, other methods have a 24-hour holding time. Selection of methods and pre-preservation of bottles should be discussed with the laboratory prior to sample collection.*
8. *A modified version of USEPA Method 537.1 is needed for the analysis of PFAS matrices other than drinking water—this includes groundwater, surface water, and TCLP/SPLP leachate. Analysis of a comprehensive list of PFAS analytes with a reporting limit of 4 ppt or less for PFAS is recommended.*

The recommended analytical methods for PFAS are referred to as targeted methods because they provide concentrations of known and commonly detected PFAS. Additional analytical methods are available to characterize the total PFAS that may be present at a site. The total oxidizable precursor (TOP) method can identify PFAS precursors (or parent compounds) that degrade to PFOS and PFOA over time. Conversely the TOP assay can indicate which sites do not have such precursors present, so future occurrence of PFOS and PFOA as degradation products can be ruled out. Consult with DEQ chemists for more information on the uses and limitations of total PFAS methods such as TOP, Non-targeted Analysis (NTA), and total organic fluorine (TOF) methods.

B.6.2 Subsequent Analyses

B.6.2.1Analytical Parameters

After the first phase of sampling is conducted, any samples subsequently collected need only be analyzed for the following compounds.

1. TICs that meet the criteria in Section B.6.1.1(2) that are retained as site contaminants must be quantified in subsequent phase analyses using a USEPA Method or method published in *Standard Methods for the Examination of Water and Wastewater* (latest edition) having the lowest method detection limit (MDL) or one that achieves the 15A NCAC 02L standards or IMACs for water samples and Branch unrestricted use remediation goals for soil.
2. All CERCLA hazardous substances and PFAS detected (including those with qualified estimated concentrations), unless the contaminant concentration is proven through sampling to be the result of a naturally-occurring condition, area –wide anthropogenic background or the contaminant is a common laboratory contaminant detected in concentrations below that detected in the method blank. If a compound that is not a common laboratory contaminant is detected in both the blank and a sample, another phase of sampling is necessary to demonstrate the absence or presence of the contaminant.
3. Potential degradation compounds (which are also CERCLA hazardous substances) of those CERCLA hazardous substances detected at the site.
4. If total chromium concentrations in the Phase I *soil* samples exceed the site-specific natural background concentrations and the hexavalent chromium soil remedial goals, hexavalent chromium analyses are required.
5. If laboratory sample dilutions were performed on Phase I samples, subsequent phase samples must be analyzed for the entire analytical fraction previously diluted in addition to the above items. Sample dilutions raise analytical detection limits and can mask the presence of other constituents at lower concentrations.

B.6.2.2 Analytical Methods

Subsequent phase samples should be analyzed using the methods specified above for Phase I samples. Other USEPA-approved Methods or methods published in *Standard Methods for the Examination of Water and Wastewater* (latest edition) may be substituted, if the substitute methods achieve equal or lower MDLs or if they achieve the 15A NCAC 02L standards, MCL (where no 15A NCAC 02L standard exists), IMACs or health advisory levels for water samples and the unrestricted use remediation goals for soils.

B.7 Data Reporting

Laboratory reports submitted with remedial investigation reports must include the following.

1. The laboratory report must state that the laboratory is either certified for applicable parameters under 15A NCAC Subchapter 02H .0800 and provide its certification number, or that it is a contract laboratory under the USEPA's Contract Laboratory Program. Full CLP documentation packages are not required.
2. A signed statement from the laboratory that the samples were received in good condition, at the required temperature and that analysis of the samples complied with all procedures outlined in the analytical method used, unless otherwise specified. Any deviation from the methods, additional sample preparation, sample dilution and unrectified analytical problems, must be justified in a narrative with the laboratory report.

3. Laboratory sheets for all analytical results, including sample identification, sampling dates, date samples were received by laboratory, extraction dates, analysis dates, analytical methods used, dilution factors and sample quantitation limits.

   Note: The laboratory must provide a written explanation for any sample having sample quantitation limits that exceed 10 times the laboratory or published MDLs.

4. All constituents detected must be reported even if they were not definitively quantified. All estimated concentrations with data qualifiers must be reported.
5. Laboratory sheets for all laboratory quality control samples, including results for bias and precision and control limits used. The following minimum laboratory quality control sample reporting information must be provided: (a) at least one matrix spike and one matrix spike duplicate per sample delivery group or 14-day period, whichever is more frequent (control limits must be specified); (b) at least one method blank per sample delivery group or 12-hour period, whichever is less; and (c) system monitoring compounds, surrogate recovery required by the method and laboratory control sample analysis (acceptance criteria must be specified). All samples that exceed control limits/acceptance criteria must be flagged in the laboratory report.
6. The results of any library searches performed for "tentatively identified compounds." See Section B.6.1.1 (2).
7. The laboratory report should include the names of the individuals performing each analysis, the quality assurance officer reviewing the data and the laboratory manager.
8. Data quality should be reviewed and validated by both the remediating party's environmental consultant and the laboratory. Any quality control concerns, data qualifiers or flags should be evaluated and discussed in the associated report.
9. Completed chain-of-custody with associated air bill (if applicable) attached.

B.8 Confirmation Sampling and Analyses

The following sections provide general guidance on "post-remediation" sampling and analyses needed to demonstrate compliance with Branch remediation goals. At most sites, the remediating party will need to prescribe additional sampling and analysis based on site-specific conditions.

Confirmation sampling should demonstrate that all contaminants identified during the remedial investigation meet applicable remedial goals or acceptable risk for the desired land use. All confirmation samples should be analyzed for all contaminants identified during the remedial investigation. Confirmation samples need to be analyzed using USEPA methods or *Standard Methods for the Examination of Water and Wastewater* (latest edition) with detection limits less than or equal to Branch remediation goals, or those with the lowest available detection limits for each contaminant of concern.

B.8.1 Soil Confirmation Sampling

B.8.1.1 Post In-Situ Remediation

For in-situ soil remedies, a confirmation sampling plan should be designed to verify that the entire soil column has been remediated to below the established remediation goals. To demonstrate that remedial goals have been attained, the remediating party should design a three-dimensional sampling grid that meets the four requirements below.

1. Design a surface sampling grid over the area(s) of concern. Grid nodes should be no more than 50 feet apart.
2. At each grid node, specify “candidate” sampling locations at the surface, at 0 - 6 inches below ground surface, and at 5-foot intervals (or less) down to the vertical limit of contamination. The result is a three-dimensional grid of "candidate" sampling locations that encompasses the area of concern.
3. Select at least two candidate locations at each grid node for sample collection, using a combination of random and biased selection. Biased samples should be collected from known “hot spots” and from soil zones that are known to be resistant to in-situ methods (e.g., clay lenses).
4. Same-depth samples from up to four adjacent grid nodes may be composited. Samples at different depths may not be composited. For samples submitted for volatiles analysis, at least five samples or 25% of the node samples, whichever is greater, need to be unmixed grab samples. Field screening methods may be used to select these unmixed samples, or the unmixed samples should be collected from locations that are evenly distributed across the area of suspected contamination. The remaining samples should be collected as either unmixed grab samples or composited samples. Composited samples will be used for qualitative data only. For very large areas (multiple acres), the remediating party may propose an alternate approach.

B.8.1.2 Post Ex-Situ Remediation

<u>Post-Excavation Sampling</u>

Post-excavation sampling plans need to be designed to verify that all soils/wastes above the established remediation goals have been removed. Excavations should be sampled using the four requirements below.

1. Design a sampling grid over the base and sidewalls of the excavation. Grid nodes should be no more than 50 feet apart. At each grid node, collect a sample from 0-3 inches into the base or sidewall.
2. For very small excavations, collect at least one composite sample from the base and one composite sample from each sidewall. Composite samples should consist of at least four aliquots each. VOC samples should be unmixed grab samples.
3. Biased samples should also be collected from areas of residual contamination, based on visible or field-screening evidence.
4. For excavations <62,500 square feet (surface area), samples from up to four adjacent grid nodes may be composited. For excavations > 62,500 square feet (surface area), a greater number of grid nodes may be composited, but a minimum of five resulting composite samples should be submitted for laboratory analysis. For all excavations, samples from

different sidewalls may not be composited. For samples submitted for volatiles analysis, at least five samples or 25% of the node samples, whichever is greater, should be unmixed grab samples. Field screening methods may be used to select these unmixed samples, or the unmixed samples should be collected from locations that are evenly distributed across the area of suspected contamination. The remaining samples should be collected as either unmixed grab samples or composited samples. Composited samples will be used for qualitative data only. For very large areas (multiple acres), the remediating party may propose an alternate approach.

Treated Soil Stockpiles

Treated soils/wastes must meet the established remediation goals before they can be replaced on site. Treated soil stockpiles should be sampled using the following four procedures.

1. Stockpiles should be divided into equal segments of approximately 100 cubic yards each.
2. Within each segment, use either random or biased selection to locate at least three hand-auger borings. Samples should be collected from two depths within each boring (minimum six samples per segment).
3. Use visible or field-screening evidence to collect additional biased samples from areas of residual contamination.
4. Samples may be composited only within each segment. For volatile sample analysis, at least 25% should be collected as unmixed grab samples.

B.8.2 Groundwater Confirmation Sampling

Groundwater confirmation sampling must demonstrate that site groundwater has been remediated to below the established remediation goals. Demonstrate this using the following procedures.

B.8.2.1 Active Groundwater Remediation

1. A minimum of four quarters of monitoring following system implementation should demonstrate the remedy's effectiveness.
2. Groundwater remediation systems may be shut down when two consecutive semi-annual (twice a year) sampling events demonstrate that *all* monitoring wells (on-property and off-property) are free of contamination above Branch remediation goals. To account for the effects of seasonal fluctuations in the water table, semi-annual sampling events should be conducted in winter and summer.
3. Following system shutdown, data from two additional sampling events (spaced at least 3 months apart) should demonstrate that *all* monitoring wells are free of contamination above the established remediation goals and contaminant concentrations are not increasing.

   Note: For remedial alternatives using injection, the first confirmation sampling event must occur after reagent is spent.

B.8.2.2 Passive Groundwater Remediation

For sites using passive groundwater remedial alternatives (e.g., monitored natural attenuation, phytoremediation, etc.), data from four consecutive sampling events (spaced at least three months apart) should demonstrate that (1) contaminant concentrations throughout the site meet

Branch remediation goals and (2) contaminant concentrations are not generally increasing over time.

B.8.3 Surface Water/Sediment Sampling

Confirmation sampling must demonstrate that site surface water and sediment have been remediated to concentrations below Branch remediation goals. Four consecutive quarterly sampling events should demonstrate that concentrations in downstream samples are less than or equal to concentrations in upstream samples or to Branch remediation goals.

B.9 Sampling and Analyses for Fill Imported from Another Property

Imported fill must meet unrestricted property use. If metals exceed levels suitable for unrestricted use, a demonstration must show that metals are within the receiving site's naturally occurring background levels to be acceptable. Note that any party accepting contaminated soil above unrestricted use levels may make themselves a responsible party under CERCLA.

The history and location of the proposed borrow source must be established and documented. The borrow source property must either be undeveloped, or the historical usage of the property has no indication of an environmental concern. Borrow sources should not include the following:

1. locations on or within 1,000 feet of a site that the Division of Waste Management (DWM) manages, permits or has inventoried;
2. soil from a contaminant cleanup or removal;
3. commercial or industrial properties where hazardous materials were used, handled or stored;
4. dredged material from a marine environment; (unless it has dried to meet the moisture content of receiving site);
5. soil from below the groundwater table; (unless it has dried to meet the moisture content of receiving site);
6. soil containing construction or demolition debris or reclaimed asphalt or concrete;
7. soil from recycling operations that collect, sort, reprocess or manufacture products;
8. soil from transfer stations that collect, consolidate, temporarily store, sort, or recover refuse or used materials from off site; or soil containing coal or wood ash.

B.9.1 Procedures

1. Document the historical uses and location of the proposed borrow source.
2. Collect samples from the fill according to whether it is in-situ or stockpiled material. Sampling methodologies must be in accordance with U. S. Environmental Protection Agency (US EPA) Region IV Laboratory Services and Applied Science Division (LSASD), formerly the Science and Ecosystem Support Division (SESD), *Field Branches Quality System and Technical Procedures, Soil Sampling (SESDPROC-300-R3)* and these Guidelines.
    a. In Situ Material
        i. Divide the borrow source area into five approximately equal-sized sections (the number of sections is independent of the total acreage).

ii. Collect a grab sample from each section at the following depths. Composite samples are not allowed.

- 0 to 0.5 ft below ground surface (bgs).
- 5 ft bgs (or a shallower depth if desired fill material will not extend beyond 5 ft bgs).

b. Stockpiles

The owner of the stockpiled borrow source will be required to sign an affidavit, attesting that the stockpiled material originated from one source location and not from multiple source(s). The stockpiled material that is sampled and tested, must be the same material that is purchased and imported for use as clean structural fill at the Site. Sample the stockpile according to the following schedule.

Volume of Material Needed	Samples per Volume
Up to 1,000 cubic yards	3 composite samples
1,000 to 5,000 cubic yards	2 composite samples for the first 1,000 cubic yards plus 1 composite sample for each additional 1,000 cubic yards
>5,000 cubic yards	5 composite samples for the first 5,000 cubic yards plus 1 composite sample for each additional 5,000 cubic yards

Note: Each composite sample should contain three grab samples from three different depths. VOC samples can, and should be, composited to characterize stockpiled borrow sources.

3. Analyze all samples for the following parameters.

   a. Volatile and Semi-Volatile Organic Compounds (USEPA Target Compound List plus 1,4-Dioxane): SW-846 Methods 8260 and 8270, respectively;

   b. Metals: SW-846 Method 6020 (Antimony, Arsenic, Beryllium, Cadmium, Chromium, Cobalt, Copper, Iron, Lead, Manganese, Nickel, Selenium, Silver, Thallium and Zinc);

   c. Mercury: Method 7471; and

   d. Organochlorine Pesticides: Method 8081 (if the borrow source was used for agricultural purposes).

   e. PFAS analysis by Method 1633 may be required on a case-by-case basis depending on whether the soil's origin has the potential to be impacted by PFAS (e.g., near airports, fire training facilities, foam manufacturing facilities, etc.).

4. Compare the soil analytical results to the direct contact unrestricted use and protection of groundwater Preliminary Soil Remedial Goals (PSRGs).
    a. If the soil analytical results are above unrestricted use direct contact PSRGs, the data must be entered into the DEQ risk calculator to confirm that concentrations are below unrestricted use risk targets, and/or a demonstration must show that metals are within the receiving site's naturally occurring background levels.
    b. If the soil analytical results are above the protection of groundwater PSRGs, TCLP or SPLP analysis is required to determine leachability.
5. Submit the borrowed fill evaluation, including the analytical data, risk calculator results and a summary cover letter to Branch staff for review and approval prior to purchase and transport of fill material to the Site.

B.10 Quality Assurance for Sampling and Analysis

1. Unless otherwise noted below, field procedures relating to sample collection techniques, sample containers, sample preservation, sample holding times equipment decontamination and field measurement procedures, should comply with the most current version of the U. S. Environmental Protection Agency (USEPA) Region IV Laboratory Services and Applied Science Division (LSASD), formerly the Science and Ecosystem Support Division (SESD), *Field Branches Quality System and Technical Procedures.* This information is available from the USEPA Region IV LSASD at: https://www.epa.gov/quality/quality-system-and-technical-procedures-lsasd-field-branches .
2. The remediating party should employ analytical and environmental monitoring data, to support recommendations or conclusions with respect to assessment, removal, treatment, or containment actions, which are scientifically valid and of a level of precision and accuracy commensurate with their stated or intended use.
3. The remediating party should only use laboratories certified to analyze applicable certifiable parameters under 15A NCAC 02H .0800, or a contract laboratory under the United States Environmental Protection Agency Contract Laboratory Program to analyze samples collected pursuant to rules under this Section.
4. Laboratory and other reports of analyses of aqueous samples should be reported as mass per unit volume; such reports of analyses of solid samples should be reported as mass per unit mass.
5. Field QC samples: (i) minimum of one duplicate sample, per medium, per container type, per field day; (ii) equipment rinsate blanks and trip blanks (VOC analysis) are strongly recommended.

    Note: If site conditions, sample frequency or number of samples warrant more limited QA/QC testing, contact the Branch.
6. Other than composited samples, all soil, sediment and waste samples for volatiles analysis should be collected directly into sample containers without mixing.
7. All laboratory reports containing the results of sample collection and analyses must include the following information:

   a. the date, location, and time of sampling;
   b. specification of all sample filtration or preservation procedures used;
   c. the date of receipt of the sample at the laboratory, and the date(s) the sample was extracted and analyzed;
   d. the name and address of the laboratory, and proof of certification under 15A NCAC 02H .0800 or the USEPA Contract Laboratory Program;
   e. the sample matrix description and identification number(s);
   f. the sample preparation and analytical method name(s) and number(s);
   g. the laboratory report including, results of the analysis, in clearly expressed concentration units;
   h. the sample quantitation limit of each reported analyte based upon analytical conditions (any quantitation limits exceeding 10 times the MDL must be justified with supporting information);
   i. details of any known conditions or findings which may affect the validity of analytical data, including but not limited to equipment blank, trip blank, method blank, surrogate, spiked sample, or other quality control data;
   j. the laboratory's written justification for any sample dilution, additional sample preparation, or deviation from specified analytical methods;
   k. laboratory sheets for all laboratory quality control samples, including results for bias and precision and control limits used. The following minimum laboratory quality control sample reporting information must be provided: (a) at least one matrix spike and one matrix spike duplicate per sample delivery group or 14-day period, whichever is more frequent (control limits must be specified); (b) at least one method blank per sample delivery group or 12-hour period, whichever is less; and (c) system monitoring compounds, surrogate recovery required by the method and laboratory control sample analysis (acceptance criteria must be specified). All samples that exceed control limits/acceptance criteria must be flagged in the laboratory report;
   l. the results of any library searches performed for “tentatively identified compounds”;
   m. a signed statement from the laboratory that the samples were received in good condition, at the required temperature and that analysis of the samples complied with all procedures outlined in the analytical method used, unless otherwise specified; and
   n. complete chain of custody documentation for each sample.

8. Data quality should be reviewed and validated by both the remediating party and the laboratory. Any quality control concerns, data qualifiers or flags should be evaluated and discussed in the associated report.
9. All constituents detected must be reported even if they were not definitively quantified. All estimated concentrations with data qualifiers must be reported.

Appendix C: PFAS Resources

Pg. 1-34	Interstate Technology & Regulatory Council (ITRC). *PFAS Technical and Regulatory Guidance Document and Fact Sheets. 11. Sampling and Analytical Methods.* 2022
Pg. 34-43	USEPA Federal Facilities Superfund Program – RPM Bulletin 2021-05. *Considerations for PFAS Conceptual Site Models (CSMs) in the Uniform Federal Policy (UFP)-) Quality Assurance Project Plan (QAPP), Part 1 Sources, Sampling and Analysis*. September 15, 2021.
Pg. 44-51	USEPA Federal Facilities Superfund Program – RPM Bulletin 2021-06. *Considerations for PFAS Conceptual Site Models (CSMs) in the Uniform Federal Policy (UFP)-) Quality Assurance Project Plan (QAPP), Part 2 Risk Assessment and Risk Communication Considerations*. November 15, 2021
Pg. 52-132	USEPA. *Multi-Industry Per- and Polyfluoroalkyl Substances (PFAS) Study – 2021 Preliminary Report*. September 2021.
Pg. 133-158	USEPA. *PFAS Strategic Roadmap: USEPA's Commitments to Action 2021-2024*. USEPA-100-K-21-002. October 2021.
Pg. 159-163	USEPA. *Proposed PFAS National Primary Drinking Water Regulation FAQs for Drinking Water Primacy Agencies*. March 14, 2023.

Printed from: Interstate Technology & Regulatory Council (ITRC). 2022. *PFAS Technical and Regulatory Guidance Document and Fact Sheets* PFAS-1. Washington, D.C.: Interstate Technology & Regulatory Council, PFAS Team. https://pfas-1.itrcweb.org/.

11 Sampling and Analytical Methods

The PFAS Team developed a Sampling and Analysis training video with content related to this section.

Due to the extensive use of a wide array of PFAS resulting in trace levels of PFAS, in most environmental media across the globe and the low parts–per–trillion screening levels, all aspects of a sampling and analysis protocol require a heightened level of rigor to avoid cross-contamination and achieve the level of accuracy and precision required to support defensible project decisions. This section focuses on providing the user with the appropriate tools and information to develop a site-specific sampling and analysis program to satisfy the project data quality objectives (DQOs). Accurate and representative data support the development of a defensible conceptual site model (CSM), and ultimately the final remedy.

Section Number	Topic
11.1	Sampling
11.2	Analytical Methods and Techniques
11.3	Data Evaluation

Information on sample collection for PFAS is sparse, with only a handful of guidance documents available for a practitioner to reference. Further, as there are limited peer-reviewed studies (Denly et al. 2019; Rodowa et al. 2020) on the potential for cross-contamination from commonly used sampling materials, most of these guidance documents default to a conservative approach in implementing measures and controls for prevention of cross-contamination (for example, washing cotton shirts with no fabric softener prior to use in the field). Although the actual methods of sample collection are similar to those used for other chemicals, there are several considerations for the practitioner when establishing a sampling program for PFAS. These include selection of proper personal protective equipment (PPE), documentation of protocols for sample handling and decontamination procedures, use of nonbiasing material (for example, tubing, sample bottles, pumps) that could come into contact with the sample, and implementation of quality control (QC) protocols to meet project DQOs, among other considerations. This section will give practitioners the tools needed to prepare a sampling program that adequately addresses project-specific DQOs and limits, to the extent practicable, potential cross-contamination and sources of potential bias.

Additionally, analytical methods are still evolving for PFAS analysis, with several in development (USEPA 2021). Although some draft methods have been published (PFAS Analytical Methods provided as an Excel spreadsheet), all are not discussed in this document because details included in these methods are subject to change prior to the methods being finalized. In the case of USEPA Draft Method 1633, an exception has been made in this document due to the USEPA Office of Water's recommendation of its use in Clean Water Act (CWA) compliance testing applications, including National Pollutant Discharge Elimination System (NPDES). The publication of this draft method followed the completion of single-laboratory validation of the method. The method is currently undergoing multilaboratory validation. Once completed, information from this study will be used to update the QC criteria included in the draft method before the final method is proposed at 40 CFR Part 136 for nationwide Clean Water Act monitoring. This method is applicable to wastewater, groundwater, surface water, landfill leachates, soil, sediment, biosolids, and tissue.

The USEPA Office of Land and Emergency Management has validated and published a sample preparation procedure and analysis procedure applicable to groundwater, surface water, and wastewater. USEPA SW-846 Method 3512 is a sample preparation method, and USEPA SW-846 Method 8327 is the associated analytical method.

Currently, there are two USEPA methods that are validated and published for the analysis of PFAS in drinking water sources: USEPA Method 537.1, Version 2.0 (USEPA 2020) (which replaced USEPA Method 537.1, and USEPA Method 537 Version 1.1) and USEPA Method 533 (USEPA 2019). For simplicity in the text, subsequent references to USEPA Method 537.1 assume the most current version. These methods are required for PFAS analyses of drinking water and include performance data for drinking water from surface water and groundwater sources. These methods are prescriptive in that changes to preservation (including sample collection, in the case of USEPA Method 537.1), sample extraction steps, and quality control requirements are prohibited by both methods.

The DOD has also validated and published a method (DoD AFFF01) for the determination of PFOA and PFOS in AFFF concentrates for demonstration of compliance to MIL-PRF-24385 (Willey 2021).

This section will be updated as new information on sampling considerations and analytical methods/procedures becomes available.

11.1 Sampling

11.1.1 General

Sampling conducted to determine PFAS concentrations in water, soil, sediment, air, biota, and other media is similar to that for other chemical compounds, but with several additional specific considerations and protocols. Typical guidance and procedures, such as ASTM International D 4823-95 and D 4448-01, USEPA compendium EPA 540/P-87/001a, OSWER 9355.0-14, USEPA SESDPROC-513-R2, and USEPA SESDPROC-305-R3, remain the basis for a PFAS sampling protocol. Examples of special considerations for PFAS sampling include the types of sampling equipment or materials used due to the widespread uses for and products containing PFAS; field and equipment blanks above and beyond what is normally required; the need for low laboratory quantitation limits; low state and federal screening levels, and in some cases, cleanup criteria; potential for background sources of PFAS in the environment; and the need for modified decontamination measures.

Examples of USEPA region-specific or program-specific PFAS sampling protocols include:

- USEPA (2019) Region 4, Laboratory Services and Applied Science Division, Athens, GA, *Field Equipment Cleaning and Decontamination at the FEC*, ASBPROC-206-R4, 2019
- Transport Canada (2017) *Per- and Polyfluoroalkyl Substances (PFAS) Field Sampling Guidance*
- Government of Western Australia (2016) *Interim Guideline on the Assessment and Management of Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS), Contaminated Sites Guidelines*
- DoD AFFF01 *Determination of Perfluorooctanoic acid and Perfluorooctanesulfonic acid in Aqueous Film Forming Foam (AFFF) for Demonstration of Compliance to MIL-PRF-24385* (Willey 2021)
- State guidance:
  - MA DEP (2020) Massachusetts Department of Environmental Protection, *Interim Guidance on Sampling and Analysis for PFAS at Disposal Sites Regulated under the Massachusetts Contingency Plan*.
  - Washington Department of Ecology (2017) *Quality Assurance Project Plan; Statewide Survey of Per- and Poly-fluoroalkyl Substances in Washington State Rivers and Lakes.*
  - NH DES (2019) New Hampshire Department of Environmental Services, *Laboratory Testing Guidelines for Per- and Polyfluoroalkyl Substances (PFAS) at Waste Sites, 2019*
  - MI EGLE 2018 to present, PFAS Sampling Guidance Documents, including:
    - MI EGLE (2021) PFAS Sampling Guidance
    - MI EGLE (2019) Surface Water Foam PFAS Sampling Guidance
    - MI DEQ (2018) Wastewater PFAS Sampling Guidance
    - MI DEQ (2018) Surface Water PFAS Sampling Guidance
    - MI DEQ (2019) Fish Tissue Sampling Guidance

A work plan, such as a comprehensive project-specific quality assurance project plan (QAPP), should be created to address PFAS-specific considerations. If a work plan is not created for a project, the sampling and quality assurance and quality control (QA/QC) elements outlined in Section 11.1.6 should be included in site-specific work plans. If regulatory procedures, methods, or guidelines are inconsistent with the needs of a PFAS sampling program, the governing agency should be contacted directly to determine if an exception can be made or an alternate approach is needed. A CSM should be completed as part of the work plan, including information on previous site uses, PFAS use/manufacturing/handling practices, other possible contaminants and their uses, and/or related remediation activities (for example, granular activated carbon (GAC), in situ treatments, or dig and haul) to determine all possible source areas of PFAS. Because PFAS is not typically analyzed along with other parameters at traditional remediation sites, and analytical methods are only recently becoming standardized, previous or ongoing remediation of other contaminants of concern can add a layer of complexity to a site's geochemistry and the fate and transport of PFAS.

Although some sampling elements (for example, sample bottle, preservation, and holding times) are defined by USEPA Methods 537.1, 533, and 8327, they do not provide all the information that is needed to conduct a sampling event for PFAS. DoD AFFF01 contains all the information needed to conduct sampling of AFFF concentrates. **Tables 11-2** and **11-5**, included in the PFAS Analytical Methods Excel spreadsheet, cover the sample container types, sample size, number of containers required, and holding time and preservation requirements for each of these finalized and draft published PFAS analytical methods, respectively.

Communication with the laboratory before, during, and after sampling is conducted, is critical in ensuring that project needs are met. If a sample is from an area known or suspected to be highly contaminated with PFAS, it is important that this is communicated to the laboratory. The chain–of–custody (COC) form should indicate samples that potentially contain a high concentration of PFAS. The laboratory should screen all samples to select the necessary sample preparation procedures and to avoid contamination of their laboratory equipment and contamination of other field samples.

Any water used for field QC blanks (for example, field and decontamination blanks) should be supplied by the laboratory performing the analysis. The laboratory should provide documentation verifying that the supplied water is PFAS-free. “PFAS-free” is the project-defined concentration that associated blank concentrations must be below (for example, less than the detection limit or less than half the limit of quantitation (LOQ)) to ensure an unacceptable bias is not introduced into the sampling and analysis processes. The work plan should clearly state the project’s definition of “PFAS-free.” Review of the laboratory’s standard definition of “PFAS-free” upfront is necessary to ensure that it meets project needs and is a critical step in laboratory selection for a project. Laboratories verify the PFAS content of each batch of supplied water through analysis. Documentation of this verification should be maintained for data validation purposes and should be reviewed by the project team to ensure that the project’s definition of “PFAS-free” is met. If the water was not supplied by the laboratory, a sample of the water used in the field should be sent to the laboratory for testing.

11.1.2 Equipment and Supplies

Many materials used in environmental sampling can potentially contain PFAS. There is limited published research or guidance on how certain materials used by field staff or in sampling equipment affect sample results (see Denly et al. 2019; Rodowa et al. 2020). However, a conservative approach is recommended to exclude materials known to contain the PFAS that are the target of the analysis from a sampling regimen, and such an approach should be documented accordingly in the work plan. Obtain and review all Safety Data Sheets (SDSs) before considering materials for use during PFAS sampling, as product manufacturing formulations can change over time. If PFAS are listed on the SDS, it is recommended that piece of equipment/supply not be used. Exclusion from the SDS does not necessarily mean the equipment/supply is not contaminated with PFAS. PFAS could have been used not as a component of the equipment/supply, but as a material used in the manufacturing process itself (for example, mist suppressant or mold coating). This can result in the equipment/supply manufactured containing PFAS. If necessary, materials in question can be sampled and analyzed for PFAS, or thorough decontamination and collection of equipment blanks can provide sufficient quality assurances. Ultimately, a sampling program should produce defensible data, and the best way to protect the integrity of samples is to ensure they are not compromised by contaminants originating from sampling equipment or otherwise.

Due to the extensive use of a wide array of PFAS, sampling crews should review all materials and sampling protocols to avoid contamination and possible adsorption issues. Examples of problematic materials that if used and contacted samples could potentially introduce bias include, but are not limited to:

- polytetrafluoroethylene (PTFE)
- waterproof coatings containing PFAS
- fluorinated ethylene-propylene (FEP)
- ethylene tetrafluoroethylene (ETFE)
- low-density polyethylene (LDPE)
- polyvinylidene fluoride (PVDF)
- pipe thread compounds and tape.

A conservative PFAS sampling program may additionally restrict materials that are allowed on the sampling personnel or in the staging area. A tiered approach is used for materials restrictions in that case, where the first tier would include restrictions on the sampling materials that will come in direct contact with the sample media, and the second tier would include restrictions on what materials are allowed on sampling personnel or within the staging area. Program-specific sampling protocols such as those previously listed in this section often identify materials and equipment that can be used in PFAS-focused investigations, as well as materials that should be avoided because they are known or suspected to be potential sources of PFAS.

Sometimes it is impossible or financially infeasible to eliminate materials that affect PFAS results in samples. For example, these materials might be needed at sites where hazards warrant the use of specific PPE such as Tyvek suits, where PFAS are the secondary or co-contaminant and the primary contaminant requires specific materials for proper sampling, or where the opportunity to collect a sample occurs before a proper sampling program is developed. At PFAS sites where co-contaminants are not a factor, the same PPE is required as at traditional sampling sites (a minimum of nitrile gloves and safety glasses).

11.1.3 Bottle Selection

Sample container recommendations are dependent on the analytical method and should be supplied by the laboratory and laboratory-verified to be PFAS-free, as defined by the work plan. USEPA Method 537.1 requires the use of polypropylene containers and caps/lids for drinking water sampling, and USEPA SW-846 Method 8327 was validated using polypropylene containers for groundwater, surface water, and wastewater sampling. However, USEPA SW-846 Method 8327 states that other types of containers such as high-density polyethylene (HDPE) may be used if the needs of the project can be met with their use. USEPA Method 533 allows for the use of polypropylene or polyethylene containers and polypropylene caps/lids for drinking water sampling. USEPA Draft Method 1633 requires the use of HDPE containers for wastewater, groundwater, surface water, landfill leachate, biosolids, soil, sediment, and tissue sampling. The volume of aqueous sample that is required for analysis varies from method to method and the mass of solid material required for analysis in accordance with USEPA Draft Method 1633 is dependent on the matrix of the sample.

> Because the concentration levels of PFAS in aqueous samples, excluding drinking water samples, determine whether the whole sample or an aliquot is used in the laboratory preparation, USEPA Draft Method 1633 requires collection of an additional aliquot of each aqueous sample for screening purposes. The method allows for a smaller aliquot of wastewater, groundwater, or surface water than collected for analysis to be collected for screening and determination of percent suspended solids. This second aliquot allows the laboratory to screen the sample without affecting the integrity of the sample collected for analysis. Coordinating with the laboratory is crucial to determine the appropriate sample numbers and volumes as well as QA/QC samples.

Best practices in sample preparation should be used when selecting the size, volume, and representativeness of samples. To minimize effects from analyte sorption on sample containers, USEPA Methods 537.1 and 533, and USEPA Draft Method 1633 all require the laboratory to prepare the entire sample collected, including sample container rinsate(s). DoD AFFF01 requires the container holding the diluted AFFF concentrate be prepared in its entirety, including a rinse of the container.

11.1.4 Sample Preservation, Shipping, Storage, and Holding Times

Sample preservation, shipping, storage, and holding time requirements are dependent on the method that is intended to be used. Drinking water methods (USEPA Method 537.1 and 533) are the only USEPA methods requiring the addition of a chemical preservative at sample collection. USEPA Method 537.1 required the addition of TRIS (Trizma), while USEPA Method 533 requires the addition of ammonium acetate. According to both of these methods, samples must be chilled during shipment and not exceed 10°C during the first 48 hours after collection. When they are received by the laboratory, samples must be at or below 10°C and stored in the laboratory at or below 6°C until extraction. These two methods differ in their required holding times, as USEPA Method 537.1 requires samples to be extracted within 14 days of collection, while USEPA Method 533 requires samples to be extracted within 28 days of collection.

The shipping, storage, and holding time requirements for wastewater, groundwater, and surface water samples stated in USEPA SW-846 Method 8327 differ than those provided by USEPA Draft Method 1633. USEPA SW-846 Method 8327 requires all samples and sample extracts to be chilled from the time of sample collection to analysis and not exceed 6°C. Since a holding time study was not performed in conjunction with the validation of USEPA SW-846 Method 8327, the method offers a holding time of 14 days from sample collection to sample extraction and a 30-day holding time from sample extraction to sample analysis as a guideline.

The shipping, storage, and holding time requirements contained in USEPA Draft Method 1633 are based on a published holding time study for PFAS in wastewater and surface water (Woudneh et al.2019) and the results of a holding time study conducted in conjunction with the single-laboratory validation study of USEPA Draft Method 1633. The holding time study performed during this validation study assessed what impact, if any, two storage temperatures had on 40 PFAS in wastewater, groundwater, surface water, soil, sediment, and biosolids samples and sample extracts. The recovery of 40 PFAS in samples stored at -20°C and 4°C and extracts stored at 4°C was evaluated at defined intervals over the course of 90 days. While the statistically determined holding time requirements are similar for each matrix, they are not identical (see **Table 11-5**, included in the PFAS Analytical Methods Excel spreadsheet). Observed losses and/or gains in the concentration of some PFAS, most likely caused by transformation of precursors, resulted in shorter holding times for samples and extracts held at 4°C as opposed to -20°C. Due to the observed extreme loss of NFDHA, an additional caveat in the method recommends samples be extracted and analyzed as soon as possible if NFDHA is an important analyte for the project.

11.1.5 Decontamination Procedures

Sampling equipment should be thoroughly decontaminated before mobilization to each investigation area and between sample locations at each investigation area or as required in the site-specific work plan. Field sampling equipment, including oil/water interface meters, water level indicators, nondisposable bailers, and other nondedicated equipment used at each sample location requires cleaning between uses. The SDSs of detergents or soaps used in decontamination procedures should be reviewed to ensure fluorosurfactants are not listed as ingredients. Use laboratory-verified PFAS-free water for the final rinse during decontamination of sampling equipment. Decontaminate larger equipment (for example, drill rigs and large downhole drilling and sampling equipment) with potable water using a high-pressure washer or steam. To the extent practical, rinse parts of equipment coming in direct contact with samples with PFAS-free water. Heavy equipment is best cleaned within a decontamination facility or other means of containment (for example, a bermed, lined pad and sump, or a portable, self-contained decontamination booth). Potable water sources should be analyzed in advance for PFAS, as well as during the sampling event. Wherever possible, rinse equipment with laboratory-verified PFAS-free water immediately before use.

An example decontamination procedure is as follows.

- Equipment caked with drill cuttings, soil, or other material will initially be scraped or brushed. The scrapings will be sampled, containerized, and appropriately disposed.
- Equipment will then be sprayed with potable water using a high-pressure washer.
- Washed equipment will then be rinsed with laboratory-verified PFAS-free water.
- Decontaminated downhole equipment (for example, drill pipe, drive casing, bits, tools, bailers, etc.) will be placed on clean plastic sheeting (PFAS-free) to prevent contact with contaminated soil and allowed to air dry. If equipment is not used immediately, it will be covered or wrapped in plastic sheeting to minimize airborne contamination.
- Field sampling equipment and other downhole equipment used multiple times at each sample location will require cleaning between uses utilizing a four-stage decontamination process. The equipment will first be rinsed in a bucket containing a mixture of potable water and PFAS-free soap. The equipment will then be rinsed in each of two buckets of clean potable water. Water used for the final rinse during decontamination of sampling equipment will be laboratory-verified PFAS-free water.

Decontamination solutions should be replenished between sampling locations as needed. Spent decontamination fluids should be containerized, properly labeled, and appropriately disposed of as investigation-derived waste (IDW), based on plans included in the site-specific QAPP or work plan.

11.1.6 Field QC Samples

Field QC samples are a means of assessing quality beginning at the point of collection. Such field QC samples typically include field reagent blanks, source water blanks, equipment rinse blanks, and field duplicates. Collection and analysis of field QC samples are important for PFAS investigations because of very low detection limits and regulatory criteria (parts per trillion (ppt)), to ensure accuracy and representativeness of the results for the sampled media, and to assess potential cross-contamination due to the extensive use of PFAS. A sampling program should be designed to prevent cross-contamination and anthropogenic influence. However, the widespread commercial use (historical and current) of PFAS-containing products, and especially their prevalence in commonly used sampling materials and PPE, should inform the sampling program. PFAS sites may also have a wide range of concentrations with varying families of PFAS, as well as co-contaminants. Furthermore, PFAS sites have the potential to be high profile in nature. Therefore, a comprehensive site-specific QAPP or work plan addressing DQOs and field QC samples, including frequency, criteria, and procedures, is vital to a PFAS sampling program (see also Section 11.3, Data Evaluation).

When planning QA/QC sample frequency, the risk of cross-contamination should be considered. Cross-contamination can occur from several sources, including field conditions, ineffective decontamination, incidental contact with PFAS-containing materials, and sampling equipment and materials that were manufactured alongside PFAS-containing equipment.

Of all the USEPA PFAS methods, only USEPA Methods 537.1 and 533 contain specific requirements for the field QC samples that must accompany samples to be analyzed for PFAS. These include a minimum of one field reagent blank for each set of samples per site and field duplicates. USEPA Methods 537.1 and 533 specify the frequency of the field duplicate in terms of extraction batch (one per extraction batch, not to exceed 20 field samples), not collection frequency. Although the other USEPA PFAS methods do not contain any field QC sample requirements, Table 11-1 provides a list of field QC samples typical for the collection of these matrix types and their typical minimum frequency. Once field QC sample data are obtained, they should be evaluated against the field samples by a person knowledgeable on the DQOs set forth in the site-specific QAPP or work plan. For laboratory QC considerations, see Section 11.2, Analytical Methods/Techniques.

Table 11-1. Typical field QC samples

QC Sample	**Description**	**Minimum Suggested Frequency**
Field reagent blank (FRB)	Laboratory-provided reagent water that, in the field, is poured into an empty sample bottle or a sample bottle containing only preservative (if required)	One per day per matrix per sample set
Source water blank	Water collected from potable water source that is used during the sampling processes (such as decontamination and drilling processes)	One per site, preferably prior to sampling event (if possible) and at least once during sampling event
Equipment rinse blank (ERB)	Final rinse of nondedicated sampling equipment with laboratory-verified PFAS-free water (decontamination blank) or rinse of sampling equipment (dedicated or nondedicated) prior to the sampling event in cases where PFAS content is unknown or suspected	One per day per type of sampling equipment used for each matrix sampled For cases in which PFAS content is unknown or suspected, prior to sampling event
Field duplicate	Two samples collected at the same time and location under identical circumstances	One per day per matrix up to 20 samples
Performance evaluation (PE) sample	A sample containing known concentrations of project analytes	One per project per matrix

11.1.6.1 Field Reagent Blank

A field reagent blank (FRB), as described in USEPA Methods 537.1 and 533 for collection of drinking water samples, consists of a sample bottle filled with reagent water prepared in the laboratory, sealed, and shipped to the sampling site along with the sample bottles. An empty sample bottle containing only preservatives (same as those used for the samples) is also shipped along with each FRB into which the sampler pours the preserved reagent water contained in another bottle that was sent to the field and seals and labels the bottle for shipment along with the samples back to the laboratory for analysis. FRBs help to determine if PFAS were introduced into the samples during sample collection/handling and help to account for additional factors, such as introduction of contaminated airborne particles. A laboratory reagent blank is also analyzed in a laboratory setting to ensure the reagent water meets USEPA Methods 537.1 and 533 requirements.

FRBs may also be warranted during collection of sampling media other than finished drinking water. In lieu of using a prepared quantity of laboratory reagent water/preservative solution as for drinking water FRB, a field blank can be prepared in the field using laboratory-verified PFAS-free water (may be equivalent to the laboratory reagent water) and filling an empty sample container in the field, which is then sealed and labeled as a field blank. This sample will be analyzed in the same manner as the normal samples and can indicate whether or not PFAS were introduced during sample collection/handling, and help to account for additional factors, such as introduction of contaminated air particulate.

As discussed above, the frequency of FRB samples for finished drinking water sampling is one FRB for every sample set at each site. A sample set is described in USEPA Methods 537.1 and 533 as “samples collected from the same sample site and at the same time” (USEPA 2020; USEPA 2019).

11.1.6.2 Source Water Blank

Large quantities of water may be necessary to carry out a field sampling program for various reasons, including decontamination and certain drilling techniques (Section 11.1.5, Decontamination Procedures). Site groundwater or surface water should not be used as source water at sites that are under investigation for PFAS, as they have potential to contain PFAS. PFAS test results of publicly supplied water at a site should be evaluated when considering it for use as source water. Regardless, it is imperative that any water used as source water be sampled and analyzed. For equipment that may come into contact with samples of any media type, a multistep process is common to adequately prevent cross-contamination. Quantities of laboratory-verified PFAS-free water are generally limited and can be costly. Therefore, potable water sources are typically used in initial decontamination steps. It is imperative that these water sources be sampled and analyzed in the same manner as normal samples, prior to and even during a PFAS sampling program, to ensure that source water is not contributing to PFAS detections in normal samples.

Collect a sample from the source the same way it is collected for use (for example, if the source water is collected through a hose, collect the source water blank from that same hose). If there are unnecessary fittings or hoses attached for collection of the source water, consider removing them for the duration of the sampling program to avoid contamination from PFAS that may be present in these materials.

Frequency of collection of such source water blanks is up to the professional judgment of the project manager, site owner, and other stakeholders. The source water should be sampled at least once prior to starting the field sampling program and once during the sampling event in case the analysis reveals that a different water source should be found. A more conservative sampling program may include provisions for additional periodic sampling in cases where the conditions of the source water change.

11.1.6.3 Equipment Rinse Blank (ERBs)

ERBs can be collected from equipment or supplies prior to the sampling event in cases where PFAS content is unknown or suspected, or to verify the cleanliness of nondedicated equipment/supplies (Section 11.1.2).

Field equipment rinse blanks (ERBs) are those collected by rinsing a piece of field sampling equipment/supplies with laboratory-verified PFAS-free water and collecting the rinse water in a sample container for PFAS analysis. ERB collection is not required by the USEPA Methods 537.1 or 533 because drinking water compliance samples are generally collected from the source without the use of other equipment. ERB collection for other programs is dependent on the sampling media and methods that are employed at a site. Generally, any equipment that is reused throughout the sampling program, or is nondedicated, and must be decontaminated, should have an ERB collected from it. That is, if a piece of equipment is decontaminated, an ERB should be collected from it prior to its next use. The frequency of collection of ERBs can be reduced by using all dedicated or disposable equipment where possible. However, many of these options are limited due to the extensive use of PFAS in many of these equipment materials. ERBs should also be considered for dedicated equipment prior to and during a sampling event if the PFAS contribution from equipment is unknown or suspected.

Field ERB collection frequency is largely up to the professional judgment of the project manager or other stakeholders and is dependent on the sampling media and methods. For instance, ERBs collected from decontaminated soil sampling trowels may only warrant a frequency of once per day, whereas ERBs collected from groundwater pumps may warrant an ERB prior to the pump being deployed down each well, due to their more rigorous decontamination procedure and higher contact time with the groundwater being sampled.

11.1.6.4 Field Duplicate

Field duplicate (FD) samples are two samples collected at the same time and location under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analysis from these identical samples helps evaluate the precision of sample collection, preservation, storage, and laboratory methods.

USEPA Methods 537.1 and 533 do not specify the frequency of FD collection for finished drinking water samples; however, they do specify the frequency of preparation (once per extraction batch, not to exceed 20 field samples). A more conservative sampling program may indicate a frequency of one FD per 10 field samples per matrix. FD collection frequency should be discussed with stakeholders as necessary and be evaluated as part of the comprehensive site-specific QAPP or work plan.

11.1.6.5 Performance Evaluation (PE) Sample

A PE sample contains project analytes with known concentrations of PFAS. This sample can be submitted to the laboratory as a blind sample. Analysis from this sample provides a positive control from a second source.

11.1.6.6 Additional QC Samples

In addition to the field quality control samples described above, split samples and matrix spikes can provide project managers additional quality assurance regarding identification of PFAS target analytes and precision and bias in measured sample concentrations. Split samples are defined here as co-located quality control samples, taken at the same time and each sent to a different laboratory. These types of samples do not apply to routine compliance monitoring situations and may not be required in all sampling events. Aqueous QC samples should not be split into two samples from the original container. Analysis of these QC samples provides a measure of interlaboratory variability.

11.1.7 Sampling Procedures

Standard sampling procedures can be used at most PFAS sites. However, there may be some exceptions and additional considerations related to PFAS behavior and issues associated with potential use of PFAS-containing or PFAS-adsorbing sampling equipment and supplies, as previously discussed. A site-specific QAPP or work plan must contain the standard operating procedures incorporating these considerations and client requirements. Refer to Section 11.1.2 for materials to avoid during sampling and drilling. Consult the supplier to determine if PFAS-free options are available.

Pretesting any equipment or supplies to be used is essential. ERBs are recommended to ensure supplies such as bailers, beakers, and dippers are PFAS-free, and that decontamination is effective.

In addition, the sampling team must document any observations during the sampling event that could be a source of bias (for example, the presence of PTFE tape on a faucet).

11.1.7.1 Drinking Water/Non-Drinking Water Supplies

Sampling a “potable water source,” as defined by the USEPA SDWA (Section 1401(4), August 1998), is conducted according to protocols established in the USEPA Methods 537.1 and 533. These protocols define sample bottle preparation, sample collection, field reagent blanks, sample shipment and storage, and sample and extraction holding times. The drinking water source is further defined here as a public drinking water supply, as opposed to a private drinking water supply, as it applies to the USEPA Methods 537.1 and 533. USEPA Methods 537.1 and 533 may also apply in instances when the water quality of the private drinking water supply source is similar to finished drinking water (for example, has low level of total organic content). The following summarizes the sampling considerations described in these protocols:

- For Method 537.1: Sample bottle is a laboratory-provided 250-mL polypropylene bottle fitted with a polypropylene screw cap. For finished (treated) drinking water sampling only, a preservation agent is provided inside each bottle prior to sample collection. This agent acts as a buffer (TRIS pH 7, 5 g/L) and removes free chlorine from chlorine-treated drinking water supplies.
- For Method 533: Sample bottle is a laboratory-provided 100-250-mL polypropylene bottle fitted with a polypropylene screw cap. For finished (treated) drinking water sampling only, a preservation agent is provided inside each bottle prior to sample collection. This agent acts as a buffer (ammonium acetate, 1 g/L) and removes free chlorine from chlorine-treated drinking water supplies.
- The sample handler must avoid PFAS contamination during sampling by thoroughly washing their hands and wearing nitrile gloves.
- Open the tap and flush the water (approximately 3–5 minutes) to obtain a “fresh” sample. Collect the sample while water is flowing, taking care not to flush out preservative. Samples do not need to be headspace-free. Cap the bottle and, if applicable, shake to completely dissolve preservative.
- Keep sample sealed and place sample on ice for shipment.
- Samples must be chilled during shipment and must not exceed 10°C during shipment.
- Laboratory extraction of the sample must take place within 14 days of collection (USEPA Method 537.1) or within 28 days of collection (USEPA Method 533).

Based on a review of industry experience and guidance, additional considerations for collecting drinking water samples for PFAS analysis are as follows.

- The sample should be collected from a tap or spigot located at or near the [glossary_exclude]well-head[/glossary_exclude] or pump house and before the water supply is introduced into any storage tanks or treatment units. If the sample must be collected at a point in the water line beyond a tank, a sufficient volume of water should be purged to provide a complete exchange of fresh water into the tank and the tap or spigot. If the sample is collected from a tap or spigot located just before a storage tank, spigots located downstream of the tank should be turned on to prevent any backflow from the tank to the tap or spigot. Several spigots may be opened to provide for a rapid exchange of water. If collecting a sample to characterize human or other exposure, the sample should be collected from the tap or spigot at the point of use.

- When sampling from a drinking water well that is not in regular use, purge water until water quality parameters (that is, pH, specific conductance, dissolved oxygen, oxidation-reduction potential, turbidity, and temperature) have stabilized, to ensure formation water (as opposed to stagnant well column water) will be sampled. An adequate purge is achieved when the pH and specific conductance of the potable water have stabilized (for example, within 10% across three consecutive measurements) and the turbidity has either stabilized or is below 10 nephelometric turbidity units. Note: According to USEPA (2013), pg. 21 *"[a] well with an intermittently run pump should, in all respects, be treated like a well without a pump. In these cases, parameters are measured and the well is sampled from the pump discharge after parameter conditions have been met. Generally, under these conditions, 15 to 30 minutes will be adequate."*
- When sampling from a tap, the tap must be protected from exterior contamination associated with being too close to a sink bottom or to the ground. Contaminated water or soil from the faucet exterior may enter the bottle during the collection procedure because it is difficult to place a bottle under a low tap without grazing the neck interior against the outside faucet surface. If the tap is obstructed in such a way that prevents direct collection into the appropriate sample container, it is acceptable to use a smaller container to transfer sample to a larger container. The smaller container should be made of HDPE or polypropylene and should be either new or decontaminated as specified in Section 11.1.5. Evaluation of the transfer container is recommended to ensure that it does not introduce a bias.
- When filling any sample container, care should be taken that splashing drops of water from the ground or sink do not enter either the bottle or cap.
- Leaking taps that allow water to discharge from around the valve stem handle and down the outside of the faucet, or taps in which water tends to run up on the outside of the lip, are to be avoided as sampling locations.
- Disconnect any hoses, filters, or aerators attached to the tap before sampling.

Taps where the water flow is not constant should be avoided because temporary fluctuation in line pressure may cause clumps of microbial growth that are lodged in a pipe section or faucet connection to break loose. A smooth flowing water stream at moderate pressure without splashing should be used. The sample should be collected without changing the water flow.

11.1.7.2 Groundwater

The following summarizes considerations for groundwater sampling.

- Non-potable water samples do not require a chemical preservative, unless otherwise required by the cited analytical method(s). Clean laboratory-provided HDPE or polypropylene bottles are recommended; typically, 125-mL to 1-L bottles may be used, but the sample volume may depend on the analytical method used.
- Low-flow sampling is preferred for collection of groundwater samples for PFAS to keep the turbidity of samples to a minimum. See Section 11.2.1.2 for issues associated with elevated levels of suspended solids in aqueous samples.
- Bailers should be used with caution due to the potential for PFAS to accumulate at the air/water interface. If bailers are used, it is important to make sure that at least one well volume is purged to remove static surface conditions.
- No-purge sampling devices can also be used for groundwater sampling, but this may be dependent upon site conditions.
- Groundwater is typically sampled from a well, and therefore additional equipment is required. Purging and sampling equipment is constructed from a variety of materials. As a result, there are more opportunities for contamination of the sample by the sampling equipment. For example, pumps, bailers, and stopcocks can contain O-rings and gaskets that may be Teflon, or another fluoropolymer, that can be changed out. The most inert material (for example, stainless steel, silicone, and HDPE), with respect to known or anticipated contaminants in the well(s), should be used whenever possible. The various types of purging and sampling equipment available for groundwater sampling are described in ASTM International *Standard Guide for Sampling*

Ground-Water Monitoring Wells, D 4448-01 (ASTM 2007) or *Compendium of Superfund Field Operations Methods* (USEPA 1987).

- Do not use dedicated sampling equipment installed in existing wells prior to the PFAS investigation without identifying all materials found within the equipment and reviewing their chemical properties to ensure they are PFAS-free. Pumps can be a source of PFAS contamination due to internal components (for example, bladder pumps that contain Teflon components can be switched out for HDPE). Consult with the equipment vendor to determine if they have PFAS-free alternatives. For circumstances that warrant, such as very deep wells or sites with co-contaminants, samples may be collected in duplicate with and without existing dedicated equipment. If PFAS analyses show that the equipment does not impact results, the equipment may be kept and used long term. However, this determination is dependent upon project-specific requirements and should be allowed by a project manager only with full disclosure to all stakeholders. It may also be acceptable to simply collect an ERB after fully decontaminating equipment containing PFAS components to confirm it does not contribute to groundwater sample concentrations. A site-specific procedure should be outlined in the QAPP or work plan.
- In addition to equipment, ensure tubing or bailer twine are PFAS-free.
- Within the context of sample collection objectives outlined in a site-specific work plan, the sample location in the water column should consider the potential stratification of PFAS in solution and their tendency to accumulate at the air/water interface. In addition to sample location within the water column, consideration should be given to the well construction, screened interval, and site geology to ensure that the well is representative of site conditions and all relevant COCs. For more information on stratification, see Section 5.2.

Samples should not be filtered, because filters may be either a source for contamination (Ahrens et al. 2009; Arp and Goss 2009) or PFAS may be adsorbed to the filter. If filtration is absolutely necessary, it should be performed in the laboratory, using a validated procedure that includes steps to eliminate the bias that can occur due to sorption issues. As an alternative, laboratory-validated procedures may include centrifuging the sample due to potential filter sorption or contamination issues. See Preparation of Aqueous Samples with Particulates/Suspended Solids in Section 11.2.1.2 for more details.

In cases where sampling for co-contaminants requires use of PFAS materials, sampling events should be separated to avoid contamination from these materials. The PFAS sampling event would be completed first, followed by the sampling event for the co-contaminants. In some cases, it may be acceptable to use the same equipment at a concurrent sampling event.

11.1.7.3 Surface Water

Surface water PFAS sampling is conducted in accordance with the traditional methods such as those described in the USEPA's *Compendium of Superfund Field Operations Methods* (USEPA 1987) with the following exceptions and/or additional considerations.

- Within the context of sample collection objectives outlined in a site-specific QAPP or work plan, the sample location in the water column should consider the potential stratification of PFAS in solution and their tendency to accumulate at the air/water interface. For more information on stratification, see Section 5.2. If possible, the transfer container will be lowered sufficiently below the water surface but above the bottom sediments.
- Transfer containers, such as beakers or dippers, which may be attached to extension rods, should be used only if sample containers have preservatives. Sampling by direct sample container immersion is not recommended.

11.1.7.4 Porewater

Similar in many ways to sampling techniques and equipment used in groundwater sampling for PFAS, porewater purging and sampling involves a variety of materials. The various types of purging and sampling equipment available for porewater sampling are described in *Pore Water Sampling Operating Procedure* (USEPA 2013). For PFAS sampling, peristaltic pumps with silicon and HDPE tubing are typically used for porewater sample collection, along with push-point samplers, porewater observation devices (PODs), or drive-point piezometers. Push-point samplers and drive-point piezometers are made of stainless steel, while PODs consist of slotted PVC pipe and silicon tubing. PODs and drive-point piezometers are permanent, or dedicated, sampling points typically installed and used for multiple sampling events, whereas push-point samplers are used as a temporary sampling location. Otherwise, the standard procedure for porewater purging and sampling using a peristaltic pump, as described in the *Compendium of Superfund Field Operations Methods* (USEPA 1987), can be followed.

11.1.7.5 Sediment

Most core and grab sampling devices are constructed of stainless steel. Some core samplers include an HDPE sleeve inserted in the core barrel to retain the sample. Ensure that materials that contact the media to be sampled do not have water-resistant coatings that contain PFAS that are the target of the analysis. Additional PPE may be required for sampling personnel, such as waders and personal flotation devices. Ensure that materials that will potentially contact sampling media do not consist of water-resistant coatings or other PFAS-containing materials or substances. Sample protocols should meet the requirements contained in the analytical methods to be used. Refer to Section 11.1.2 for typical materials used during sampling and drilling.

11.1.7.6 Surface Soil

For surface soil sampling, refer to Section 11.1.2 for equipment and supplies, and Section 11.1.5 for decontamination procedures. Sample protocols should meet the requirements contained in the analytical methods to be used. No additional considerations are recommended for PFAS sampling of surface soil.

11.1.7.7 Subsurface Soil

Sample protocols should meet the requirements contained in the analytical methods to be used. No additional considerations are recommended for PFAS sampling of subsurface soil.

11.1.7.8 Fish

The species of fish collected, as well as the portion of fish sampled (whole versus fillet), depends on the project goals (for example, ecological risk or human health). Studies have shown that the majority of the PFAS in fish are stored in the organs, not the flesh (Martin et al. 2004) (Yamada et al. 2014). Communicating project objectives to the laboratory is important prior to fieldwork to determine the necessary quantity and quality of tissue, fish handling requirements, laboratory sample preparation (including single fish or composite fish samples, and whole or fillet preparation), and packing and shipping requirements. According to USEPA Draft Method 1633, whole fish or other biota samples should be wrapped in aluminum foil or food-grade polyethylene wrap and homogenized tissue samples should be placed in HDPE containers.

11.1.7.9 Air Emissions to Air and Ambient Air

There is an increasing need for the measurement of PFAS in emissions from stationary sources (for example, chemical manufacturing, industrial use, combustion and thermal treatment), as well as in ambient air. Due to the diverse nature of PFAS, multiple measurement approaches are needed to measure polar and nonpolar, volatile, semivolatile, and nonvolatile (particulate-bound) PFAS.

11.1.7.9.1 Emissions to Air

Currently, there are no multilaboratory-validated, published sampling methods for PFAS in air emissions (for example, from thermal treatment in manufacturing plants or incinerators). In their absence, emissions measurements have been performed using modifications to USEPA SW846 Method 0010 (Modified EPA Method 5 Sampling Train) (USEPA 1986), a method designed for measurement of semivolatile organic compounds. Other methods have been adapted to capture specific individual compounds of interest.

USEPA and European groups (Verein Deutscher Ingenieure [VDI], an association of German engineers) are currently evaluating and investigating which sampling and analytical methods might be, in principle, the most suitable to capture PFAS and resulting byproducts in all fractions of the emissions (particles, moisture, gas phase).

PFAS can be partitioned in stack emissions into several different fractions due to the physical properties of these species. At the elevated temperatures typically encountered in stack emissions the vapor pressure can be sufficiently high that some PFAS are present in the gas phase. The lower molecular weight fluorotelomer alcohols (FTOHs) have lower boiling points and so may primarily be present as vapors. PFAS can adsorb to particulate matter, are highly water soluble, and can dissolve in water droplets if present in the stack. To measure these partitioned fractions, the stack effluent is sampled isokinetically (that is, the air enters the probe at the same velocity as it is moving in the stack, to accurately sample particles and droplets) and captured on a heated filter, an XAD-2 sorbent resin tube, and in water impingers. In some test programs a second XAD-2 sorbent cartridge is included in the sample train to determine if breakthrough has occurred. The filter, sorbent cartridge, and water impingers are recovered separately, and the sample train components are rinsed with a methanol/ammonium hydroxide solution.

In 2021, the USEPA released Other Test Method (OTM) 45 *Measurement of Selected Per- and Polyfluorinated Alkyl Substances from Stationary Sources* (USEPA 2021). This performance-based method was made available by USEPA as a recommended method that can be used to measure 50 specific semivolatile and nonvolatile polar PFAS from a variety of stationary sources. USEPA OTM-45 is largely based on the USEPA SW-846 Method 0010 (Modified EPA Method 5 Sampling

Train) with several modifications. PFAS are collected in four sample fractions: 1) filter; 2) primary XAD-2; 3) impingers (containing water); 4) secondary XAD-2 (for breakthrough determination). Each fraction, with its associated rinses, is extracted and analyzed separately. Analyses are performed by isotope dilution LC/MS/MS. The analytical approach is based largely on EPA Method 533 and includes many of the same analytical qualification criteria. USEPA OTM-45 was released as an "Other Test Method (OTM)" by USEPA's Emission Measurements Center to promote consistency and is considered by USEPA to represent the current best practices to sample and analyze PFAS targets from stationary sources. USEPA OTM-45 is a draft method under evaluation that will be updated as more data from stakeholders become available. Field sampling programs must include collection of field blanks as a means of assessing PFAS artifacts present in sampling media and potentially introduced during sample handling in the field. Other QC measures that should be considered include the use of isotopically labeled PFAS field spikes. The latter compounds are typically applied/spiked by the laboratory into the XAD-2 sorbent media prior to field deployment. These compounds serve to assess analyte ("native PFAS in air") collection efficiency, breakthrough, and the accuracy of the combined sample collection and analysis method on a sample-specific basis.

Additional measurement approaches are needed to sample and analyze for other PFAS species, such as volatile and nonpolar PFAS compounds, including PFAS that are specific to chemical manufacturing (for example, hexafluoropropylene oxide or HFPO). For example, some volatile (boiling point $< 100^{\circ}$C) polar PFAS can be sampled by modified USEPA Method 18 *Measurement of Gaseous Organic Compound Emissions by Gas Chromatography* (USEPA 2017), in which the analytes are captured in chilled methanol impingers. The methanol reacts with some PFAS compounds to form an ester which enables the capture and subsequent analysis. Other approaches include evacuated passivated canisters and sorbent traps for the more volatile PFAS species.

The ability to identify compounds not targeted for measurement by existing methods is an important need. Nontargeted analyses are critical to being able to identify these compounds. With nontargeted analyses, chromatography (liquid and gas) is combined with high resolution mass spectrometry and multiple ionization techniques to determine atomic molecular weight and associated fragments. These results can be compared to databases for tentative identification. Further spectral interpretation may result in structural identification. This is particularly useful where no chemical standards exist. USEPA Office of Research and Development (ORD) has used nontargeted analysis to support New Hampshire Department of Environmental Services and the Saint Gobain Performance Plastics emission characterization in Merrimack, New Hampshire. (NH DES 2019). It is anticipated that nontargeted analyses will be an important tool to thermal treatment research and the ability to identify potential products of incomplete combustion (PICs).

USEPA ORD is researching and evaluating multiple PFAS measurement approaches for polar and nonpolar, volatile, semivolatile and nonvolatile PFAS, including further development of USEPA OTM-45 for nonpolar PFAS. USEPA ORD is using evacuated passivated canisters for select targeted volatile PFAS. USEPA ORD is also evaluating the use of Fourier Transform Infrared (FTIR) spectroscopy to measure select volatile PFAS in real-time. Several of the PFAS compounds capable of being measured are also being evaluated as potential indicators or surrogates of PFAS destruction performance.

Multiple PFAS emissions tests have been performed at a variety of sources for multiple purposes including source characterization, assessment of control technology performance, and evaluation of treatment technologies. Stationary source, or stack, emissions of PFAS have been measured in North Carolina (NC DEQ 2019) and New Hampshire (NH DES 2019) from industrial facilities that synthesized PFAS (Chemours, NC) or used PFAS in manufacturing processes (Saint Gobain Performance Plastics, NH) (Beahm and Marts 2019). These test programs confirmed that stack emissions from industrial facilities contribute to ground and surface water contamination (NC DEQ 2019). An additional study at Chemours, NC, reported on the commissioning of the recently installed thermal oxidizer control system (Weston Solutions, Inc. 2020).

According to the USEPA's *Per- and Polyfluoroalkyl Substances (PFAS) Action Plan* (2/19) (USEPA 2019), a method for sampling and analyzing PFAS in factory stack air emissions was anticipated in 2020 and is now expected in 2022 (USEPA 2021). USEPA has been participating in the testing at Saint Gobain Performance Plastics (NH) and Chemours (NC) by either evaluating alternate sampling methods or performing independent analysis of the stack test samples. As stated in the Action Plan pg. 51, USEPA is "*testing and developing additional methods for possible refinement, including methods to quantify PFAS precursors; Total Organic Fluorine for a general PFAS detection method; and refinement of non-targeted high-resolution mass spectrometry approaches for suspect screening and novel PFAS discovery.*"

To date, test reports from ten stack tests conducted at Chemours have been published on the North Carolina Department of Environmental Quality website (NC DEQ 2019). One stack test report from the Saint Gobain Performance Plastics facility has been published on the New Hampshire Department of Environmental Services OneStop Navigation website (NH DES 2019).

These test reports detail the sampling and analysis methodologies used thus far in measuring PFAS stack emissions.

11.1.7.9.2 Ambient Air

There are currently no USEPA Federal Reference Methods (FRM) or Toxic Organic Methods (TO series) available specifically for the measurement of PFAS compounds in ambient air. In their absence, some sampling and analysis of ambient air have been performed using modified TO methods, such as TO-13A and TO-9 (USEPA 2020). Both of these methods use high-volume air samplers fitted with a particulate glass fiber filter/quartz fiber filter (GFF/QFF) and sorbent cartridge for the collection of particulate and gaseous phases, respectively. USEPA TO-13A specifies collection of air samples at a flow rate of approximately 225 liters/minute, resulting in an air volume greater than 300 m^3. The solid sorbent used consists of a “sandwich” of polyurethane foam (PUF) and XAD-2 (polymer of styrene divinyl benzene).

PFAS in ambient air have been measured using both active (with actual flow) and passive (gas diffusion) sampling techniques. The majority of techniques have made use of solid sorbents such as PUF, XAD-2, and sorbent-impregnated PUF (SIP). (Finely ground XAD-4 resin is often the sorbent of choice for impregnating the PUF). Active samples also include a particulate filter (glass or quartz fiber) ahead of the sorbent module. To optimize detection limits, high-volume air samples have been used most often.

Detection limits of air and emissions methods can be greatly influenced by PFAS artifacts found in the neat filter, sorbent media, or components within the sampler itself. For example, use of Teflon gaskets in high-volume samplers is not recommended. Field sampling programs must include collection of field blanks as a means of assessing PFAS artifacts present in sampling media and potentially introduced during sample handling in the field. Other QC measures that should be considered include collection of field duplicate or co-located samples and the use of isotopically labeled PFAS field spikes. The latter compounds are typically applied/spiked by the laboratory into the sorbent media prior to field deployment. These compounds serve to assess analyte (“native PFAS in air”) collection efficiency, breakthrough, and the accuracy of the combined sample collection and analysis method on a sample-specific basis.

Passive samplers should also make use of mass-labeled PFAS as a sample-specific quality control measure to account for native PFAS losses during each sampling event. Volatilization of labeled PFAS during the deployment period provides sampling rates on a site-specific basis and accounts for both temperature and wind influences.

USEPA and European groups (VDI) are currently evaluating and investigating which sampling methods might be, in principle, the most suitable to capture PFAS and resulting byproducts in all fractions of the emissions (particles, moisture, gas phase). An important consideration is that fluorinated polymers are used in common sampling equipment, which may cause contamination of the samples. For the purposes of PFAS determinations, this material must be replaced. In addition to concerns over using fluorinated polymers in sampling equipment being a source of contamination, there are also concerns about the potential for adsorption of PFAS to fluorinated polymers, thereby effectively reducing the observed concentration, or affecting any attempt to quantify the phase distributions between condensed and non-condensed phases (for example. PM vs gaseous). This is related, in part, to the concerns about quantification of ambient air concentrations and distributions, as published by Johansson, Berger, and Cousins (2017), showing that the use of GFF (and chemically deactivated glass fiber filters) may irreversibly bind fluoro-carboxylates during collection of samples containing fluoro-acids, which can be in the gas phase depending on their vapor pressure and ambient temperature, and the corresponding carboxylates. This issue for ambient air appears to negatively impact the estimates of phase distributions and so far, may not have a technique that is applicable for quantitative recovery (no answer to this problem has yet been published). A related issue, though separate from the ambient sampling confounding issues, is that quantification of acids/carboxylates via LC/MS does not/cannot distinguish between these two oxidation states, which is important to the phase distribution in ambient air (and emissions to air). There are other techniques (GC/MS or Chemical Ionization Mass Spectrometry (CIMS) (Riedel et al. 2019) that may help address these LC/MS deficiencies, particularly with regard to air measurements.

11.1.7.10 Human Blood, Serum, Tissue

Currently, there are no official or standardized methods for testing human blood, serum, or tissue. Laboratories and the Centers for Disease Control and Prevention (CDC) are in the process of developing best methods. A procedure developed by the CDC’s National Center for Environmental Health has been published (CDC 2016). There are also several laboratories advertising this capability; however, the analytical methods and modifications from validated environmental laboratory protocols may not be consistent among these vendors. Human testing is outside the scope of this document; however reference points that could be used for comparison of whole blood or serum results to geometric mean serum levels generated from the U.S. population are included in the ATSDR ToxGuide for Perfluoroalkyls (ATSDR 2020).

11.1.7.11 Potential High Concentration Samples

The CSM or previous sampling may indicate areas of high concentrations of PFAS for which single-use, disposable equipment is recommended. If single-use is not possible, take additional precautions such as implementing a greater frequency of ERBs and not reusing equipment to sample potentially low PFAS concentration samples. High concentration samples should be segregated during shipping to the laboratory, and clearly identified on the sample chain-of-custody form.

Some projects may require the analysis of AFFF product that has been used at the site. All AFFF product samples must be considered high concentration samples. The method DoD AFFF01 contains steps to prepare AFFF concentrate samples. This method requires that a dilution of a subsample of the AFFF concentrate be prepared for extraction. A critical step in this procedure is the amount of time that must elapse, a minimum of 3 hours (Willey 2021) from the time PFAS-free reagent water is added to the aliquot of AFFF concentrate to create the dilution to the time when extracted internal standard can be added to the diluted sample. It was determined during method development and validation that some AFFF concentrates can take up to 3 hours to fully dissolve in the reagent water. In addition, this method requires each AFFF concentrate sample to be prepared and analyzed in duplicate, using another aliquot of the collected sample. It is recommended that these samples be segregated from other samples during sampling and shipping to avoid cross-contamination. AFFF concentrate samples should be segregated during shipping to the laboratory, and clearly identified on the sample chain-of-custody form.

11.2 Analytical Methods/Techniques

11.2.1 Quantitative Techniques

11.2.1.1 General

Analytical methods are still evolving for PFAS analysis. Currently, very few methods are multi-laboratory validated and published. Two multi-laboratory-validated methods, USEPA Methods 537.1 and 533 have been published for analysis of drinking water samples (USEPA 2020; USEPA 2019) and one, USEPA SW-846 Method 8327, has been published for analysis of groundwater, surface water, and wastewater samples. The DoD has published a multi-laboratory validated method, DoD AFFF01 (Willey 2021), for the analysis of AFFF concentrates to demonstrate compliance to the military specification for AFFF (MIL-PRF-24385). The USEPA has also published a single-laboratory validated method, USEPA Draft Method 1633, which they recommend for use for analysis of wastewater, groundwater, surface water, landfill leachates, soils, sediments, biosolids, and tissue. The USEPA released OTM 45 for measurement of PFAS in emissions to air from stationary sources. In April 2022, USEPA released the Draft Method 1621, Screening Method for the Determination of Adsorbable Organic Fluorine (AOF) in Aqueous Matrices by Combustion Ion Chromatography (USEPA 2022).

- USEPA Method 537.1 tests for 18 PFAS analytes (including 4 PFAS not included in USEPA Method 533)
- USEPA Method 533 tests for 25 PFAS analytes (including 11 not included in USEPA Method 537.1)
- USEPA SW-846 Method 8327 tests for 24 PFAS analytes (does not include all PFAS included in USEPA Method 537.1 or 533)
- USEPA Draft Method 1633 tests for 40 PFAS analytes (includes all PFAS included in USEPA Method 537.1, 533, and 8327 and 8 additional PFAS analytes)
- DoD AFFF01 tests for PFOA and PFOS
- USEPA Other Test Method (OTM) 45 for 50 specific semivolatile and nonvolatile polar PFAS

Other methods have been published by other organizations. Lists of these methods, by various categories, are provided in the PFAS Analytical Methods Excel spreadsheet in the **separate tabs**:

- External **Table 11-2**–Published Method Basics. Provides information on basic principles of each method (media type, validation status, method type, sample container requirements, holding time, preservation requirements, and analytical instrument.
- External **Table 11-3**–Published Methods Specifics. Provides more details of the methods such as sample preparation requirements, quantitation scheme, confirmation requirements, quantitation limits, and isomer profile.
- External **Table 11-4**–Analyte Lists. The method analyte list for each of these methods varies.
- External **Table 11-5**–Draft Published PFAS-Related Methods Basics. Provides information on basic principles of each method (media type, validation status, method type, sample container requirements, holding time, preservation requirements, and analytical instrument).

11.2.1.2 Sample Preparation

USEPA Method 537.1 and 533, USEPA Draft Method 1633, and DoD AFFF01 all require aqueous samples to be prepared using the solid-phase extraction (SPE) technique. USEPA Draft Method 1633 and DoD AFFF01 both require cleanup procedures be used on extracts and all associated batch QC samples to help eliminate matrix interferences (for example, bile salts, gasoline range organics) that could be present. USEPA Method 537.1 and 533, USEPA Draft Method 1633 and DoD AFFF01 all require extraction of the entire sample collected (in the case of DoD AFFF01, the entire prepared dilution), including a rinse of the sample container (in the case of DoD AFFF01, the dilution container).

USEPA SW-846 Method 3512 prepares groundwater, surface water, and wastewater samples by diluting the sample collected with an equal volume of methanol and does not require a rinse of the sample container to be included unless the sample had to be transferred to a larger container in order to allow for the addition of the appropriate volume of methanol. USEPA SW-846 Method 3512 does not use SPE or carbon clean-up steps, which is another significant difference from the other USEPA published methods. USEPA SW-846 Method 8327 states that during method development, some PFAS analytes showed a potential for loss during sample preparation or standard preparation using USEPA SW-846 Method 3512. This method states that a minimum organic cosolvent content must be maintained in standards and samples, and it cautions against aqueous subsampling prior to adding sufficient organic solvent.

USEPA Draft Method 1633 prepares groundwater, wastewater, surface water, and landfill leachates using solid phase extraction (SPE) followed by carbon clean-up steps to eliminate matrix interferences. USEPA Draft Method 1633 uses solvent extraction for soil, sediment, biosolids, and tissue preparation, followed by use of SPE cartridges and carbon clean-up steps to eliminate matrix interferences. Regardless of the method used, care must be taken to prevent sample contamination during preparation and extraction because the limits of quantitation and detection are 1,000 times below (ppt) those for more routine analyses such as volatiles or semivolatile analysis (ppb). It is recommended that all supplies be checked and confirmed as PFAS-free prior to sample preparation. Intermittent contamination can occur due to vendor or manufacturing changes.

Some PFAS analytical methods, such as USEPA Method 533 and USEPA Draft Method 1633, use isotope dilution and extracted internal standard quantification schemes to calculate sample concentrations. Isotope dilution analysis (IDA) quantitation requires the use of extracted internal standards (EIS) that are the isotopically labeled analogs of the method analytes. Method analytes for which corresponding isotopically labeled analogs are not commercially available are quantitated using the isotopically labeled analogs of a method analyte of similar chemical properties. Since isotopically labeled analogs of PFOA and PFOS are commercially available, DoD AFFF01 uses isotope dilution quantitation. USEPA SW-846 Method 8327 uses an external quantitation scheme to calculate the percent recovery of isotopically labeled analogs that are added to the sample prior to dilution with methanol. This method does not use isotopically labeled analog recoveries to account for sample preparation and matrix interference biases in the sample result. All of the methods discussed above require these isotopically labeled standards be added to the sample at a designated point in sample preparation, depending on the sample matrix:

- aqueous samples–added to field samples while in the original container prior to extraction/dilution
- AFFF concentrates or high concentration aqueous samples – added to dilution of sample prior to extraction
- solid samples and biota–added after homogenization and subsampling, prior to addition of water or extraction solvent prior to extraction

Ensuring a representative sample/subsample for analysis is critical. For aqueous samples, the entire sample and rinsate of the sample container received by the laboratory must be extracted by SPE to recover any PFAS that adhere to the sample container. Filtration is not recommended for samples with high particulate content because retention of PFAS onto filters has been noted. Centrifugation is often used to reduce or separate out sample particulates. See Preparation of Aqueous Samples with Particulates/Suspended Solids below for more details.

USEPA Draft Method 1633 requires the screening of all aqueous samples using a separate sample container from the one which will be used for sample preparation. Due to limitations in SPE cartridge capacity and potential contamination of sample preparation and/or analytical equipment, the method requires samples containing high concentrations of PFAS (for example, AFFF concentrates) to be diluted prior to SPE and sample clean-up. In these cases, adsorption onto the original sample container is not an issue, depending on the identified project-specific DQOs, because the amount of PFAS adsorbed onto the container walls is negligible compared to the amount of PFAS in the sample.

USEPA Draft Method 1633 requires the entire soil, sediment, and biosolid, sample that is collected be homogenized in the laboratory prior to subsampling. Tissue samples are to be prepared according to project requirements (for example whole fish versus fillet) and homogenized prior to subsampling.

Cleanup procedures can be used on extracts and all associated batch QC samples (for example, method blank and laboratory control samples) when matrix interferences (for example, bile salts, gasoline range organics) could be present. USEPA Draft Method 1633 and DoD AFFF01 require carbon cleanup techniques to be used for all sample matrices. USEPA Draft Method 1633 and DoD AFFF01 require samples to be slowly concentrated to remove methanol from the extract to avoid loss of neutral and other highly volatile method analytes. In addition, USEPA Draft Method 1633 states that if methanol is not sufficiently removed, its presence during SPE can result in poor recovery of long-chain carboxylic acids and sulfonates. Care must be taken to avoid these outcomes.

The preparation batch QC samples that are required by these PFAS methods varies. Common laboratory QC samples included are as follows:

- Method blank – (one per preparation batch of 20 or fewer field samples) PFAS are ubiquitous and found in many analytical instrument systems, reagents, containers, and common laboratory environments. The method blank is the same media as associated field samples and undergoes the same sample preparation procedure as the associated field samples. It is a vital indicator for the analysis. Note that method blank is also referred to as Laboratory Reagent Blank in EPA drinking water methods.
- Sample duplicate – (minimum of one per preparation batch of 20 or fewer field samples) An AFFF concentrate sample that undergoes dilution prior to SPE is prepared and analyzed in duplicate in a single laboratory to ensure the laboratory’s subsampling procedures are capable of achieving a known level of precision as defined in the QAPP or work plan.
- Laboratory Control Sample (LCS) or Ongoing Precision and Recovery (OPR) – (one per preparation batch of 20 field samples) Must contain all project-specific PFAS in the same media as associated field samples and is used to evaluate bias associated with sample preparation as well as analytical processes.
- Low-Level Laboratory Control Sample (LLLCS) or Low-Level Ongoing Precision and Recovery (LLOPR) – (one per preparation batch of 20 field samples) Must contain all project-specific PFAS at a specified concentration (for example, 0.5 to 2 times the LOQ for USEPA SW-846 Method 8327 or 2 times the LOQ for USEPA Draft Method 1633) in the same media as associated field samples and is used to evaluate bias associated with sample preparation as well as analytical processes.
- Certified reference material (CRM) – (if available, one per preparation batch of 20 or fewer field samples) Unlike LCSs, which contain no matrix interferences, CRMs can be of significant value when dealing with complex matrices such as soil, sediment, and tissue.
- Matrix spike (MS) and MS duplicate (MSD) – (one pair per prep batch of 20 or fewer field samples) An MS/MSD QC pair is not needed if IDA can be used for all PFAS of interest because the EIS recoveries account for the influence of matrix interferences in each sample, not just 1 in 20. If EIS standards are not available for a PFAS of interest, an MS/MSD QC pair may be warranted to assess the effects of matrix interference on that specific PFAS.

For samples with high concentrations of PFAS, it is recommended that an LCS duplicate and a sample duplicate are prepared in lieu of an MS and MSD. DoD AFFF01 requires the sample duplicate to be prepared using a different aliquot from the same sample bottle to create the sample duplicate.

Preparation of Aqueous Samples with Particulates/Suspended Solids

USEPA Draft Method 1633 contains a procedure for determining the solids content of aqueous samples. This determination is to be performed using the second container that is required for each sample, not the sample container used to prepare the sample for the analysis for PFAS. The sample preparation procedures for PFAS analysis in USEPA Draft Method 1633 are applicable to aqueous samples containing less than 50 mg of solids. The method provides additional steps to take if particulates clog the SPE cartridge during extraction, including the use of additional SPE cartridges. Solids accumulate high concentrations of PFAS and specifically some of the longer-chain PFAS. Care should be taken to resuspend any solids and to rinse bottles so that measured concentrations consider the entire sample. When these solids settle out in the water samples, concentrations can decrease by an order of magnitude or more.

When samples contain greater than or equal to 50 mg of solid, depending on the laboratory procedure, the resulting concentrations could be different and the reported PFAS compounds (fingerprint) could be different. Ultimately, the data user needs to work with the laboratory to determine the appropriate procedure to be performed, depending on the end use of the data (for example, remedial action, human or ecological risk assessment, permit compliance, etc.). The project objectives may also vary depending on whether the sample matrix is wastewater, surface water, stormwater, or groundwater.

A survey conducted in 2020 by ITRC received responses from 16 laboratories. These responses demonstrated that the laboratory community is *not* consistently preparing aqueous samples in the presence of suspended solids.

- Several laboratories centrifuge the samples and decant off the water portion for subsequent extraction.
- Some laboratories may also extract the remaining particulates and combine the extract with the aqueous extract. However, only one of the surveyed laboratories does this routinely; the others do this only if requested by the client.
- Routine laboratory procedures are not dependent upon whether the matrix is groundwater, surface water, or wastewater.
- Several laboratories filter the samples, if requested by the client. This may result in adsorption of PFAS from the samples and potential cross-contamination, and should be implemented with care.
- After decanting, some laboratories rinse the remaining particulates in the bottle with solvent for the SPE.
- Not all laboratories disclose when samples require special preparation (decanting, centrifuging, or filtering) due to the presence of particulates. Therefore, this would not always be known to the data user.
- Laboratories have different procedures for when particulates clog the SPE cartridge; some labs may re-extract at a dilution, while others may start a new cartridge and attempt to continue with the extraction of the remaining sample.

Laboratories should clearly state whether reported concentrations are dissolved (water only) or “total” PFAS measurements (including sample particulates). Laboratories that are not including the extraction of the particulates (water only) are providing a dissolved PFAS measurement, rather than an overall or “total” PFAS measurement of these aqueous samples that would include the PFAS content of the particulate. Project objectives will determine whether dissolved or total PFAS measurements are appropriate. Sampling procedures should be designed to gather representative samples to meet project objectives. Some issues to consider in the determination of the need for a total or dissolved PFAS measurement are as follows:

Groundwater

- If minimizing presence of particulates is within the project objectives, use of low-flow groundwater sampling or no-purge samplers should be considered.
- If turbidity is >10 NTU, consider a “total” measurement if sampling groundwater for compliance, delineation, remedial design, or risk assessment. “Total” can be defined as centrifuge, decant, and extract both phases, to report the dissolved concentration and the suspended/solid concentrations either individually or summed in the report.
- Collect samples for TSS analysis to assist in the evaluation of sample data. In lieu of TSS, turbidity measurements can also be helpful in the evaluation.

Drinking Water

- “Total” PFAS measurements are always required under the SDWA. However, drinking water samples rarely have issues with suspended solids.

Surface Water and Wastewater

- Use a “total” measurement if sampling surface water or wastewater for compliance, permitting, remedial design, or risk assessment.
- A “total” measurement may not be required if sampling for a line of evidence in a source area, rather than for a regulation.
- Collect samples for TSS analysis to assist in the evaluation of sample data. In lieu of TSS, turbidity measurements can also be helpful in the evaluation.

For laboratories performing centrifugation and subsequent decanting, it should be noted that it is important that the extracted internal standards are spiked into the aqueous samples prior to centrifugation. It is important to communicate with your laboratory prior to the collection of samples on the approach that will be used.

11.2.1.3 Sample Analysis

Instrument Type–LC/MS/MS

Currently all methods published by the USEPA and DOD use liquid chromatography-mass spectrometry-mass spectrometry (LC/MS/MS), which is especially suited for analysis of ionic compounds, such as the PFSAs and PFCAs. Gas chromatography-mass spectrometry (GC/MS) can also be used for PFAS analysis, specifically the neutral and nonionic analytes, such as the fluorotelomer alcohols (FTOHs), perfluoroalkane sulfonamides, and perfluoroalkane sulfonamido ethanols. GC/MS may be appropriate for (ambient) atmospheric samples. PFAS are either directly detected using large-volume injection (Scott et al. 2006) or detected as a derivatized compound that is GC/MS amenable (Langlois et al. 2007). At this time, there is no published GC/MS method and very limited commercial availability for the technique for PFAS analysis.

In contrast, LC/MS/MS analysis of PFAS is widely available. LC/MS/MS operated in multiple reaction monitoring (MRM) mode offers a unique fragment ion that is monitored and quantified from a complex matrix. MRM is performed by specifying the mass-to-charge ratio of the compound of interest for fragmentation within the MS/MS. This is facilitated by specifying the parent mass of the compound of interest for MS/MS fragmentation and then monitoring only for product ions. Ions arising from that fragmentation are monitored for by the MS/MS, which yields improved specificity and sensitivity.

Standards Preparation and Storage

Certified analytical standards are available from several manufacturers. Products may have variable purity and isomer profiles, which may compromise the accuracy, precision, and reproducibility of data. Certified standards of the highest purity available, for example, American Chemical Society grade, are quantitative standards that can be used for accurate quantitation. Quantitative standards containing linear and branched isomers are not commercially available for all applicable analytes. Currently, quantitative standards are available only for PFOS, perfluorohexane sulfonic acid (PFHxS), 2-(N-methylperfluorooctanesulfonamido) acetic acid (NMeFOSAA), and 2-(N-ethylperfluorooctanesulfonamido) acetic acid (N-EtFOSAA). Technical grade standards, or qualitative standards, that contain branched and linear isomers are available for other PFAS, but these standards do not have the accuracy needed for quantitation purposes. Currently, qualitative standards that contain branched and linear isomers are available for PFOA, PFNA, PFOSA, NMeFOSE, NEtFOSE, NMeFOSA. USEPA Draft Method 1633 and DoD AFFF01 both require the analysis of technical grade standards to verify which peaks represent the branched isomers for method analytes when these standards are available. Stock standards of PFAS analytes, internal standards, and surrogate standards supplied in glass ampoules by the manufacturer are acceptable. Manufacturers of the certified analytical standards often provide laboratories storage and shelf-life guidance for stock and working standards. USEPA Methods 533, 537.1, 8327, Draft Method 1633, OTM-45, and DoD AFFF01 contain storage requirements for both reagents and/or standards.

Steps to Help Eliminate Laboratory/Instrument Contamination (Verification of Supplies, Instrument Blanks, Isolator Columns)

Laboratory and instrument contamination is of particular concern for PFAS, given that the limits of detection are in the ppt range. Additionally, nonpolymer PFAS may be found at trace levels as impurities in some polymer products (3M 1999). PFAS are found in commonly used laboratory items such as PTFE products, solvent lines, and methanol, which could lead to method interferences and elevated baselines in chromatograms if not checked. The evaluation criteria for equipment and standards that is applicable depends on the analytical method used. For instance, USEPA Method 537.1 recommends that all of the above items must be “less than 1/3 the MRL (minimum reporting limit) for each method analyte under the conditions of analysis by analyzing laboratory reagent blanks.” (USEPA 2020, pg. 7). USEPA Method 533 further specifies that the isotopically labeled analogs of method analytes and isotope performance standards meet this same requirement (USEPA 2019, pg. 7). The liquid chromatograph can be fitted with an isolator column to separate contamination arising from the solvent delivery system, which allows for quantitation at low detection limits. Guard columns should be used to protect analytical columns.

Ion Transition Selection (Recommended Transitions for Primary and Confirmation Ions, Including Ratio Criteria)

Quantification by LC/MS/MS may be accomplished using a variety of techniques. For relatively simple matrices such as drinking water, USEPA Method 537.1 quantifies analytes by comparing the product ion of one precursor ion and retention time in samples to calibration standards. For more complex matrices, additional product ions and their ion ratios can be used to distinguish analytes from matrix interference. In an MS/MS system, most analytes can be fractured into more than one ion. By monitoring the area of each ion and comparing the ratio of those area counts, a more definitive identification can be made. This identification allows the analyst to distinguish true target analytes from false positives. This more detailed quantification is not required for drinking water matrices, but it is useful for more complex matrices. USEPA SW-846 Method 8327, USEPA Draft Method 1633, and DoD AFFF01 all require two ion transitions from parent to characteristic product ions be monitored and documented for each analyte, with the exception of those analytes without a suitable secondary transition (for example, not detectable or has an inadequate signal-to-noise (S/N) ratio such as PFBA, PFPeA, NMeFOSE, NEtFOSE, PFMPA, and PFMBA). These methods determine ion transition ratio criteria based on information obtained from analysis of standards and use the ratio to detect potential bias in sample results.

Mass Calibration, Calibration Criteria, and Calibration Verification

All LC/MS/MS instruments require mass calibration prior to initial calibration. Mass calibration and mass calibration verification should be performed at setup, after performing maintenance that is required to maintain instrument sensitivity and stability performance, and as needed based on QC indicators, such as calibration verifications, as required by USEPA Draft Method 1633. Mass calibration should be performed according to the manufacturer’s instructions. If manufacturer’s instructions are not available, USEPA Draft Method 1633 provides an alternative procedure for mass calibration and mass calibration verification. Regardless of the procedures used, this method requires the mass calibration and mass calibration verification ranges to encompass the ion masses monitored by the method.

Following mass calibration and mass calibration verification, an initial calibration should be performed and verified. All of the published USEPA methods and the DoD methods contain requirements for instrument calibration and calibration verification specific to the PFAS concentrations expected in the media for which the method is applicable. USEPA Method 537.1 uses internal standard quantitation, while USEPA Method 533 and USEPA Draft Method 1633 use isotope dilution and extracted internal standard quantitation. USEPA SW-846 Method 8327 uses external calibration quantitation. Isotope dilution quantitation is recommended for complex matrices. The instrument is required to be calibrated at setup and as needed following QC failures such as initial calibration verification (ICV) or continuing calibration verification (CCV) failure. The lowest calibration point should be a concentration at or below the Minimum Reporting Limit (MRL), Minimum Level (ML), or Limit of Quantitation (LOQ), depending on the method. Most methods require the analysis of a standard at the MRL, ML, or LOQ concentration at least daily to document the instrument’s ability to accurately quantitate down to that concentration. In addition, some methods also require a reporting limit check QC sample (for example, LLLCS or LLOQ verification sample) to be included with each sample preparation batch to demonstrate adequate quantitation at the lowest concentration is achievable using the sample preparation techniques required by the method.

Some methods, such as USEPA SW-846 Method 8327, require analysis of an ICV, which is a calibration verification standard prepared from a source separate from the calibration standards and analyzed after each initial calibration and before sample analyses are performed. The minimum frequency for ICV is after each initial calibration, prior to sample analyses. CCVs or CVs should be analyzed at the frequency specified in the analytical method. Most USEPA methods require at a minimum CCV/CV to be analyzed prior to sample analysis on days an ICV is not analyzed, after every 10 field samples, and at the end of the analytical sequence. Some methods rotate the concentration of CCVs/CVs to cover the entire calibrated range of the instrument and vary the acceptance criteria depending on the concentration. For example, in USEPA Method 537.1, the calibration acceptance criteria for each analyte are that the lowest calibration point must be within 50–150% of its true value while the other calibration points must be within 70–130% of the true values.

Isotope dilution and extracted internal standard quantitation can correct bias resulting from loss during sample preparation, such as in USEPA Methods 533 and Draft Method 1633. In the case of USEPA SW-846 Method 8327, isotopically labeled analogs are used as surrogates to monitor for loss without being used for recovery-correction of target analyte concentrations. Isotope dilution is a quantitation technique that considers sample matrix effects on each individual PFAS quantitation in the most precise manner possible. This technique quantifies an analyte of interest against the isotopically labeled analog of that analyte, which is added to the sample both prior to and after the onset of sample preparation. EIS quantitation is the comparable technique used when an isotopically labeled analog of an analyte is not commercially available. Addition of EIS prior to sample preparation helps account for loss of analyte during the preparation process and for bias associated with the instrumentation. Calibration criteria for methods using isotope dilution and EIS quantification schemes can be found USEPA Methods 533 and Draft Method 1633. Methods using isotope dilution should include isotopically labeled analog recovery for each sample and analyte in data reports. Isotopically labeled analog recoveries should be reported, and minimum/maximum isotopically labeled analog recoveries may be required by specific analytical procedures. For instance, USEPA Draft 1633 determines the recovery of these analogs through comparison to the response of analogs typically called non-extracted internal standards (NIS) added to the sample after extraction, prior to analysis.

Depending on project DQOs, low isotope recovery may indicate that quantitation was inadequate; the data are then reported as estimated values or not at all.

Instrument Blanks: When Are They Needed, Criteria, and Corrective Actions to Take

Due to the extensive use of PFAS, instrument blanks are critical in determining if the instrument is potentially affecting PFAS concentrations in samples. Some methods, such as USEPA Draft Method 1633, require instrument blanks to be analyzed following the highest calibration standard analyzed, daily prior to sample analysis, after each CCV/CV, and following samples that exceed the calibration range to ensure carryover does not occur. The acceptance criteria for instrument blanks are dependent on the method. For example, USEPA SW-846 Method 8327 requires the concentration of each analyte to be ≤ ½ the LLOQ or < 10% of the sample concentration. USEPA Draft Method 1633 is the only method which states that if instrument blanks indicate contamination following the highest calibration standard, corrective action, such as calibrating with a lower concentration for the highest standard, must be taken and successful analysis of an instrument blank following the highest standard analyzed determines the highest concentration for which carryover does not occur.

Matrix-Specific Information

Drinking Water

Interferences related to the matrix can be caused by the co-extraction of contaminants from the sample. These matrix interferences can have considerable variation from sample to sample. For example, there are problems associated with free chlorine in chlorinated finished waters and using TRIS (Trizma; for USEPA Method 537.1) or ammonium acetate (for USEPA Method 533) can help overcome some of these issues.

Tissue

Interferences related to the matrix can be caused by the co-extraction of bile salts (for example, taurodeoxycholic acid, taurochenodeoxycholic acid, and tauroursodeoxycholic acid) with PFOS from the tissue sample. These bile salts can vary considerably from sample to sample and by species. The use of carbon clean-up steps, such as those required by USEPA Draft Method 1633, helps eliminate these interferences in most extracts; however, when excessive amounts are present in the extract, these steps may only reduce the amount of these bile salts. To address the potential interference, USEPA Draft Method 1633 requires that the chromatographic conditions are adjusted such that these bile salts elute at a retention time beyond 1 minute from the retention time window of PFOS.

11.2.2 Qualitative Techniques

Several techniques employing indirect measurement have been developed that can more comprehensively assess the range of PFAS contamination at a site. These qualitative techniques are not yet standardized through a published USEPA method and range in commercial availability. To date, these techniques have not undergone multilaboratory validation. Data from these qualitative techniques may augment the definitive data from quantitative methods.

11.2.2.1 Overview of Qualitative Techniques

Because of the large number of PFAS and their varied structural characteristics, a single targeted method on either LC/MS/MS or GC-MS/MS is unable to quantify all PFAS that may be present in a sample. When the release source is well understood and the types of PFAS present are both known and amenable to regular PFAS analysis methods (for example, LC/MS/MS of ionic PFAS or GC-MS/MS analysis of neutral PFAS), a targeted analytical approach may be sufficient to adequately characterize a release. For releases that are not well understood or consist of multiple sources, alternative ways of measuring PFAS in a more comprehensive but less targeted fashion may be desirable. Additionally, PFAS that are in polymeric form, such as those used in coatings for paper and textiles, are not amenable to LC- and GC-based separation techniques; they may also not be effectively extracted, even with rigorous methods.

Five primary techniques have been developed to characterize unknown PFAS in a sample. These techniques are not multilaboratory validated or promulgated by USEPA. They are described in more detail in the sections below.

- The total oxidizable precursor (TOP) assay measures PFAA precursors or polyfluorinated compounds that can be converted to PFAAs.
- Particle-induced gamma-ray emission (PIGE) spectroscopy measures elemental fluorine isolated on a thin surface.
- Adsorbable organic fluorine (AOF) or extractable organic fluorine (EOF), paired with combustion ion

chromatography (CIC), measures the organofluorine content of a sample as fluoride on an ion chromatograph.

- High-resolution mass spectrometry techniques, such as quadrupole time-of-flight (qTOF) MS/MS, can tentatively identify PFAS structures through library matching or in-depth data analysis.
- Chemical Ionization Mass Spectrometry (CIMS) technique detects gas-phase PFAS, particularly fluorotelomer alcohols (FTOHs) and other oxygenated PFAS.

11.2.2.2 TOP Assay

Technique Description

The TOP assay (or TOPA) converts PFAA precursor compounds to PFAAs through an oxidative digestion. The increase in PFAAs measured after the TOP assay, relative to before, is an estimate of the total concentration of PFAA precursors present in a sample, because not all PFAS present will be subject to quantitation or oxidation, and some will remain as undetected PFAS. The PFAAs generated have perfluoroalkyl chain lengths equal to, or shorter than, the perfluoroalkyl chain lengths present in the precursors (Houtz et al. 2013; Houtz and Sedlak 2012; Weber et al. 2017; Dauchy et al. 2017).

The TOP assay is a technique developed to estimate oxidizable precursors that can transform to perfluoroalkyl acids (PFAAs) end products that are included in the target analyte list (Houtz et al. 2013; Houtz and Sedlak 2012). A sample is analyzed using conventional LC/MS/MS to determine the baseline levels of PFAAs present in the sample. A separate aliquot of the sample is then exposed to a highly basic persulfate solution and then placed in a sealed container at an elevated temperature (for example, 85°C, in a water bath or other heating device) to thermolyze persulfate into a sulfate radical. At elevated pH, the sulfate radical is scavenged by hydroxide and forms a hydroxyl radical, which then converts the free PFAA precursor compounds to PFAAs. The predominant products (for example, > 95% in control experiments) of the precursors are the perfluoroalkyl carboxylates, whether or not the precursors contain sulfonamido or telomer functionalities. After sufficient time has elapsed to convert all the persulfate, the samples are removed from the heated environment (for example, a water bath), cooled to room temperature, and neutralized prior to LC/MS/MS analysis. The increased concentrations of PFAAs generated after the oxidation step provide an estimate of the concentration of oxidizable PFAA precursors.

The technique can be applied to aqueous (Houtz et al. 2013; Houtz and Sedlak 2012; Houtz et al. 2016; Weber et al. 2017; Dauchy et al. 2017) and solid samples (Houtz et al. 2013). In most cases, samples need to be pretreated prior to oxidation to remove competitive organic compounds. For aqueous samples, dilution may be sufficient, although extraction techniques may be used to further remove matrix effects. Soil samples are extracted prior to persulfate treatment, and the extracts are cleaned with ENVI-Carb prior to treating the evaporated extract. The specific extraction procedure used may affect which PFAA precursors are retained for oxidation. For example, acidic extraction procedures may be required to remove cationic precursor compounds from soils (Barzen-Hanson 2017; Mejia-Avendaño et al. 2017).

Possible Technique Uses

The TOP assay may be used to estimate a total concentration of free PFAA precursors in a sample. When measuring strictly a sample post-TOP assay, the total concentration of PFAS measured may be thought of as a conservative measurement of the sample’s total PFAS concentration. In some cases, oxidation can be incomplete (Ventia 2019). The total PFAA precursor or total PFAS concentration is considered conservative for the reasons explained below in Technique Limitations. Because the method depends on a compound containing a perfluoroalkyl group, it is highly specific to PFAS. The chain lengths of the PFAAs generated after oxidation provide an indication of whether the precursors are predominantly short- or long-chained, although the production of a particular Cn (where “n” signifies the number of carbons in the alkyl chain) PFAA is not equivalent to the concentration of PFAA precursors containing the same chain length. However, if significant amounts of PFOA are generated after oxidation, that is an indication that the sample contains a comparable concentration of C8 or longer PFAA precursor compounds.

The TOP assay is the most widely commercially available of the qualitative techniques and is typically accepted as a means of determining PFAS load on remediation substances to estimate the replacement cycle, but not for site characterization.

Technique Limitations

As mentioned above, the TOP assay does not differentiate between precursors that contain telomer or sulfonamide functionalities, because all these precursors are chemically oxidized primarily to perfluoroalkyl carboxylates. This is significant because a precursor that would likely form PFOS in the presence of a mixed consortium of aerobic bacteria will convert to PFOA under the conditions of TOP assay. The production of branched perfluoroalkyl carboxylates could be attributed to precursors derived from an ECF-based manufacturing process, but environmental samples may not contain the same distribution of branched and linear isomers as was originally generated from the ECF manufacturing process.

The TOP assay results in a mixture of PFCA products from the conversion of fluorotelomer-based compounds (Houtz and Sedlak 2012). For example, 8:2 FTS was converted to 3% PFNA, 21% PFOA, 27% PFHpA, 19% PFHxA, 12% PFPeA, and 11% PFBA in control experiments. Two limitations of the technique arise because of this effect. First, the production of PFOA, for example, is not equivalent to the C8 precursor concentration, because PFOA can be generated from longer-chain telomer compounds and is only a partial product of C8 telomer products. Second, some shorter-chain PFCA products of telomer compounds are not captured. Only 73% of 6:2 FTS was recovered as PFCA products PFBA and longer in control experiments (Houtz and Sedlak 2012). As a result, the TOP assay may under-quantify short-chain PFAA precursors that are telomer-based. Sulfonamido compounds in control experiments did not exhibit a distribution of products; the Cn precursor forms the Cn PFCA in a 1:1 molar ratio.

Some studies have been published on the effectiveness of the oxidative process of the TOP assay on large molecular weight polymer compounds or newer ether-linked PFAS such as ADONA (Zhang et al. 2019). Because PFAS polymers have shown limited ability to biodegrade (Russell et al. 2008; Russell et al. 2010; Washington et al. 2009) relative to low molecular weight free PFAA precursor compounds (Wang, Szostek, Buck, et al. 2005; Lee, D'eon, and Mabury 2010; Wang et al. 2011; Harding-Marjanovic et al. 2015), the TOP assay may be similarly ineffective at converting PFAS polymers to free PFAAs. The TOP assay cannot be used to measure large molecular weight polymeric PFAS unless they are proven to break down in the assay.

For many samples, the TOP assay requires adjustments in dilution, sample preparation, or reagent dosing to achieve complete conversion of PFAA precursors. Standardized quality guidelines are needed to ensure that TOP assay data reflect full conversion of PFAA precursors.

11.2.2.3 PIGE

PIGE is a nondestructive analytical technique that takes advantage of the unique gamma-ray wavelength emission of fluorine when impacted with a proton ion beam. The technique is not compound-specific, but is able to assess total fluorine content of a variety of materials isolated on a thin surface (0.22 mm) (Ritter et al. 2017). The sample is secured in the instrument and bombarded ex vacuo under a 3.4 MeV beam with an intensity of 10 nA for approximately 180 s. Two gamma rays characteristic of the decay of the 19F nucleus (110 keV and 197 keV) are measured and the responses integrated. Quantification is achieved with comparison to fluorine-based calibration standards.

In the published literature, PIGE has been used to demonstrate total organofluorine concentrations in papers and textiles (Ritter et al. 2017; Robel et al. 2017) and in food packaging (Schaider et al. 2017). It has also been used on an experimental basis to evaluate organofluorine concentrations in extracted water and soils, but those results are not yet available in the peer-reviewed literature.

Possible Technique Uses

PIGE is a rapid screening technique to measure fluorine on surfaces. If a sample does not contain significant amounts of fluoride or can be prepared to remove inorganic fluoride, PIGE can become a technique specific for organofluorine; however, it is not specific for PFAS. It is a proven way to measure total fluorine in matrices containing high concentration of fluorinated polymeric material, which is a limitation of both the TOP assay and AOF. It also requires relatively minimal sample preparation to analyze fluorine content in commercial products. Sample preparation of environmental samples for PIGE analysis is likely to require a similar level of sample preparation, along with the limitations of extraction techniques, as the TOP assay or AOF.

Technique Limitations

PIGE is not specific to PFAS and, depending on the preparation, it is also not specific to organofluorine. The polymeric compounds that PIGE has been used to detect in consumer products may not contain perfluoroalkyl groups or may not be capable of breaking down to free PFAS.

PIGE also does not provide any differentiation on PFAS perfluoroalkyl chain length present in a sample. Depending on how the sample is prepared prior to the instrumental analysis, samples may be biased toward measurement of long-chain PFAS, as with the TOP assay and AOF.

Extraction methods for PFAS in environmental samples have not yet been demonstrated for this technique. When using SPE to extract environmental aqueous samples prior to PIGE analysis, cartridges that are suitable to hydrophobic and anionic PFAS may not retain positively charged PFAS of interest. For soil samples, the extraction method also determines the PFAS likely retained. However, by using targeted extraction techniques for PFAS in environmental samples, the method becomes much more specific for PFAS.

The range of operating conditions for PIGE has not been standardized and so far, the technique has been demonstrated with only one commercial laboratory.

11.2.2.4 Adsorbable Organic Fluorine with Combustion Ion Chromatography

AOF (Wagner et al. 2013) or extractable organofluorine (EOF) (Miyake, Yamashita, So, et al. 2007; Miyake, Yamashita, Rostkowski, et al. 2007; Yeung et al. 2008) paired with CIC (AOF/CIC or EOF/CIC) are complimentary terms for an analysis for fluorine content of environmental samples. In this application, an aqueous sample is passed through a carbon-based sorbent on which the fluorine-containing organics adhere. The carbon sorbent is then combusted at high temperatures that should completely decompose the organics into their elemental constituents. The gaseous stream is passed through deionized water, which is then analyzed for fluorine content (as fluoride) by ion chromatography.

The technique has been demonstrated on human blood samples (Miyake, Yamashita, So, et al. 2007; Yeung et al. 2008) and various environmental aqueous samples (Miyake, Yamashita, Rostkowski, et al. 2007; Wagner et al. 2013; Dauchy et al. 2017; Willach, Brauch, and Lange 2016). Presumably, the method could be adapted to other types of matrices to measure organofluorine in soils or biota. The matrices could be extracted for PFAS, the extract resuspended into an aqueous solution that could adhere to the activated carbon, and then analyzed with CIC. As with the TOP assay, the specific extraction procedures would influence whether some or all PFAS are retained and ultimately measured as combusted fluoride product. Alternatively, it is possible that the technique could be used without extraction to directly combust organofluorine-containing products.

Possible Technique Uses

AOF can be used to measure PFAS or other fluorine-containing compounds as an aggregate organofluorine concentration. If the method is available more readily or at a lower cost than LC/MS/MS measurement of PFAS, it can be a screening tool to determine if a significant concentration of fluorine-containing compounds is present in an aqueous sample or other sample from which the organofluorine content can be extracted. A detection limit of 0.77 µg/L fluorine (13 µg/L PFOS equivalent) (Willach, Brauch, and Lange 2016) was reported for one laboratory offering the technique, although the detection limit will vary by amount of sample processed and laboratory conducting the procedure.

Technique Limitations

Like PIGE, AOF is not specific to PFAS. If a sample contains relatively high concentrations of chemical compounds that are not targets of the investigation (for example, fluorine-containing pharmaceuticals), then the organofluorine may be falsely attributed to PFAS content and bias “total PFAS” measurements high.

AOF does not provide any differentiation on PFAS perfluoroalkyl chain length present in a sample. Some short-chain PFAS may be unable to sorb to the activated carbon material that is combusted, but this will depend significantly on laboratory-specific procedures.

Extraction methods for PFAS in commercial products and solid samples, coupled with this technique, have not yet been demonstrated for this technique. Inorganic fluoride concentrations may be challenging to remove from some matrices and would result in samples biased high for total organofluorine that was actually attributable to fluoride.

Like PIGE, the range of operating conditions for AOF-CIC has not been standardized. In addition to the limitations mentioned above, some matrices may contain sufficient competitive organics or other materials that coat the activated carbon to prevent complete retention of organofluorine compounds.

11.2.2.5 High-Resolution Mass Spectrometry (qTOF)

Technique Description

Quadrupole time-of-flight mass spectrometry (qTOF/MS) can be used to determine both the chemical formula and structure of unknown PFAS in a sample, but analytical standards are required for unequivocal structural identification.

High-resolution mass spectrometry has been used to tentatively identify the molecular formulas and structures of unknown PFAS (Newton et al. 2017; Moschet et al. 2017; Barzen-Hanson et al. 2017). Similar to targeted PFAS analysis, techniques such as LC or GC are used to separate compounds in a sample so that individual PFAS can be resolved. The mass is measured using a time-of-flight or other high-resolution detector, and the molecular formula is proposed. If an MS-MS technique is used, the fragments of the parent compound can be used to piece together the structural arrangement of the compound. To identify compounds that are specifically PFAS, versus other organics present in the sample, compounds with negative mass defects (that is, the accurate mass is slightly less than the nominal mass) can be selected. Fluorine is one of the few elements that has a negative mass defect, and the inclusion of multiple fluorines in a PFAS molecule means that net mass defect of the molecule will likely be negative. Compounds that are either 50 or 100 mass units apart also identify homologous series of PFAS separated by one or two CF_2 groups. MS libraries of previously identified PFAS exist for targeted matching, although they will not definitively identify an unknown compound.

Possible Technique Uses

Such high-resolution mass spectrometry analyses of PFAS can tentatively identify the structures of unknown PFAS and can also be used, in comparison with analytical standards of known compounds, to semiquantitatively estimate their concentrations. Accurate identification of compounds using high-resolution MS is a time-intensive and expensive process. Therefore, a high motivation for knowing the exact PFAS structure, for instance in differentiating forensically between two different sources, may be the biggest driver of its use for PFAS analysis. High-resolution MS is best suited for media in which unknown PFAS are likely to be present in significant concentrations. When many other non-PFAS compounds are present in the sample, the MS signal of competing compounds will likely obscure the signal of PFAS. Sample preparation steps can inadvertently or intentionally select for certain types of PFAS. As user skill and data interpretation time increase, accurate identification of PFAS is likely to improve.

Technique Limitations

High-resolution mass spectrometry cannot definitively identify the exact structure or formulas of PFAS without comparison to reference materials or analytical standards.

Not all PFAS, even if present in a prepared sample, can or will ionize under the conditions to which the instrument is tuned. A skilled instrument operator may be able to adjust the instrument conditions to match the types of compounds expected.

False positives are much more likely to result using high-resolution MS than with the TOP assay, AOF, or PIGE. Compounds may be mistakenly identified as PFAS, and even when correctly identified, their concentrations may be greatly over- or underestimated when other compounds are used for comparative quantitative purposes.

11.2.2.6 Chemical Ionization Mass Spectrometry (CIMS)

Chemical ionization mass spectrometry (CIMS) can be used to detect gas-phase PFAS, particularly fluorotelomer alcohols (FTOHs), and other oxygenated PFAS.

Although GC/MS is not a common or well-established technique used for PFAS analysis, it is particularly applicable to (ambient) atmospheric samples. Currently, there are no published methods for using GC/MS for PFAS analysis, despite the distinct advantages for certain compound classes (for example, fluoro-telomer alcohols) of using GC/MS directly or after derivatization (chemical reaction to convert analyte of interest to a GC/MS-amenable “derivatized compound”) (Langlois et al. 2007), or using large-volume injection (Scott et al. 2006).

11.3 Data Evaluation

Evaluation of data involves looking at all the factors that indicate whether the data are:

- precise (agreement between results that are supposed to be similar)
- accurate (how close they are to the true concentrations)
- representative (results characterize the site properly)
- comparable (data compare well to other data)
- complete (all the samples and compounds requested were reported, especially for critical samples that represent a point of exposure, such as drinking water)

- sensitive (nondetect data reported with concentrations below required regulatory or risk level)

These factors are illustrated in Figure 11-1, and guide users through the process of looking at their data (field collection and laboratory information) with a critical eye.

The USEPA has guidance to aid in evaluating PFAS drinking water data generated in accordance with USEPA 537, *Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFASs) Analyzed Using EPA Method 537*, as well as a technical bulletin to aid in the review of PFAS data generated for all other media, *Per- and Polyfluoroalkyl Substances (PFAS): Reviewing Analytical Methods Data for Environmental Samples*. The USDOD EDQW has published PFAS Data Validation Guidelines, for evaluation of PFAS data generated in accordance with the *DoD/DOE Quality System Manual for Environmental Laboratories, Version 5.4* (USDOD 2021). *A summary of key points from these data validation guidance documents, and others as noted in the table, has been compiled as* ***Table 11-6***, *PFAS Analytical Data Usability Table (included as a separate PDF).*

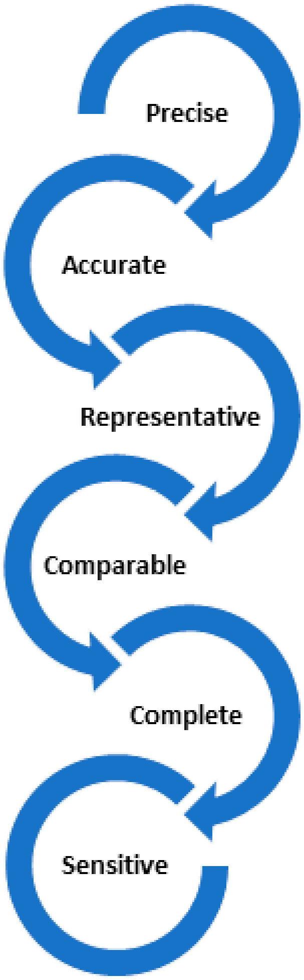

Figure 11-1. Data evaluation factors.

Source: H. Albertus-Benham, Wood Environment & Infrastructure, used with permission.

11.3.1 Presampling Planning

To ensure the usability of the data, communication with the laboratory that is performing the analysis is important. Until there are accepted methodologies for matrices other than drinking water, it is incumbent on the data user to collect information about the methodology to be employed by the laboratory. Figure 11-2 contains laboratory considerations related to data usability in order to plan a sampling program.

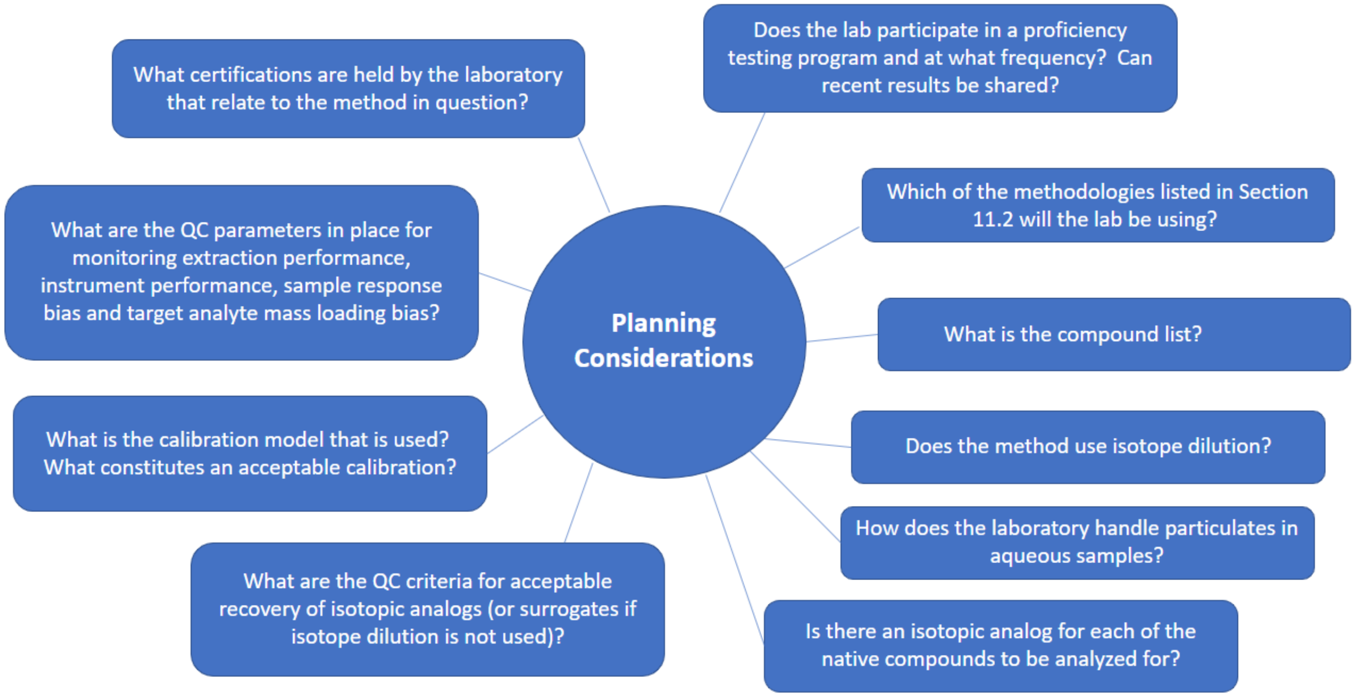

Figure 11-2. Laboratory planning considerations for data usability.

Source: Modified from figure by H. Albertus-Benham, Wood Environment & Infrastructure. Used with permission.

The most important goal of data usability is to ensure that the PFAS data generated are usable to meet the stated data needs and that the user understands any limitations in the use of the data due to potential uncertainty or bias. Overall usability of data is judged by evaluating the quality of the results compared to the data quality objectives (DQO) of the project. Therefore, establishing these project DQOs and communicating them to the field sampling team and the laboratory prior to sample collection and sample analysis is vital to ensuring that the correct methods, correct compounds, and adequate sensitivity are reported for your samples.

Throughout the rest of section 11.3, pre-sampling planning will be indicated by these yellow call-outs.

11.3.2 Overall Usability of the Data

Three questions are most important in evaluating data: (1) Have the results exceeded a level of concern?, (2) Do these results make sense?, and (3) Are data of acceptable quality? To judge whether results have exceeded a level of concern, the potential bias or uncertainty in the data should be evaluated along with the sensitivity of the results. At a minimum, it is recommended that a report from the laboratory contains a cover letter (or narrative) explaining sample receipt, analytical methods, and any QC deviations plus data sheets for field samples and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes), which should also contain results for sample-specific QC (such as internal standard recoveries). Often the most critical data for a project are the non-detects to prove the absence of compounds of concern at specific concentration levels (quantitation limits). Therefore, before evaluating QC associated with your samples, the data should be evaluated to ensure that all compounds required are reported with quantitation limits at or below the project's required sensitivity objective. If this sensitivity is not acceptable, then the data may be of very limited use.

If the compound list reported and quantitation limits are acceptable, then the associated QC results (for example, EIS recoveries, results of blanks, blank spike recoveries, etc.) can be compared to project DQOs to evaluate potential uncertainty in the data. The formal systematic process of this QC evaluation is called data review or validation. The approach to data validation is well documented; for example, see the USEPA *National Functional Guidelines* (USEPA 2020, USEPA 2020), and beyond the scope of this document; however, evaluation of all of the QC associated with the sampling and analysis of a set of samples will lead to an understanding of the uncertainty in the data.

Some critical QC issues might result in unusable data or concern for project actions. For example, if the data are considered biased low based on low QC results and the sample concentrations are at or near the level of concern or an action level, it may be that the true sample concentration actually exceeded the action level. Conversely, if the sample data are considered biased high based on high QC results and the sample concentrations are near but below the levels of concern or action level, then there is added certainty that the data do not exceed the action levels.

Once the data have been adequately reviewed for accuracy to determine if there are limitations to their use or uncertainties to be considered during use, the results should be evaluated by answering the following questions:

- Do field duplicates, if performed, indicate acceptable precision for the sampling and analysis?
- Do the data from the current sampling event compare well with historical data?
- Do the data make sense from a temporal point of view?
- Do the data make sense from a spatial point of view from one sampling point to the next across the project area?

Evaluating results may lead to overall project changes such as a need to increase sampling density to improve data representativeness, correction of procedures for collecting samples to minimize contamination, changes in methods of analysis to achieve project sensitivity requirements, etc.

This type of review can point out data trends or areas of concern (for example, interferences with project analytes) that could not be elucidated by looking at a single data point and may lead to overall project changes such as a need to increase sampling density to improve data representativeness, correction of procedures for collecting samples to minimize contamination, changes in methods of analysis to achieve project sensitivity requirements, etc. Following this review, the data user can determine whether the data set is complete and sufficient for project decisions and data uses or whether additional samples need to be collected and analyzed.

11.3.3 Sensitivity

A quantitation limit (QL), or Limit of Quantitation (LOQ) is the limit of accurate quantitation for a specific analyte in a specific sample after any adjustments have been made for sample amount, dilutions, or percent moisture. Typically, the QL or LOQ concentration is selected as the lowest nonzero standard in the calibration curve for each analyte. It takes into account the sample size, matrix effects, and any dilutions made during the analysis of that particular sample. Because of varying properties between samples, the QL can vary from sample to sample and analyte to analyte. The QL should represent the level at which reliable qualitative and quantitative information is routinely reported (**Table 11-3**, included in the PFAS Analytical Methods Excel spreadsheet). When project-specified decision levels or action levels are near the QL, at least one quantitation limit check is recommended in all sample batches to demonstrate adequate quantitation at the lowest concentration.

Sensitivity is related to the QL in that sensitivity refers to the capability of a method or instrument to identify a given analyte at a given concentration and reliably quantitate the analyte at that concentration. If a specified analyte is not reported by a laboratory to be in a specified sample, it does not necessarily mean that the chemical is not present; it is an indication that the concentration of the analyte may be below the method sensitivity.

Detected PFAS results between the method detection limit (MDL) and QL (that is, “J” values) can generally be reported as long as all qualitative identification criteria are achieved. Typical QLs for PFAS are as follows:

- common PFAS analytes in aqueous matrices: 2–8 ng/L
- common PFAS analytes in solid matrices: 0.2–2 ng/g

It is imperative that the RLs (and not the MDLs) for each method are evaluated versus the project screening criteria prior to submitting samples to the laboratory. The RLs should be below the project screening criteria to ensure achievement of project objectives.

Sometimes even though lower QLs were planned for, the laboratory may have to perform dilutions, which causes the QLs to be elevated. Ensure that the dilution performed by the laboratory was reasonable. If there are elevated concentrations of specific target analytes or interferences, then the dilution is likely justified and the presence of elevated QLs may not be an issue if these other target analytes are present at very high levels.

If a dilution was performed and it is not obvious why (for example, low concentrations or nondetect results for target analytes), then inquire with the laboratory why the dilution was performed. This could happen due to elevated concentrations of nontarget compounds but should be documented.

The QLs can also be affected by the sample preparation parameters, the mass of solid sample or volume of aqueous sample used in the extraction, or the final volume of the extracts. If a complex matrix is encountered, the sample sizes may be reduced and/or the injection volumes may be increased, causing the QL to be elevated accordingly.

11.3.4 Target Analyte Lists

Target analyte lists for PFAS will vary by laboratory and regulatory program. The data user should work with the laboratory to ensure that the correct list is being reported, as dictated by the project objectives. In general, Table 11-4 (included in the PFAS Analytical Methods Excel spreadsheet) includes the common PFAS reported by existing laboratories. The selected list may be dependent upon project objectives, regulatory requirements, as well as the potential source of PFAS contamination (for example, AFFF, landfill, chromium electroplating).

The data user should work with the laboratory to ensure that the correct list is being reported, as dictated by the project objectives.

11.3.5 Linear and Branched Isomers

It is also important to note that PFOS and PFOA (and other PFAS as well) contain a mixture of linear and branched isomers, which can be significant when the laboratory is quantifying these chemicals. Very few standards are available for branched isomers; some are qualitative and some are quantitative. If branched isomers are not included in the sample quantitation by the lab, the resulting concentrations will be underestimated.

In general, all laboratories should be reporting the sum of the linear and branched isomers for PFHxS, PFOS, PFOA, PFNA, PFOSA, NMeFOSE, NEtFOSE, NMeFOSA, NEtFOSA, NMeFOSAA, and NEtFOSAA because these are the PFAS for which both linear and branched analytical standards currently exist. In the absence of a standard that includes branched isomers, only the peak associated with the linear isomer is integrated. As more analytical standards become available, more PFAS should be reported as linear and branched in the future.

Figure 11-3 shows an example of the integration performed correctly and incorrectly. It is the responsibility of the data user to ensure that the laboratory is performing the integration of the target PFAS to include both linear and branched isomers. This requires upfront communication with the laboratory and a possible independent review of the laboratory raw data by a qualified chemist/data user to verify the integrations were properly performed.

Ensure that the contracted laboratory is performing the integration of the target PFAS to include both linear and branched isomers.

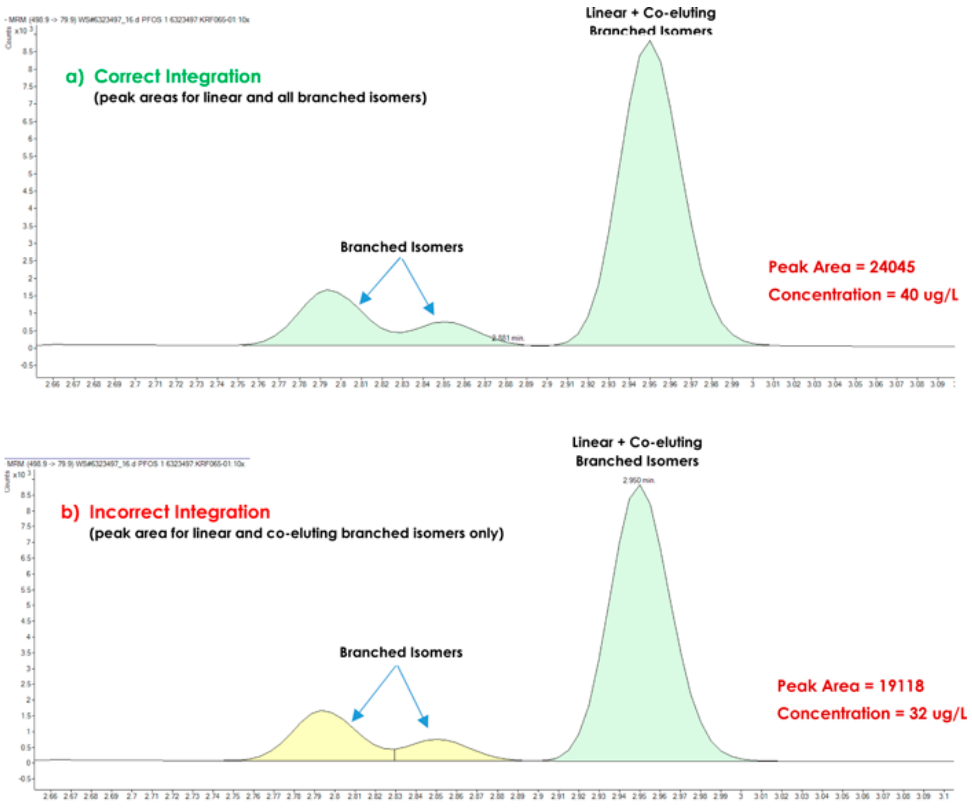

Figure 11-3. LC/MS/MS data illustrating a) complete integration of linear and branched PFOS, and b) partial integration of PFOS. Discrepancies in concentration will depend on the fraction of branched versus linear PFAS present, but in the current example PFOS concentrations in b) were 20% lower than in a).

Source: Bureau Veritas Laboratories, Mississauga, Ontario, Canada. Used with permission.

11.3.6 Isotope Dilution Standard Results and Surrogates

Isotope Dilution Standards

The isotope dilution technique involves quantitation of a compound of interest using a labeled isotope of that very compound. A variety of isotopically labeled analogs (for example, carbon-13 analogs of the compounds of interest) are added to a sample prior to extraction or prior to analysis when extraction is not required. These isotopically labeled analogs are sometimes referred to as extracted internal standards, as defined in the USEPA Draft Method 1633, and function from a data usability standpoint as both an internal standard (used in the calculation of the target compounds) and as a surrogate standard (calculation of the recovery of the standard). Ideally, the number of isotopically labeled analogs used in the isotope dilution technique matches the number of target compounds. For example, USEPA Method 537.1 uses three surrogates for 18 target compounds, while the isotope dilution procedure in USEPA Method 533 attempts to use an isotopically labeled analog for each compound being reported (USEPA 2020). Non-extracted internal standards are also added to the sample or extract immediately preceding analysis.

For the isotope dilution methods, quantitation of the target compounds is performed relative to the response of the isotopically labeled analog, which should recover in a manner similar to how the non-labeled compounds recover. Effectively, the sample data are recovery-corrected for losses that might have occurred during sample processing. The isotope dilution recovery correction procedure greatly improves the accuracy of the analysis and is considered to be an improvement over other techniques for the analysis of complex samples for analytes requiring high sensitivity. Chemical standards manufacturers are working to make a wider variety of labeled isotope compounds available to further improve accuracy of the methods for all compounds under investigation (for example, fluorotelomers, precursors, various isomers of carboxylates and sulfonates).

Acceptance criteria or control limits for isotopically labeled analog recoveries are either developed by the laboratory or dictated by the requirements of the project (for example, work plan or QAPP-specified criteria, regulatory criteria, or method criteria). Poor recovery of EIS in complex matrices is common; however, if a project requires ongoing analysis of a problematic matrix, the laboratory should perform method development to improve recovery, if possible (for example, change in cleanup procedures, change in the transition ions monitored, etc.). If EIS recovery is very low (for example, < 10% recovery), nondetects associated with the EIS may be false negatives and should not be used for project decisions. If EIS recovery is low and ≥10%, there may be an indeterminate bias for the affected PFAS. If EIS recovery is high, there will be no effect on non-detects, but positive results for PFAS may have an indeterminate bias.

In the case where an isotope dilution extract is analyzed and requires re-analysis at a high dilution, the sample extract may need to be refortified with labeled isotope compounds or, if possible, a smaller aliquot of sample may need to be extracted to obtain adequate responses of EISs. In reporting the final data, the isotope recovery results from the initial analysis should not be used to adjust the data from the secondary dilution analysis because these recoveries may be affected by ion suppression or ion enhancement due to the elevated concentrations of target PFAS and therefore may not be reflective of the extraction efficiency or other matrix interferences. The result from this scenario is no longer quantitated from an isotope dilution but is calculated from an internal standard calculation and should be noted as such in the case narrative.

Surrogates in Non-Isotope Dilution Procedures

Method 537.1 uses three surrogates for 18 target compounds, while EPA Method 8327 uses 19 surrogates for 24 target compounds. Injection internal standards are also added to the sample extract immediately preceding analysis. Quantitation of the target compounds and surrogates is performed relative to these injection internal standards. The results from the non-isotope dilution technique report concentrations of the target compounds and recovery results for surrogates, and it is up to the data user to determine the impact (that is, bias) of the extraction and analysis on the sample results because results are not recovery-corrected.

Acceptance criteria or control limits for surrogate recoveries are either developed by the laboratory or dictated by the requirements of the project (for example, work plan or QAPP-specified criteria, regulatory criteria, or method criteria). Poor recovery of surrogates in complex matrices is common; however, if a project requires ongoing analysis of a problematic matrix, the laboratory should perform method development to improve recovery, if possible (for example, change in cleanup procedures, change in the transition ions monitored, etc.). If the recovery for a surrogate is below criteria, compounds associated with this surrogate may be biased low. If surrogate recovery is very low (for example, < 10% recovery), nondetects associated with the surrogate may be false negatives and should not be used for project decisions. If surrogate recovery is high, there will be no effect on non-detects but positive results for PFAS may be biased high.

11.3.7 Blank Contamination

As a consequence of the extensive use of PFAS, samples that may not contain PFAS can become contaminated if they come into contact with samples or materials containing PFAS. The types of blanks commonly used to evaluate contamination are field-based blanks and lab-based blanks. Field-based blanks include field reagent blank (field blank), source water blank, and equipment rinse blank. Laboratory-based blanks include method blank, lab reagent blank, and instrument blank. Reagent, field, trip spike, and method blanks are prepared and analyzed using the same procedures as for the field samples. Instrument blanks are analyzed periodically to verify the instrument is clean for analysis of subsequent samples.

The possible sources of contamination that may occur during field collection activities and sample preparation and analysis and the recommended procedures to minimize contamination have been previously addressed in Section 11.1.

The reagent blank is used to evaluate the potential PFAS contamination from the reagent water source used to generate the field-based and laboratory-based blanks. A systematic review of all of the blank results compared to the associated field sample results (the group of samples associated with the field-based and lab-based blanks, or the analytical batch of samples associated with a specific method blank) must be made to determine whether the field sample results are accurate. For example, if the reagent water source used in the field is nondetect for PFAS, but contamination is found in any of the other field-based blanks, this indicates potential contamination of the associated field samples from the sample bottle itself and/or during collection, handling, or transport to the laboratory. However, if a laboratory-based blank is also contaminated, the contamination observed in the field-based blanks may have been due to sample handling at the laboratory.

If the conclusion of this systematic blank data review is that an associated sample result may have been contaminated, then the sample result is considered to be biased high or may be a false positive, depending on the magnitude of the blank contamination compared to the field sample result. A general rule of thumb is that if a sample contains a contaminant within 5x–10x the concentration in the associated blank, the results may be biased high or result in a false positive in the sample (USDOD 2019).

11.3.8 Duplicate Results

Laboratory replicates are two separate aliquots of the same sample prepared at the laboratory and put through the entire sample preparation and analytical procedures. Field duplicates are two separate samples collected at the same location at the same date and time that are prepared and analyzed in the same manner as the sample. Laboratory replicates may be performed in lieu of an LCS duplicate or MSD. It should be noted that sometimes laboratories report the results of laboratory replicates performed on samples that are from a different project (that is, batch QC); if the laboratory replicates reported are not from a sample at the site of interest, then these results should not be used in the evaluation of sample data.

During data evaluation, the relative percent difference of each detected analyte versus the acceptance limits should be reviewed. The acceptance limits should be provided within the laboratory report and are either regulatory- or method-specific. When both results are < 2x the QL, the potential uncertainty increases and therefore the acceptance criteria may need to be adjusted.

Review regulatory or method-specific acceptance limits with the laboratory, whether from a QAPP or laboratory-generated.

- If both results are < 2x the QL, relative percent difference criteria can be doubled.
- If one result is detected and one result is not detected, then the evaluation will depend on whether the detected result is > 2x the QL or not. If one result is > 2x the QL and the other result is nondetect, then the variability is considered unacceptable and there may be potential uncertainty in the

results for this sample.

Variability in laboratory replicate and field duplicate analyses could be from the sampling process, possibly due to an inefficient homogenization procedure in the field. It could also be from the laboratory aliquoting process, or it could be due to heterogeneity in the sample matrix. The effect on project objectives will depend on the screening criteria and how far above or below these criteria the results are. If the results are close to the criteria with significant variability, this may require collection of more samples to better represent the location. If results are significantly above or below the screening criteria with high variability, it may not adversely affect the ultimate decision-making process.

11.3.9 Acid Versus Anion Form of PFAAs

The data user should be aware of the form of PFAS the laboratory is reporting when comparing to project screening criteria. PFAS are typically formulated as acids, but they are present in the environment and in humans in the anionic form. The differences in names used are the result of the different names for the acid form and the anion form of the chemical (see also Section 2.2.3.1). For example, when perfluorobutanoic acid (PFBA) disassociates and loses its hydrogen in water, it becomes the anionic form (perfluorobutanoate). This becomes more important when looking at physical and chemical properties of these chemicals, because whether they exist as an acid, an anion, or a salt (cation) will affect how they behave in the environment. Typically, laboratories are reporting the acid form of the perfluorocarboxylic acids and perfluorosulfonic acids.

Some target PFAS, such as PFHxS and PFOS, are not available as acids, but rather as their corresponding potassium or sodium salts (K^+ or Na^+). These salts are acceptable starting materials for the stock standards, provided the measured mass is corrected for the salt content according to the equation below. Note that this correction will result in a minimal change to the mass of the acid, but still should be performed for consistency and comparability with other results to ensure the data user that the correct form of PFAS is represented in the final concentration.

$$mass_{acid} = measured\ mass_{salt} * (MW_{acid}/MW_{salt})$$

MW_{acid} = molecular weight of PFAA

MW_{salt} = molecular weight of purchased salt

CAS numbers will change depending on if the acid, anion, or salt form of the PFAS is reported (Table 11-7).

Table 11-7. Example of CAS number differences between acid and anion

Chemical	**CAS number**
PFOA: Perfluorooctanoate (anion)	45285-51-6
PFOA: Perfluorooctanoic acid (acid)	335-67-1

11.4 Source Identification

As discussed in Section 10.5 Source Identification, one area of focus for PFAS investigations has been environmental forensics, specifically source identification. With so many industrial processes and transport pathways from which PFAS contamination can originate, attributing the occurrence of one or more PFAS to a particular source is of growing interest.

Source identification relies on the compilation of multiple lines of evidence from analytical data and site information, and the use of uni- and multivariate statistical analyses. Analytical lines of evidence include:

- Extended Target Lists
- Linear/Branched Speciation
- Non-Target Analysis (NTA)

These methods are briefly described in this section.

11.4.1 Extended Target Lists

Many commercial laboratories now analyze for more than 40 individual PFAS and some of these compounds may be indicative of a particular product or industrial process. Analyzing samples taken from or near these possible sources and comparing the profiles of those results to the area of concern, also known as chemical fingerprinting, may provide a demonstration of comparability.

11.4.2 Linear/Branched Speciation

The separate reporting of branched and linear PFAS can provide some indication of the process used to synthesize the PFAS detected. Electrochemical fluorination (ECF) produces a larger number of branched isomers than fluorotelomerization, which may be useful in differentiating sources (Benskin, DeSilva, and Martin 2010). This is not default laboratory reporting and needs to be arranged in advance to confirm availability and method details. However, due to isomer-specific differences in instrument response, and fate, transport and organism uptake/depuration, the use of speciated data for any inference more than presence/absence of a manufacturing source type is challenging.

11.4.3 Nontargeted Analysis (NTA)

The ability to identify compounds not targeted for measurement by existing methods is an important need. Nontargeted analyses are critical to being able to identify these compounds. With nontargeted analyses, chromatography (liquid and gas) is combined with high resolution mass spectrometry and multiple ionization techniques to determine atomic molecular weight and associated fragments. These results can be compared to databases for tentative identification (Liu et al. 2019). Further spectral interpretation may result in structural identification. This is particularly useful where no chemical standards exist.

Nontargeted analysis includes high-resolution mass spectrometry and suspect screening and is discussed in more detail below.

11.4.3.1 High-Resolution Mass Spectrometry

Although not widely commercially available, recent source research has focused on the use of high-resolution mass spectrometry (HRMS) for more comprehensive qualitative determination and fingerprinting for source attribution. As evidenced by SERDP-ESTCP funded projects listed below, the development of mass spectral libraries to match non-targeted analytes to source profiles is part of the ongoing research. NTA uses HRMS such as time-of-flight (TOF), ion-trap, or Fourier transform ion cyclotron resonance (FT-ICR) to generate high resolution accurate mass data. As extensive data sets are generated using HRMS, informed data filtering approaches are used to filter the data specific to PFAS related analytes. The data are first screened against previously generated suspect screening libraries that contain chromatographic/spectrometric information for PFAS characteristic to sources such as AFFF formulation, industrial process, and/or manufactured products. Then, mass spectrometry-specific data analyses, such as Kendrick mass defect plots, and general uni- and multivariate statistical analyses, are used to attempt source identification based on the presence/relative abundance of PFAS identified against the suspect screening libraries and other information (Benotti et al. 2020; Charbonnet et al. 2021).

Secondary Sources

SERDP PFAS Novel Methods for PFAS Source Tracking and Allocations

https://www.serdp-estcp.org/News-and-Events/Blog/Novel-Methods-for-PFAS-Source-Tracking-and-Allocations

ER20–1375 Comprehensive Forensic Approach for Source Allocation of Poly- and Perfluoroalkyl Substances – Chris Higgins, Colorado School of Mines

ER20–1121 Establishing an Approach to PFAS Forensics and a PFAS Source Materials Forensic Library – Mark Benotti, NewFields Government Services

ER20–1205 Machine Learning Pattern Recognition for Forensic Analysis of Detected Per- and Polyfluoroalkyl Substances in Environmental Samples – Tohren Kibbey, University of Oklahoma

ER20–1265 Ultrahigh-Resolution Fourier-Transform Ion Cyclotron Resonance Mass Spectrometry for Fingerprinting, Source Tracking, and Allocation of Per- and Polyfluoroalkyl Substances (PFAS) – Jens Blotevogel, Colorado State University

ER20–1056 Improving Access and Utility of Analytical Data for the Confident Discovery, Identification, and Source-Attribution of PFAS in Environmental Matrices – Benjamin Place, NIST, Department of Commerce

Updated June 2022.

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EPA Federal Facilities Superfund Program – RPM Bulletin 2021-05

Considerations for PFAS Conceptual Site Models (CSMs) in the Uniform Federal Policy (UFP-) Quality Assurance Project Plan (QAPP),
Part 1 Sources, Sampling and Analysis

15 September 2021

Purpose

The purpose of this document is to identify per- and polyfluoroalkyl substances (PFAS)-specific issues to be considered when reviewing the conceptual site model (CSM) section (e.g., Worksheet #10) in PFAS-focused Remedial Investigation (RI) Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPPs). Part 1 of this bulletin addresses sources, sampling, and analysis while Part 2 addresses risk assessment and risk communications considerations. These considerations are rooted in existing guidance and represent best practices that have been observed to date. They are also based on observations from EPA review of numerous PFAS RI QAPPs prepared for Department of Defense (DOD) NPL sites.

Existing Guidance

EPA, 2020. Smart Scoping of an EPA-Lead Remedial Investigation/Feasibility Study. EPA 542-F-19-006. October 2020. https://semspub.epa.gov/work/HQ/100002571.pdf

EPA, 2018. Smart Scoping for Environmental Investigations, Technical Guide. EPA 542-G-18-04. November 2018. https://semspub.epa.gov/work/HQ/100001799.pdf

EPA, 2011. Environmental Cleanup Best Management Practices: Effective Use of the Project Life Cycle Conceptual Site Model. EPA 542-F-11-011. July 2011. https://www.epa.gov/remedytech/environmental-cleanup-best-management-practices-effective-use-project-life-cycle

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EPA, 1988. Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA. Interim Final. EPA 540-G-89-1004. https://semspub.epa.gov/work/06/901141.pdf

Intergovernmental Data Quality Task Force (IDQTF), 2005. Uniform Federal Policy for Quality Assurance Project Plans. Part 1. UFP-QAPP Manual. https://www.epa.gov/sites/default/files/documents/ufp_qapp_v1_0305.pdf

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Summary

1. PFAS are a complex group of chemicals; there are data gaps and rapidly evolving science. It is important to track ongoing analyses and research to anticipate updates.
2. CSMs (Worksheet #10 in the UFP-QAPP)[1] are critical tools that inform site sampling needs for assessing nature and extent of the contamination and providing data for human health and ecological risk assessments. They are also useful tools for documenting the state of knowledge and data gaps.
3. There are significant benefits to harmonizing (to the extent practicable) our expectations for PFAS sources, media, exposure pathways and receptors in the CSM.
4. Because sampling and analytical methods, fate and transport properties and toxicity data are evolving rapidly, the CSM should aim for a comprehensive characterization of ***all known and potential*** PFAS sources, PFAS analytes, co-contaminants, fate and transport, media, extent of contamination, exposure pathways and receptors. When EPA PFAS methods are not available, or cannot be used for other reasons, EPA expects that the lead federal agency will provide additional information to site regulators to independently review the adequacy of the chosen sampling and analytical method(s) to produce data that meet QAPP-defined quality requirements. Refer to EPA Federal Facilities Superfund Program RPM Bulletin 2021-01 for additional information on analytical method selection.
5. This documentation will allow us to anticipate changing needs and rapidly expand the scope of quantitative assessments as new methods and toxicity values become available, as well as to support planning for phased Remedial Investigation/ Feasibility Studies (RI/FS) if that approach is found acceptable by the lead agency and regulators (e.g., short-term focus on most likely and greatest PFAS sources to facilitate early actions, with other potential sources, extents and pathways addressed in follow on phases).
6. A site's PFAS-specific CSM should build on previous CSMs if available, and should be a three-dimensional evaluation that includes but is not limited to comingled contaminants, total depth of soil samples and/or wells, well screen information in

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[1] Found at: https://www.epa.gov/sites/default/files/documents/ufp_qapp_worksheets.pdf

relation to depth of GW, variations of GW depth over time (e.g., tidal influence, drought impacts, water reclamation/reinfiltration, etc.), seasonal variation/understanding and climate change considerations.

7. EPA expects that CSMs will be updated periodically based on new information and support the current phase of work at the site.
8. RPMs should consult with regional and national subject matter experts, including EPA's Environmental Response Team, throughout the process to determine which considerations are appropriate and apply to site-specific conditions.

Background

Quality Assurance Project Plans (QAPPs)

The purpose of the QAPP prepared by lead federal agencies is to integrate all technical and quality aspects for the life cycle of the project, including planning, implementation, and assessment (IDQTF, 2005). Many QAPPs, such as those prepared for remedial investigations, are subject to EPA's regulatory review and approval (IDQTF, 2005). PFAS QAPPs are generally EPA's first opportunity to weigh in on the proposed sampling and analysis plans and as such, the CSM is the cornerstone to determining sampling media and locations to inform our understanding of the nature and extent of the contamination and collect samples to support risk assessment. QAPPs for RIs are particularly important as they establish the approach to site sampling for collection of data needed to assess the nature and extent of the contamination and provide information for use in risk assessments.

Conceptual Site Models

EPA considers a robust, realistic, and comprehensive conceptual site model (CSM) essential to response action development, selection, and implementation (EPA 2018). Importantly, the CSM may function as a primary project planning and management tool (EPA 2020), and as such the CSM should incorporate all that is known about the site's current and potential future environmental conditions and use noting that it will evolve and mature over the project's life cycle. A realistic CSM accurately portrays critical conditions that affect the success of response actions and at a scale that addresses heterogeneity. In particular, the preliminary CSM, which is likely to be most appropriate for the baseline QAPP, provides a comprehensive overview of the site, based on available site-related documents (EPA 2011). As more information is collected, the CSM evolves to reflect updated knowledge and documents stakeholder consensus (or divergence) on known site conditions; uncertainty hypotheses; data gaps, needs, and collection plans; and potential remedial challenges. In the case of PFAS, initial sampling as well as site-specific knowledge from previous investigations hovering between preliminary and baseline stages may also support the CSM, depending on the site. We expect that the PFAS CSM will present most known and potential PFAS source areas, transport mechanisms, and pathways, exposure routes and receptors (i.e., a PFAS CSM would be incomplete if it focuses solely on one use/release scenario when the site may have other types of PFAS uses and releases).

PFAS CSM Challenges

The rapidly evolving state of the science and regulations on PFAS presents challenges to PFAS site remediation. The difficulty is having sufficient information to proceed with a response action knowing that information on PFAS will evolve as well as regulatory developments. For example, the focus of PFAS to date has been on human exposures via drinking water, but we know that we should characterize and assess PFAS releases to soil, surface water and sediment as well as uptake into biota, including wild food and agricultural products consumed by humans and impacts on ecological receptors. For example, today we do not know enough to make specific recommendations regarding PFAS in air. We know releases to air may occur, PFAS can be transported via air, and that deposition can occur. We know very little about PFAS toxicity via inhalation, so risks will be difficult to quantify. The intent of this document is to help Remedial Project Managers (RPMs) consider these PFAS-specific issues as they review and approve lead federal agency PFAS RI QAPPs.

Expectations and Considerations

Sources

Key Issue: The PFAS RI QAPP should account for all sources that are relevant to the site, and not just aqueous film forming foam (AFFF) releases.

Most of DoD's RI Work Plans prepared so far focus on AFFF releases as the main source of PFAS in the environment. In some instances, early scoping (e.g., Preliminary Assessment, Site Investigation) did not even consider other potential sources. When given the opportunity, EPA may have commented on this and in most cases, these comments were rejected. EPA maintains that other potential sources of PFAS contamination should be addressed. While AFFF may be a major source at many federal facilities, focusing solely on this one source may cause incomplete characterization of the nature and extent of contamination and other inadequacies in RIs, FSs and Records of Decision. In general, available PFAS toxicity values may lead to very low screening levels, triggers for action and response goals (e.g., parts per trillion in current and potential future drinking water), therefore, relatively small releases of PFAS can result in concentrations of interest. Documenting all known and potential sources in the CSM does not mean that all sources are high priority and must be investigated now, rather a complete CSM still allows for triage and prioritization, and facilitates tracking of work over time.

Potential PFAS sources may not be obvious. For example, metal plating and finishing shops, commonly found in the industrial areas of military bases, may have used fume suppressants that contain PFAS to reduce air emissions from plating baths. Installations often have or had their own wastewater treatment plants; activated sludge may accumulate PFAS from wastewater, may later be land disposed and leach PFAS. Landfills are also ubiquitous on federal facility sites and may contain a wide range of PFAS-containing materials. Many military facilities often include open burning (OB)and open detonation sites (OD), and since certain munitions may contain significant quantities of PFAS, these additional areas are of interest (Olsavsky et al 2020, Valluri et al 2019).

In some cases, sites have been ruled out based on lack of documentation of PFAS as an ingredient in a formulation. For example, we have reviewed Preliminary Assessments that rule out metalworking shops because there is no documentation of PFAS use. The lack of documentation of PFAS use is not sufficient to rule out a site for further investigation because

PFAS chemicals are generally not listed on Safety Data Sheets (SDS), or other product inserts. Instead, it is recommended to rely on peer-reviewed published data that documents PFAS use in certain processes and products. Based on current research, examples of products, uses and sources that are likely most relevant to other federal agencies include the following (Gluge et al 2020; ITRC2020a):

Aqueous Film Forming Foam (AFFF) fire-fighting foam
Wetting agent, mist suppression for harmful vapors, and surfactants in metal plating/electroplating and finishing
Hydraulic fluids additive in aviation and aerospace
Films and electrolytes in batteries and fuel cells
Anti-corrosives in various industrial operations (including DOE Gaseous Diffusion Plants)
Finishing agents for textiles, including use in firefighting protective clothing andwaterproofing tents/fabrics
Fillers and binders in munitions and munitions components
Research and development laboratories
Landfills
Wastewater treatment plants, industrial wastewater treatment plants, and associated drainage ditches
Paint shops, paint booths
Car and vehicle wash facilities
Open Burning/Open Detonation areas
Munitions filling areas

Comments on the scope should address the need to identify all potential PFAS sources in landfills, metal working, OB/OD, etc., in addition to AFFF use. We can emphasize the need to work with EPA and state regulators to prioritize sources and plan to address lower priority sources. A recent DOD report from their Office of the Inspector General supports this recommendation, noting that due to the exclusive focus on AFFF, "…a major source of potential PFAS exposure, and not on all sources of potential PFAS exposure... people and the environment may continue to be exposed to preventable risks" (Inspector General, DOD, 2021). Furthermore, the OIG recommends that DOD address potential exposures to PFAS from sources other than AFFF. An example of language that can be used in comments on the QAPP is:

> Under CERCLA the purpose of the remedial investigation is to define source areas of contamination, the potential pathways of migration, and the potential receptors and associated exposure pathways. To do this, all potential sources should be identified at the outset of the investigation. For PFAS, this is particularly important since the available toxicity values result in screening levels in the low parts per trillion, meaning that even small releases could result in the identification of PFAS as a chemical(s) of potential concern in the risk assessment. It is understandable that source areas may need to be prioritized, but this process should be well documented and agreed upon by the project team. EPA expects that the scoping of the CSM will strive to identify all known and potential source areas, that the responsible party will work with EPA to prioritize source areas and document the rationale, and that there will be a commitment, plan and timeline to evaluate lower priority sources.

PFAS Sampling and Analysis Plans

Determining what and where to sample is fundamental to the remedial investigation, and the sampling and analysis plan (SAP) should be analyzed in conjunction with the QAPP, making sure that field personnel are coordinating with laboratory analysts. Given the complexities and data gaps associated with PFAS, RPMs should work closely with regional hydrogeologists, risk assessors, chemists and data quality experts when assessing data needs and data collection approaches.

PFAS Analytical Methods

Key Issue: EPA expects that the lead federal agencies will use EPA analytical methods when they are available and meet site-specific DQOs. When they are not available or cannot be used for other reasons including data quality, EPA expects that the other federal agency will provide additional information to allow for independent quality reviews.

Specifically, EPA has had a longstanding dialogue with DOD, and in particular the Air Force, over the use of non-EPA methods to measure PFAS in groundwater. To ensure that data is usable for decision-making, we ask that the laboratory provides the most recent Demonstration of Capability (DOC) and limit of quantitation (LOQ) or lower limit of quantitation (LLOQ) verification data for EPA review. The DOC should include all target analytes as well as isotopic surrogates. PFAS analysis SOPs should be made available upon request. Collectively, these documents will allow EPA to review the quality of the submitted data, to make sure it is suitable for decision-making (e.g., see EPA RPM Bulletin onAnalytical Methods in QAPPs).

PFAS Fate and Transport

Key Issue: Most recent RI QAPPs focused on three PFAS analytes with current EPA toxicity values: PFOA, PFOS and PFBS. The EPA and non-EPA analytical methods commonly used in 2021 can quantify over 20 PFAS analytes. Reviewing all available PFAS data will be helpful to understanding PFAS fate, transport, and migration.

PFAS transport in the environment is influenced by PFAS characteristics such as chain length, ionic state, functional groups, and extent of fluorination. For example, shorter chain length chemicals tend to migrate more quickly than the longer chain versions. As a result, shorter chain PFAS may serve as sentinels for migration of PFOA and PFOS plumes. In addition, the conditions of the site such as soil type, water infiltration or precipitation, depth to groundwater, groundwater to surface water discharges, oxidation-reduction conditions, water flow rates, atmospheric conditions, and co-contaminants may affect transport (EPA, 2008; ITRC, 2020b). Many PFAS have surfactant properties. As a result, they may be found at interfaces, such as air-water, soil-water, etc., and if present at sufficient concentrations, may alter surface tension and other transport properties. In addition, PFAS may interact with other surfactants at sites.

A best practice is for the RI QAPP to include a description of how the investigation will make use of all available data in the characterization of the nature and extent of contamination, and the limitations of those data (e.g., drinking water or agricultural production wells have very long screen and may represent water quality differently than environmental investigation wells), and the identification of potential exposure pathways of interest. Transport and migration of other (non-PFAS) contaminants may be useful in identifying potential PFAS migration pathways; however, PFAS may move at different rates.

PFAS Media and Exposure Pathways

Key Issue: In general, it is a best practice to identify all known and potentially contaminated media and relevant exposure pathways in the CSM. This is particularly true for PFAS as we anticipate the development of new information that will allow us to assess more PFAS analytes in different media.

Due to knowledge and data gaps, there may be pressure to produce a more limited CSM that excludes uncertain pathways. In some cases, there may be insufficient information to assess risks associated with that pathway, e.g., there may not exist route-specific toxicity data. But one benefit of documenting a pathway that is known to be present but cannot be quantified is to track that pathway for future reference, given the evolving nature of PFAS. In addition, exposures that cannot be quantitively assessed can contribute to the uncertainties associated with the risk assessment, which may be important to document.

The best practice is to begin with identifying all known and potential contaminated media and exposure pathways, and document whether they are complete, potentially complete, incomplete, unknown, or not relevant. Include in the narrative the basis for inclusion in the CSM, along with assumptions and logic.

Air Pathway Considerations

Based on existing data and new research PFAS may be present in air (Wu et al. 2021). Measured and modeled Henry’s Law constants are used to predict that some PFAS may be volatile. These PFAS include fluorotelomer alcohols, PFSAs such as PFBS, and PFCAs such as PFBA. Fluorotelomer alcohols may oxidize to form PFCAs such as PFOA.

ITRC (2020c) summarized available data on PFAS in outdoor air, noting that some of the highest concentrations are associated with PFAS production and manufacturing, e.g., Barton et al measured PFOA ranging from 120,000 – 900,000 picograms per cubic meter (pg/m^3) at the fence line of a Chemours plant in Parkersburg, WV (Barton et al, 2006), but in general outdoor air concentrations tend to be much lower (1-20 pg/m^3). In most cases, it seems unlikely that the use of PFAS-containing materials at Federal Facilities sites would range as high as the values observed near PFAS manufacturing plants. In addition, different sources may have different arrays of PFAS present. One consequence of airreleases is both short- and long-term transport via wind, followed by deposition. There is evidence that PFAS can be released to air, where they can be deposited to the surface via deposition and/or rainwater (ITRC, 2020a).

There are studies of indoor air, and these measurements can be higher than outdoor air (e.g., averaging 1-400 pg/m^3) due to the presence of indoor consumer sources, which may off-gas overtime (ITRC 2020c). The presence of indoor sources would also complicate the assessment of potential vapor intrusion.

EPA published Other Test Methods 45 (OTM-45) in January 2021. This method is suitable for measuring PFAS emissions from stationary sources (https://www.epa.gov/sites/default/files/2021-01/documents/otm_45_semivolatile_pfas_1-13-21.pdf).

Currently there are no federal toxicity values that can be used to assess risks associated with inhalation exposures, but New York State has an air guideline concentration for PFOA of 0.0053 micrograms per cubic meter (µg/m^3), which is based on the 2001 ACGIH APFO occupational TLV-TWA of 0.01 milligrams per cubic meter (mg/m^3). This follows on Michigan's allowable concentration level of 0.07 µg/m^3 or less, for PFOA and PFOS individually or combined, with a 24-hour averaging time.

Identifying potential air pathway issues in the CSM that may or may not be a risk to known and potential receptors could result in significant risk communication challenges. RPMs should carefully consider the relative risks and benefits of raising this issue at this time. One consideration may be the availability of state guidance or state regulations. In addition, some issues to consider include:

- Presence of high levels of PFAS in shallow groundwater beneath buildings.
- Known vapor intrusion issues associated with other chemicals.

Options for addressing PFAS in air, include requesting that the releases and pathways be included in the CSM, using dashed lines in the visuals and caveated language in the narrative to capture potential concerns. In addition, the potential presence of air pathways and inhalation exposures can be described in the uncertainty section.

PFAS and Co-contaminants

Key Issue: Knowledge of all co-contaminants that may be present in media is critical to all phases of response decision-making, including the protection of staff that may be handling contaminated media, and is vital to accurate data interpretation and remedy selection.

Many facilities are separating PFAS investigations into separate operable units. Administratively, this approach may make sense, but care must be taken to refrain from focusing on PFAS in isolation of other contaminants that may be present. Specifically, EPA strongly recommends that each PFAS QAPP include a table that identifies co-contaminants in each OU, by media. This table is expected to be important for all facets of the investigation, as it provides historical knowledge. For example, having a working knowledge of all potential co-contaminants will ensure the protection of workers during field sampling.

Co-contaminants may interact with PFAS. For example, the surfactant nature of some PFAS may enhance transport of hydrocarbons and other hydrophobic chemicals. Existing groundwater monitoring networks (e.g., well construction) may or not be suitable for PFAS monitoring. Remedial actions for other co-contaminants may also affect PFAS. For example, bioventing to remediate hydrocarbons may oxidize PFAS precursors and intermediates. Conversely, anaerobic reduction technologies common in treating chlorinated solvents may retard PFAS precursor and intermediate oxidation. In addition, it will be necessary to consider the impact of co-contaminants when assessing remedial options. Finally, the presence of co-

contaminants can affect response protectiveness evaluations and site close-out.

Actions and Options for EPA Reviewers

1. Request in writing consideration and documentation of issues described in this Bulletin. Explain the basis for these requests, as outlined above.
2. Ensure that there is a record of our requests for the administrative record.
3. Consider elevating issues to Regional Management and FFRRO.

Citations

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EPA Federal Facilities Superfund Program – RPM Bulletin 2021-06

Considerations for PFAS Conceptual Site Models (CSMs) in the Uniform Federal Policy (UFP-) Quality Assurance Project Plan (QAPP), Part 2 Risk Assessment and Risk Communication Considerations

15 November 2021

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1. Purpose

EPA's Federal Facilities Restoration and Reuse Office (FFRRO) is developing a series of bulletins ("Remedial Project Manager (RPM) Bulletins") that summarize issues that arise during our work on federal facility National Priorities List (NPL) sites and propose a path forward to resolution. The bulletins are designed for rapid communication across regions and to promote consistent interpretation and implementation of issues of importance. The recommendations include suggestions for how to consider the issue or challenge, based on existing guidance, requirements, and flexibilities. The RPM Bulletin represents a coordinated consensus on the FFRRO's approach to addressing issues and challenges and help RPMs and technical support staff address provide a consistent response across regions. They are not intended to create new or revise existing policy and/or guidance.

The purpose of this document is to identify per- and polyfluoroalkyl substances (PFAS)-specific issues to be considered when reviewing the conceptual site model (CSM) section (e.g., Worksheet #10) in PFAS-focused Remedial Investigation (RI) Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPPs). Part 1 of this bulletin addresses sources, sampling, and analysis while Part 2 addresses risk assessment and risk communications considerations. These considerations are rooted in existing guidance and represent best practices that have been observed to date. They are also based on observations from EPA review of numerous PFAS RI QAPPs prepared for Department of Defense (DOD) NPL sites.

2. Existing Guidance

EPA, 2020. Smart Scoping of an EPA-Lead Remedial Investigation/Feasibility Study. EPA 542-F-19-006. October 2020. (https://semspub.epa.gov/work/HQ/100002571.pdf)

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3. Summary

1. PFAS are a complex group of chemicals; there are data gaps and the science is rapidly evolving. It is important to track ongoing analyses and research to anticipate updates.
2. Conceptual site models (Worksheet # 10 in the UFP-QAPP) are critical tools that inform site sampling needs for assessing nature and extent of the contamination and providing data for risk assessments. They are also useful tools for documenting the state of knowledge and data gaps.

3. There are significant benefits to harmonizing our expectations for PFAS sources, media, fate and transport, exposure pathways and receptors in the CSM.
4. A PFAS-specific CSM should build on previous CSMs if available.
5. EPA expects that CSMs will be updated periodically based on new information and support the current phase of work at the site.
6. It is important to get input from regional risk assessors on the initial CSM. This is an opportunity to work collaboratively to identify known and potential exposure scenarios, including those involving sensitive, susceptible and highly exposed populations, to ensure adequate sampling coverage.
7. RPMs should consult with regional subject and national subject matter experts, including EPA's Environmental Response Team, OSRTI's Human Health Regional Risk Assessment Forum (OHHRRAF), and Ecological Risk Assessment Forum (ERAF) throughout the process to determine which considerations are appropriate and apply to site-specific conditions.

4. Background

4.1. Quality Assurance Project Plans (QAPPs)

The purpose of the QAPP prepared by lead federal agencies is to integrate all technical and quality aspects for the life cycle of the project, including planning, implementation, and assessment (IDQTF, 2005). Many QAPPs, such as those prepared for remedial investigations, are subject to regulatory review and approval by EPA (IDQTF, 2005). PFAS QAPPs are generally EPA's first opportunity to weigh in on the proposed sampling and analysis plans and as such, the CSM is the cornerstone to determining sampling media and locations to inform our understanding of the nature and extent of the contamination and collect sampling to support risk assessment. QAPPs for remedial investigations (RIs) are particularly important as they establish the approach to site sampling for collection of data needed to assess the nature and extent of the contamination and provide information for use in risk assessments.

4.2. Conceptual Site Models

EPA considers a robust, realistic, and comprehensive CSM essential to response action development, selection, and implementation (EPA 2018). Importantly, the CSM is a primary project planning and management tool (EPA 2020), and as such the CSM should incorporate all that is known about the site's current and potential future environmental conditions and use noting that it will evolve and mature over the project's life cycle. A realistic CSM accurately portrays critical conditions that affect the success of response actions and at a scale that addresses heterogeneity. In particular, the Preliminary CSM, which is likely to be most appropriate for the baseline QAPP, provides a comprehensive overview of the site, based on available site-related documents (EPA 2011). As more information is collected, the Baseline CSM documents stakeholder consensus (or divergence) on known site conditions; uncertainty hypotheses; data gaps, needs, and collection plans; and potential remedial challenges. In the case of PFAS, the CSM may also be supported by initial sampling, as well as site-specific knowledge from previous investigations hovering between the Preliminary and Baseline stages.

The PFAS CSM should present most known and potential PFAS source areas, transport mechanisms, and pathways, exposure routes and receptors. A PFAS CSM would be incomplete if it focuses solely on one use/release scenario when the site may have other types of PFAS uses and releases).

4.3. PFAS CSM Challenges

The rapidly evolving state of the science and regulations on PFAS may have the biggest impact on PFAS site remediations. Our challenge is to find the right balance of anticipating scientific breakthroughs and regulatory action versus the need to make decisions and move forward expeditiously to address known and potential concerns. For example, the focus of PFAS to date has been on human exposures via drinking water; however, site-specific characterization and assessment of PFAS release to soil, surface water and sediment as well as uptake into biota and crops consumed by humans and impacts on ecological receptors are needed. Currently, we do not know enough to make specific recommendations regarding PFAS in air. PFAS can be transported via air, and deposition can occur. We know very little about PFAS toxicity via inhalation, so risks will be difficult to quantify. The intent of this document is to help RPMs consider these PFAS-specific issues as they review and approve lead federal agency PFAS RI QAPPs.

5. Expectations and Considerations

5.1. PFAS Risk Assessment Considerations

Considering risk assessment needs during RI scoping and QAPP preparation will help identify site-specific data needs and inform the sampling and analysis plans. Risk assessors can help identify areas or communities that may be more vulnerable to PFAS exposures, including communities with environmental justice concerns, areas with high co-exposures (such as elevated pesticide use or seafood consumption). PFAS-specific hazards and vulnerable populations can prompt consideration of potential exposure scenarios relevant to these communities. Risk assessment scoping can also identify species or ecological communities at increased risk. Exposure scenarios should consider the potential for bioaccumulation and biomagnification and include evaluations of sensitive wildlife receptors. These considerations should provide opportunities to ensure that the risk assessment sampling design will be comprehensive in its inclusion of relevant communities and exposure pathways for the site. RPMs should consult with regional risk assessors when considering data needs for risk assessment purposes.

5.1.1. PFAS Exposure

Key Issue: A review of PFAS data [e.g., measured analytes in addition to perfluorooctanoic acid (PFOA), perfluorooctane sulfonate (PFOS) and perfluorobutane sulfonic acid (PFBS)] available for the site can uncover hot spots of PFAS that may result in human and ecological exposures that require attention.

Currently, most other federal agencies are focusing solely on PFAS with current toxicity values. There are many reasons why it is important to consider the results of all measured PFAS analytes [refer to RPM Bulletin 2021-05, Considerations for PFAS Conceptual Site Models (CSMs) in the Uniform Federal Policy (UFP) Quality Assurance Project Plan (QAPP), Part 1 Sources, Sampling and Analysis in the RPM Bulletins Folder on Teams]. If a toxicity value is lacking for some PFAS analytes, potential exposures and risks may be considered qualitatively in the risk assessment. Refer to Risk Assessment Guidance for Superfund (RAGS) Part A, chapter 7.5, and Ecological Risk Assessment Guidance for Superfund (ERAGS) Step 1, Step 3, and Step 7. In addition, EPA's Office of Land and Emergency Management (OLEM) can provide this information to the Office of Research and Development (ORD), illustrating the need for toxicity value development due to documented exposures.

5.1.2. PFAS Toxicity Values

Key Issue: As more PFAS toxicity values are developed and/or adopted by EPA, the data will be re-evaluated to update CSMs.

Research on PFAS toxicity is ongoing and our access to toxicity values for use in risk assessments is evolving rapidly. As described above, it is prudent to have a working knowledge of, or easy access to, PFAS occurrence data so that areas can be rapidly reassessed, if needed, following issuance of new or updated PFAS toxicity values. For example, in April 2021, EPA updated the toxicity value for PFBS, and the resulting EPA Regional Screening Levels are significantly lower than the previous value proposed. An assessment of Gen X chemicals was released in October 2021, and PFOA and PFOS health advisories are under review. There is a trend to revising down reference values, based on new information. Sites previously evaluated that had no exceedances of the screening values may need to be re-evaluated using the updated toxicity value. EPA expects that as additional toxicity values are developed, iterative re-assessment will be needed. The fluid nature of the toxicity values should not be taken as a reason to "wait" to proceed at sites, as that would potentially postpone certain response actions for many years. Rather, management of uncertainty is a hallmark of the Superfund program and can be incorporated into investigations, decision making and cleanup actions.

5.1.3. Human Receptors

Key Issue: The identification of potentially exposed receptors is a critical step in making decisions about what media to sample. It is important to ensure the other federal agencies identify all potentially exposed receptors, incorporating environmental justice considerations and including vulnerable and susceptible populations, based on reasonably anticipated future uses.

According to ATSDR (2021), which assessed hazard data on 12 PFAS chemicals, available epidemiological studies suggest associations between PFAS exposure and several health outcomes; however, cause-and-effect relationships have not been established for these outcomes:

- Pregnancy-induced hypertension/pre-eclampsia (PFOA, PFOS)
- Increases in serum hepatic enzymes, particularly alanine aminotransferase (ALT), and decreases in serum bilirubin levels (PFOA, PFOS, PFHxS)
- Increases in serum lipids, particularly total cholesterol, and low-density lipoprotein (LDL) cholesterol (PFOA, PFOS, PFNA, PFDA)
- Decreased antibody response to vaccines (PFOA, PFOS, PFHxS, PFDA)
- Small decreases in birth weight (PFOA, PFOS)

Key Issue: PFAS sampling and analysis plans should be developed with relevant highly exposed, vulnerable and/or sensitive receptors in mind. RPMs should work with regional and/or national risk assessment personnel to review available information and select appropriate media-specific screening values, and toxicity values for use on a site-specific basis.

Determining the reasonable maximum exposure (RME) is a fundamental concept in Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) risk assessments. To identify the RME, it is important to consider populations that may experience differential or disproportionate exposures, including communities that may have environmental justice considerations, as well as relevant vulnerable and/or sensitive receptors (refer to RAGS Part A, Chapter 6.1.2).

Receptors who *may* be more vulnerable to PFAS exposures, based on associated health effects include:

- Women of childbearing age (Pregnancy-induced hypertension/pre-eclampsia)
- Developing fetuses (low birth weight, linked to several adverse health outcomes)
- Children (response to vaccines)
- People with compromised immune systems (response to vaccines)
- People with compromised liver function (hepatic enzymes)
- Overweight and obese people (changes in serum lipids, cholesterol)

5.1.4. Ecological Receptors

Key Issue: The CSM should include site-specific ecological receptors and RPMs should work with regional and/or national ecological risk assessment support personnel to review available information and select appropriate media-specific screening values, and toxicity values for use on a site-specific basis.

The CSM should include ecological receptors groups by evaluating impacts on food webs, including but not limited to aquatic and terrestrial birds, aquatic and terrestrial mammals, aquatic and terrestrial plants, benthic and soil invertebrates, fish, amphibians, and reptiles. Ecological effects will be site-specific due to the characteristics of the contaminants, environment and habitat, species present, and presence of potentially complete ecological exposure pathways. Threatened and endangered species and sensitive habitats should be identified in the CSM, using standard tools, such as US Fish and Wildlife Services Information for Planning and Consultation[1]

Regional ecological risk assessment personnel can review toxicity values available in the published literature (e.g., Conder et al., 2019; Zodrow et al, 2021; Johnson et al 2021; Ankley et al. 2021), and additionally from state agencies, to determine which toxicity values may be appropriate for use on a site-specific basis. Identifying analytes of ecological concern, appropriate receptors, assessment endpoints, media-specific screening values, and toxicity reference values early in the remedial investigation and risk assessment process will provide maximum support to the RPM in the oversight of media sampling and the identification of sampling locations; PFAS analysis requirements; and biological tissue sampling needs.

5.2. PFAS Risk Communication Considerations

The challenge of communicating potential hazards, exposures and risks associated with PFAS, given all the data gaps and uncertainties, should not be underestimated. A strategy of planning ahead is recommended for PFAS risk communication. This may include preparing to explain what we know, what we don't know, what EPA is doing to address risks from PFAS and protect public health and the environment, and what the public can do to reduce risks. Risk communication and community outreach can and should start early in the remedial investigation process, and as members of the project team, EPA RPMs can encourage other federal agencies to do so.

6. Actions and Options for EPA Reviewers of CSMs

1. RPMs should make sure they are familiar with existing guidance and best practices for CSMs and ensure that federal facilities are following these.
2. Consult with regional and national subject matter experts, including EPA's Environmental Response Team, OHHRRAF, FFRRO and Science Policy Branch, throughout the process to determine which considerations are appropriate and apply to site-specific conditions. A good way to find subject matter experts is through the Superfund TechHub In-House Technical Advice and Support Resources.
3. In written comments to the other federal agency, request consideration and documentation of issues described in this Bulletin. Explain the basis for these requests, as outlined above.
4. If there is resistance from the other federal agency, ensure that there is a record of our requests for the administrative record.

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[1] https://ecos.fws.gov/ipac/

5. If necessary, elevating issues to Regional Management and to FFRRO.

7. Citations

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United States
Environmental Protection
Agency

Multi-Industry Per- and Polyfluoroalkyl Substances (PFAS) Study – 2021 Preliminary Report

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Abbreviations

ACC	American Chemistry Council
ADONA	trade name for ammonium 4,8-dioxa-3H-perfluorononanoate, one chemical used in a 3M fluoropolymer processing aid technology
AF&PA	American Forest and Paper Association
AFFF	aqueous film-forming foam
APFO	ammonium perfluorooctanoate (ammonium salt of PFOA)
ASTSWMO	Association of State and Territorial Solid Waste Management Officials
ATSDR	United States Department of Health and Human Services, Agency for Toxic Substances and Disease Registry
BAF	bioaccumulation factor
BCF	bioconcentration factor
CAFE	National Oceanic and Atmospheric Administration Chemical Aquatic Fate and Effects database
CBI	confidential business information
CDR	Chemical Data Reporting
CFR	Code of Federal Regulations
CWA	Clean Water Act
DMR	discharge monitoring report
DOD	United States Department of Defense
DONA	trade name for 4,8-dioxa-3H-perfluorononanoic acid, one chemical used in a 3M fluoropolymer processing aid technology
DWTD	Drinking Water Treatability Database
DWTP	drinking water treatment plant
ELGs	Effluent Limitations Guidelines and Standards
EPA	United States Environmental Protection Agency
EPA OPPT	United States Environmental Protection Agency, Office of Chemical Safety and Pollution Prevention, Office of Pollution Prevention and Toxics
ETFE	ethylene tetrafluoroethylene
F-53B	trade name for chlorinated polyfluoroalkyl ether sulfonic acids, including 9Cl-PF3ONS (“F-53B major”), 11Cl-PF3OUdS (“F-53B minor”), and their potassium salts
FAA	United States Department of Transportation, Federal Aviation Administration
FASA	perfluoroalkane sulfonamide
FASAA	perfluoroalkane sulfonamido acetic acid
FASE	perfluoroalkane sulfonamido ethanol
FCN	food contact substance notification
FCS	food contact substance
FDA	United States Department of Health and Human Services, Food and Drug Administration
FR	Federal Register
FTCA	fluorotelomer carboxylic acid
FTOH	fluorotelomer alcohol
FTSA	fluorotelomer sulfonic acid
GAC	granular activated carbon
GenX	trade name for fluoropolymer processing aid technology that involves includes HFPO-DA and its ammonium salt
HFPO-DA	hexafluoropropylene oxide dimer acid, one chemical used in the GenX fluoropolymer processing aid technology
IWTT	Industrial Wastewater Treatment Technology
IX	ion exchange
K-9Cl-PF3ONS	potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate (potassium salt of F-53B major)
K-11Cl-PF3OUdS	potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate (potassium salt of F-53B minor)
lb/year	pounds per year
LHA	lifetime health advisory
MCL	Maximum Contaminant Level

MI EGLE	Michigan Department of Environment, Great Lakes, and Energy
MGD	million gallons per day
NaDONA	sodium dodecafluoro-3H-4, 8-dioxanonanoate
NASF	National Association for Surface Finishing
NESHAP	National Emission Standards for Hazardous Air Pollutants
NFDHA	perfluoro-3,6-dioxaheptanoic acid
NEtFOSAA	N-ethyl perfluorooctane sulfonamido acetic acid
NEtFOSE	N-ethyl perfluorooctane sulfonamido ethanol
NEtPFOSA	N-ethyl perfluorooctane sulfonamide
ng/L	nanograms per liter
NIH	United States Department of Health and Human Services, National Institutes of Health
NJ DEP	New Jersey Department of Environmental Protection
NMeFOSAA	N-methyl perfluorooctane sulfonamido acetic acid
NMeFOSE	N-methyl perfluorooctane sulfonamido ethanol
NMePFOSA	N-methyl perfluorooctane sulfonamide
NOAA	United States Department of Commerce, National Oceanic and Atmospheric Administration
NPDES	National Pollutant Discharge Elimination System
OCPSF	organic chemicals, plastics, and synthetic fibers
PAC	powdered activated carbon
POTW	publicly owned treatment works
PFAA	perfluoroalkyl acid
PFAS	per- and polyfluoroalkyl substances
PFBA	perfluorobutanoic acid
PFBS	perfluorobutane sulfonic acid
PFCA	perfluoroalkyl carboxylic acid
PFDA	perfluorodecanoic acid
PFDS	perfluorodecane sulfonic acid
PFDoA	perfluorododecanoic acid
PFEA	per- and polyfluoroalkyl ether acid
PFECA	perfluoroalkyl ether carboxylic acid
PFECA-G	perfluoro-4-isopropoxybutanoic acid
PFESA	perfluoroalkyl ether sulfonic acid
PFESA-BP1	perfluoro-3,6-dioxa-4-methyl-7-octene-1-sulfonic acid
PFESA-BP2	Perfluoro-2-{[perfluoro-3-(perfluoroethoxy)-2-propanyl]oxy}ethane sulfonic acid
PFHpA	perfluoroheptanoic acid
PFHpS	perfluoroheptane sulfonic acid
PFHxA	perfluorohexanoic acid
PFHxDA	perfluorohexadecanoic acid
PFHxS	perfluorohexane sulfonic acid
PFMOAA	perfluoro-2-methoxyacetic acid
PFNA	perfluorononanoic acid
PFNS	perfluorononane sulfonic acid
PFOA	perfluorooctanoic acid
PFODA	perfluorooctadecanoic acid
PFO2HxA	perfluoro-3,5-dioxahexanoic acid
PFO3OA	perfluoro-3,5,7-trioxaoctanoic acid
PFO4DA	perfluoro-3,5,7,9-tetraoxadecanoic acid
PFO5DA	perfluoro-3,5,7,9,11-pentaoxadodecanoic acid
PFOS	perfluorooctane sulfonic acid
PFOSA	perfluorooctane sulfonamide
PFPE	perfluoropolyether
PFPeA	perfluoropentanoic acid
PFPeS	perfluoropentane sulfonic acid
PFSA	perfluoroalkane sulfonic acid
PFTeA	perfluorotetradecanoic acid
PFTrA	perfluorotridecanoic acid
PFUnA	perfluoroundecanoic acid

PITT	PFAS Innovative Treatment Team
PMPA	perfluoromethoxypropyl carboxylic acid
PTFE	polytetrafluoroethylene
RfD	reference dose
RO	reverse osmosis
SIC	United States Department of Commerce Bureau of the Census Standard Industrial Classification system
SNUR	Significant New User Rule
TOXNET	National Institutes of Health Toxicology Data Network
TRI	Toxics Release Inventory
TSCA	Toxic Substances Control Act
WI DNR	Wisconsin Department of Natural Resources
µg/L	micrograms per liter
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (F-53B minor)
4:2 FTSA	4:2 fluorotelomer sulfonic acid
5:3 FTCA	2H, 2H, 3H, 3H-perfluorooctanoic acid
6:2 FTOH	6:2 fluorotelomer alcohol
6:2 FTSA	6:2 fluorotelomer sulfonic acid
7:3 FTCA	2H, 2H, 3H, 3H-perfluorodecanoic acid
8:2 FTSA	8:2 fluorotelomer sulfonic acid
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (F-53B major)

1. Executive Summary

The purpose of this preliminary report is to summarize the readily available information and data the United States Environmental Protection Agency's (EPA) Office of Water collected and reviewed concerning industrial discharges of per- and polyfluoroalkyl substances (PFAS) from five industrial point source categories: organic chemicals, plastics, and synthetic fibers (OCPSF) manufacturing; metal finishing; pulp, paper, and paperboard manufacturing; textile mills; and commercial airports.[1]

PFAS are a family of thousands of synthetic organic chemicals that contain a chain of carbon-fluorine bonds, one of the strongest chemical bonds. Many PFAS are highly stable, water- and oil-resistant, and exhibit other properties that make them useful in a variety of consumer products and industrial processes. Owing to these properties, PFAS do not easily degrade naturally and thus accumulate over time. According to the United States Department of Health and Human Services, Agency for Toxic Substances and Disease Registry (ATSDR), the environmental persistence and mobility of some PFAS, combined with decades of widespread use, have resulted in their presence in surface water, groundwater, drinking water, rainwater, soil, sediment, ice caps, outdoor and indoor air, plants, animal tissue, and human blood serum across the globe. Exposure to certain PFAS can lead to adverse human health impacts (ATSDR, 2021).

The global regulatory community has historically been interested in two groups of PFAS: (1) long-chain perfluoroalkane sulfonic acids (PFSAs), including perfluorooctane sulfonic acid (PFOS); and (2) long-chain perfluoroalkyl carboxylic acids (PFCAs), including perfluorooctanoic acid (PFOA). Long-chain PFAS, including PFOA and PFOS, were manufactured and used in the United States since the 1940s. Due to evidence of long-term persistence and adverse health outcomes associated with long-chain PFAS, EPA implemented restrictions on the manufacture, use, and import of certain long-chain PFAS in the United States and some manufacturers have voluntarily phased out these chemicals. More recently, industry has developed and adopted alternative short-chain PFAS chemistries to replace long-chain PFAS. Many short-chain PFAS are structurally similar to their long-chain predecessors being replaced and are manufactured by the same companies. Publicly available health, toxicity, and hazard assessments of short-chain PFAS are limited. Available information suggests short-chain PFAS generally pose less risk to overall human health and exhibit lower persistence in humans than long-chain PFAS such as PFOA and PFOS. However, short-chain PFAS are environmentally persistent and some demonstrate potential to cause adverse effects on animal and human health.

This preliminary report summarizes the manufacture, use, and discharge of PFAS from facilities in the five industrial point source categories EPA reviewed. This preliminary report presents EPA's estimates of the types and concentrations of PFAS, including legacy long-chain PFAS and replacement short-chain PFAS, present in wastewater discharges from these facilities. Few facilities in these industries currently have monitoring requirements, effluent limitations, or pretreatment standards for PFAS in their wastewater discharge permits. EPA identified available wastewater treatment technologies, such as activated carbon, ion exchange, and membrane filtration, that may reduce PFAS in wastewater discharges from facilities in these industrial point source categories.

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[1] For this study, EPA focused on PFAS manufacturers and PFAS formulators for its review of the OCPSF point source category. "PFAS manufacturers" refers to facilities that manufacture PFAS through electrochemical fluorination, fluorotelomerization, or other processes. "PFAS formulators" refers to facilities that blend, convert, or integrate PFAS feedstocks with other materials to produce new commercial or intermediate products.

2. Multi-Industry PFAS Study Background

The Clean Water Act (CWA) directs EPA to promulgate Effluent Limitations Guidelines and Standards (ELGs) that specify the attainable effluent pollutant reduction based on performance of pollution control technologies which are, or can be, employed within each industrial point source category. EPA develops ELGs on an industry-by-industry basis. These national, technology-based controls apply to pollutants discharged from facilities directly into surface waters of the United States or to publicly owned treatment works (POTWs). EPA's goal in establishing ELGs is to ensure that industrial facilities with similar characteristics will, at a minimum, meet similar effluent limitations or pretreatment standards. These effluent limitations and pretreatment standards represent the performance of the "best" pollution control technologies, regardless of geography or the nature of their receiving water or POTW. Although the limitations are based on performance of specific technologies, the regulations do not require use of a specific control technology to achieve the limitations. Facilities may use any method or technology (other than dilution) to comply with the limitations. See EPA's Industrial Effluent Guidelines webpage for more information on ELGs.

To date, EPA has promulgated ELGs for 59 industrial point source categories. These ELGs apply to between 35,000 and 45,000 facilities that discharge to surface waters (direct dischargers), as well as another 129,000 facilities that discharge to POTWs (indirect dischargers), in the United States. The effluent limitations for direct dischargers are implemented through National Pollutant Discharge Elimination System (NPDES) permits issued by authorized states or EPA regional offices. The standards for indirect dischargers are implemented through pretreatment permits or other control mechanisms issued and enforced by POTWs, states, and EPA regional offices. EPA has not established any national technology-based numeric standards for PFAS in industrial wastewater discharges and none of the current ELGs establish effluent limitations or pretreatment standards for PFAS.[2]

As part of the statutorily required ELG planning process, EPA's Office of Water examined readily available public information about PFAS discharges. The *Preliminary Effluent Guidelines Program Plan 14* and a supporting report, *The EPA's Review of Per- and Polyfluoroalkyl Substances (PFAS) in Industrial Wastewater Discharge*, published in October 2019, describe the review activities and findings of the initial examination and identify several industries with facilities that are likely to be discharging PFAS in their wastewater (EPA, 2019a, 2019b). EPA determined that further data collection and study were necessary to inform decisions about how best to address industrial PFAS discharges and announced the Multi-Industry PFAS Study. The Multi-Industry PFAS Study focuses on continuing data collection and review of PFAS manufacture, use, control, and discharge by industries that EPA determined were likely to be discharging PFAS in their wastewater in the preliminary review. The objectives of the Multi-Industry PFAS Study are to:

- Examine specific industrial categories and facilities manufacturing or using PFAS.
- Identify specific industrial facilities discharging PFAS in their wastewater.
- Collect, compile, and review information and data on PFAS in industrial discharges.
- Determine the types and concentrations of PFAS discharged in wastewater, based on available data and information collected by EPA.
- Assess availability and feasibility of control practices and treatment technologies capable of reducing or eliminating PFAS in wastewater discharges.

EPA focused on five industrial point source categories in the Multi-Industry PFAS Study: organic chemicals, plastics, and synthetic fibers (OCPSF); metal finishing; pulp, paper, and paperboard; textile mills; and commercial airports.[3] Table 1 describes these five point source categories, applicable ELGs in the Code of Federal Regulations (CFR), and potential uses/sources of PFAS.

[2] Where EPA has not promulgated an applicable ELG for direct or indirect dischargers, technology-based effluent limitations or pretreatment standards may be established based on the best professional judgement of the permitting authority.

[3] For this study, EPA focused on PFAS manufacturers and PFAS formulators for its review of the OCPSF point source category. "PFAS manufacturers" refers to facilities that manufacture PFAS through electrochemical fluorination, fluorotelomerization, or other processes. "PFAS formulators" refers to facilities that blend, convert, or integrate PFAS feedstocks with other materials to produce new commercial or intermediate products.

Table 1. Point Source Categories Included in Multi-Industry PFAS Study

Point Source Category	Description	Uses or Sources of PFAS [a]
Organic Chemicals, Plastics, and Synthetic Fibers (OCPSF)	Industrial facilities that manufacture organic chemicals, plastics, synthetic fibers or resin products, including those that manufacture PFAS or process PFAS in production of such products. Subject to ELGs in 40 CFR Part 414.	- Manufacture PFAS through electrochemical fluorination, telomerization, or other processes. - Polymerization processing aids. - Production of plastic, rubber, and resin. - Present in manufacture of commercial chemical products (e.g., carpet cleaning sprays, cleaning agents, protective coatings).
Metal Finishing	Industrial facilities that change the surface of an object to improve its appearance or durability. Includes six primary operations: electroplating, electroless plating, anodizing, coating, printed circuit board manufacturing, and chemical etching and milling. Subject to ELGs in 40 CFR Part 433.	- PFAS-containing chemicals used as wetting agents, mist and fume suppressants to prevent air emissions of toxic metal fumes, agents to reduce mechanical wear, and surface coatings to impart certain characteristics (e.g., reduced corrosion, enhanced appearance).
Pulp, Paper, and Paperboard	Mills that convert wood into pulp, paper, paperboard, and other cellulose-based products. Subject to ELGs in 40 CFR Part 430.	- PFAS-containing chemicals used to impart products with water and grease repellency (e.g., food packaging, coated papers). - Recycling of paper and paperboard products treated with PFAS.
Textile Mills	Mills that receive and prepare fibers; transform materials into yarn, thread, or webbing; convert yarn and webbing into fabric or related products; or finish these materials to produce consumer products (e.g., thread, yarn, bolt fabric, hosiery, towels, sheets, carpet). Subject to ELGs in 40 CFR Part 410.	- PFAS-containing chemicals used to impart outdoor gear, clothing, household, and other textile products with water, oil, soil, and heat resistance.
Commercial Airports	Commercial facilities associated with commercial air transport or aircraft flight operations. Excludes facilities operated by the United States Department of Defense (DOD). Subject to ELGs in 40 CFR Part 449.	- PFAS are a component of aqueous film-forming foam (AFFF), used for exterminating hydrocarbon fuel fires and firefighting training.

a – In general, PFAS may be used as coatings or surfactants for mechanical components (e.g., semiconductors, wiring, tubing, piping, seals, gaskets, etc.) used at many types of industrial facilities.

3. PFAS Overview

This section provides background information on PFAS, with a focus on chemicals and classes discussed in this preliminary report, and discusses industrial trends in the manufacture, import, and use of certain PFAS; environmental fate and transport of PFAS; and PFAS exposure and health effects. This report focuses on 52 PFAS, listed in Table 2, for which EPA collected discharge data as part of the Multi-Industry PFAS Study and are included in EPA's Cross-Agency PFAS Research List. As of August 2021, EPA's Cross-Agency PFAS Research List comprises 199 PFAS compiled from public and internal sources and literature searches by EPA researchers and program office representatives (EPA, 2021a). This list includes the PFAS most frequently detected in organisms and the environment, those included in state or federal standards, and PFAS reported in EPA's national data sets.

3.1 PFAS Classifications and Characteristics

PFAS are a family of thousands of synthetic organic chemicals characterized by linear or branched carbon-fluorine chains connected to a functional group. For the Multi-Industry PFAS Study, EPA used the following technical definition for PFAS:

> *Per- and polyfluorinated substances that structurally contain the unit R-(CF_2)-C(F)(R')R'' where both the CF_2 and CF moieties are saturated carbons and none of the R groups (R, R', or R'') can be hydrogen.*

EPA's National Center for Computational Toxicology maintains a master list of more than 5,000 chemicals with defined structures that are potential PFAS (EPA, 2020a); however, it is likely that less than half of these are commercially active in the United States. PFAS vary widely in chemical and physical properties, behavior, and potential risks to human health and the environment. Differences in the chemical structure, carbon chain length, degree of fluorination, and chemical functional group(s) of individual PFAS have implications for their mobility, fate, and degradation in the environment, as well as uptake, metabolism, clearance, and toxicity in humans, plants, and animals.

Many PFAS are chemically and thermally stable, reduce surface tension, and are resistant to heat, water, and oil. These properties make PFAS useful in many consumer products and industrial processes, but also make PFAS persistent in the environment. The small size, high electronegativity, and low polarizability of the fluorine atom, and the strength of the carbon-fluorine covalent bond are responsible for many of the unique and desirable characteristics of PFAS. See EPA's 2019 report *The EPA's Review of Per- and Polyfluoroalkyl Substances (PFAS) in Industrial Wastewater Discharge* for a summary of potential industrial sources of PFAS identified during EPA's 2019 preliminary review (EPA, 2019b).

Two processes, electrochemical fluorination and fluorotelomerization, are commonly used to manufacture PFAS. PFAS have been manufactured and used in many industries in the United States and internationally since the 1940s, but were not widely documented in environmental samples until analytical methods became commercially available in the 2000s. Since that time, analytical methods have been continuously developed for different environmental media and PFAS chemicals, and to detect PFAS at lower concentrations. Today, PFAS are detected ubiquitously in the environment, biota, and humans, and in remote areas around the globe (Glüge et al., 2020; ITRC, 2020).

The thousands of chemicals that make up the PFAS family can be divided into two classes: nonpolymers and polymers. Each class may contain subclasses, groups, and subgroups. Figure 1, adapted from ITRC (2020), shows the PFAS classes and groups discussed in this preliminary report. Figure 1 is not an exhaustive list of chemical classes and groups that may be considered PFAS. This preliminary report focuses on nonpolymer PFAS with an emphasis on perfluoroalkyl acids (PFAAs), PFAAs precursors, and replacements for long-chain PFAS that have been or are being phased out.

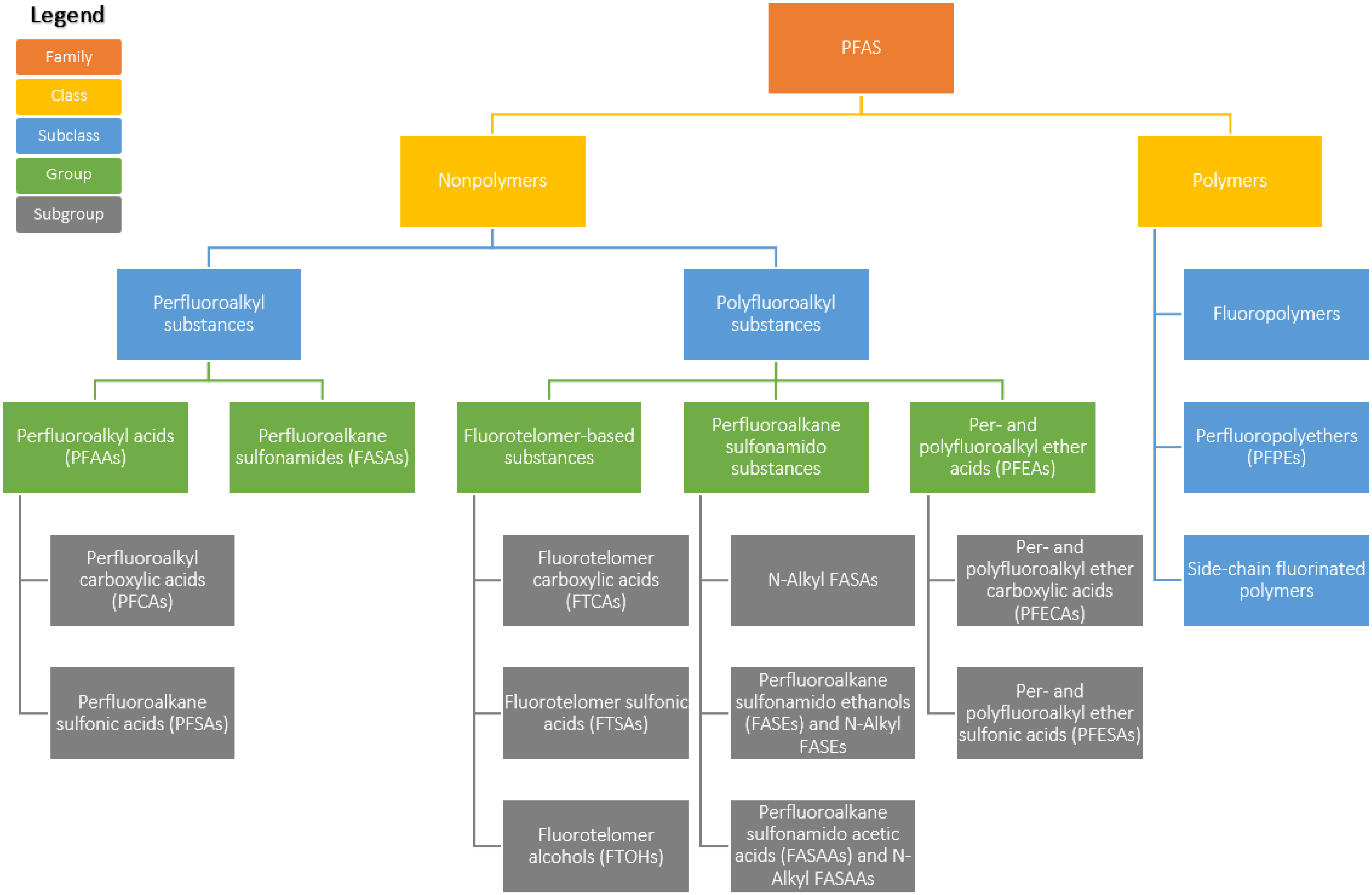

Figure 1. PFAS Classes and Groups Discussed in this Preliminary Report [4]

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[4] Figure 1 was adapted from ITRC (2020) and is not an exhaustive list of chemical classes and groups that may be considered PFAS.

3.1.1 Nonpolymer PFAS

The nonpolymer PFAS class includes two subclasses, perfluoroalkyl substances (fully fluorinated carbon chain) and polyfluoroalkyl substances (partly fluorinated carbon chain), which include various groups and subgroups of chemicals. Table 2, at the end of this section, presents the nonpolymer PFAS for which EPA collected discharge data for this study and are included in EPA's Cross-Agency PFAS Research List (EPA, 2021a).

Perfluoroalkyl Substances

Perfluoroalkyl substances are fully fluorinated alkane molecules consisting of a two-or-more carbon chain (tail) with a charged functional group (head). This preliminary report discusses two groups, PFAAs and perfluoroalkane sulfonamides (FASAs), but others exist and are receiving increasing attention as they are added to commercial laboratory target analyte lists and detected in the environment.

PFAAs are the simplest PFAS molecules and most frequently tested for in the environment. PFAAs do not degrade under ambient environmental conditions and are the terminal products of degradation of more complex PFAS (precursors). Longer chain PFAAs do not naturally degrade into PFAAs with a shorter carbon-fluorine chain length.

PFAAs are divided into two main subgroups: PFCAs and PFSAs. PFCAs may be manufactured using either electrochemical fluorination or fluorotelomerization, while PFSAs are only manufactured using electrochemical fluorination. The PFAAs group includes the two most studied PFAS: PFOA and PFOS. PFOA and PFOS are demonstrated to accumulate and remain in the human body for long periods of time, and to cause adverse health outcomes in animals and humans (EPA, 2016a, 2016b). Figure 2 illustrates the chemical structure of these two chemicals.

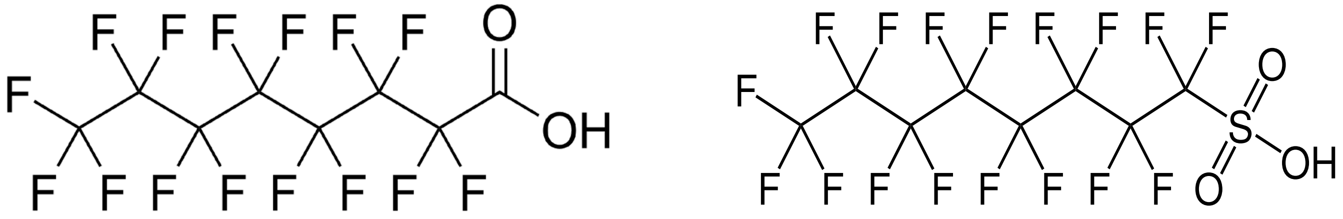

Figure 2. Molecular Structures of PFOA (left) and PFOS (right)

PFAAs are described as long-chain or short-chain PFAS as a shorthand to group PFCAs and PFSAs that may behave similarly in the environment. PFAAs are classified as either long-chain or short-chain depending on the number of carbons covalently bonded to fluorine. Long-chain PFCAs have eight or more carbons (seven or more carbons are perfluorinated) and long-chain PFSAs have six or more carbons (six or more carbons are perfluorinated) (ITRC, 2020). In terms of chemical behavior, PFCAs are more analogous to PFSAs that contain one more carbon than PFSAs that contain the same number of carbons because one carbon in the PFCA molecule is associated with the functional group rather than the fluoroalkyl tail (e.g., the eight carbon PFCA behaves more similar to a seven carbon PFSA than an eight carbon PFSA). Table 2 identifies short-chain PFCAs and PFSAs in blue text, while long-chain PFCAs and PFSAs are designated in red text. EPA notes that other factors besides carbon-fluorine chain length may affect behavior and bioaccumulation potential of PFAS.

FASAs are used as raw material in the electrochemical fluorination process to make perfluoroalkyl sulfonamido substances that are used for surfactants and surface treatments. FASAs may degrade to form PFAAs (ITRC, 2020).

Polyfluoroalkyl Substances

Polyfluoroalkyl substances are distinguished by not being fully fluorinated. Instead, they have a nonfluorine atom (typically hydrogen or oxygen) attached to at least one, but not all, carbon atoms, while two or more of the remaining atoms in the carbon chain tail are fully fluorinated. The nonfluorinated bond in polyfluoroalkyl molecules create a weak point that is susceptible to degradation, thus many of these PFAS have potential to be transformed into PFAAs. This preliminary report discusses three groups of polyfluoroalkyl substances: fluorotelomer-based substances, perfluoroalkane sulfonamido substances, and per- and polyfluoroalkyl ether acids (PFEAs).

Fluorotelomer-based substances are produced by fluorotelomerization and have an x:y naming convention whereby x identifies the number of fully fluorinated carbon atoms and y identifies the number of carbon atoms not fully fluorinated. Fluorotelomer-based substances are potential PFCA precursors but are not observed to transform into PFSAs (NASF, 2019a; Zhang et al., 2016). Three subgroups are discussed in this preliminary report: fluorotelomer carboxylic acids (FTCAs), fluorotelomer sulfonic acids (FTSAs), and fluorotelomer alcohols (FTOHs).

Perfluoroalkane sulfonamido substances have a fully fluorinated tail and also contain one or more methylene (CH_2) groups in the head of the molecule, attached to the sulfonamido spacer. Perfluoroalkane sulfonamido substances are manufactured by electrochemical fluorination and may degrade into PFCAs and PFSAs (ITRC, 2020). Three subgroups are discussed in this preliminary report: N-alkyl FASAs; perfluoroalkane sulfonamido ethanols (FASEs), and perfluoroalkane sulfonamido acetic acids (FASAAs).

PFEAs are manufactured by fluorotelomerization and include per- and polyfluoroalkyl ether carboxylic acids (PFECAs) and per- and polyfluoroalkyl ether sulfonic acids (PFESAs). Certain PFECAs and PFESAs have been developed and are used as replacements for phased out long-chain PFAAs such as PFOA and PFOS. The PFEAs gaining the most attention are hexafluoropropylene oxide dimer acid (HFPO-DA), 4,8-dioxa-3H-perfluorononanoic acid (DONA) and its ammonium salt (ADONA), and chlorinated PFESAs. See Section 3.2 for further discussion of these PFEAs.

Table 2. Nonpolymer PFAS Included in Collected Discharge Data and EPA's Cross-Agency PFAS Research List

Subclass	Group	Subgroup	General Chemical Structure [a] $C_nF_{2n+1}R$, where R =	PFAS Chemicals [a]
Perfluoroalkyl Substances	Perfluoroalkyl acids (PFAAs)	Perfluoroalkyl carboxylic acids (PFCAs) [b c]	-COOH	- Perfluorobutanoic acid (PFBA) (C4) - Perfluoropentanoic acid (PFPeA) (C5) - Perfluorohexanoic acid (PFHxA) (C6) - Perfluoroheptanoic acid (PFHpA) (C7) - Perfluorooctanoic acid (PFOA) (C8) - Ammonium perfluorooctanoate (APFO) (C8) - Perfluorononanoic acid (PFNA) (C9) - Perfluorodecanoic acid (PFDA) (C10) - Perfluoroundecanoic acid (PFUnA) (C11) - Perfluorododecanoic acid (PFDoA) (C12) - Perfluorotridecanoic acid (PFTrA) (C13) - Perfluorotetradecanoic acid (PFTeA) (C14) - Perfluorohexadecanoic acid (PFHxDA) (C16) - Perfluorooctadecanoic acid (PFODA) (C18)
		Perfluoroalkane sulfonic acids (PFSAs) [b c]	$-SO_3H$	- Perfluorobutane sulfonic acid (PFBS) (C4) - Perfluoropentane sulfonic acid (PFPeS) (C5) - Perfluorohexane sulfonic acid (PFHxS) (C6) - Perfluoroheptane sulfonic acid (PFHpS) (C7) - Perfluorooctane sulfonic acid (PFOS) (C8) - Perfluorononane sulfonic acid (PFNS) (C9) - Perfluorodecane sulfonic acid (PFDS) (C10)
	Perfluoroalkane sulfonamides (FASAs) [d]	*Not Applicable*	$-SO_2NH_2$	- Perfluorooctane sulfonamide (PFOSA)
Polyfluoroalkyl Substances	Fluorotelomer-based substances [e]	Fluorotelomer sulfonic acids (FTSAs)	$-CH_2CH_2SO_3H$	- 4:2 fluorotelomer sulfonic acid (4:2 FTSA) - 6:2 fluorotelomer sulfonic acid (6:2 FTSA) - 8:2 fluorotelomer sulfonic acid (8:2 FTSA)
		Fluorotelomer carboxylic acids (FTCAs)	$-CH_2COOH$	- 2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA) - 2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA)
		Fluorotelomer alcohols (FTOHs)	$-CH_2CH_2OH$	- 6:2 fluorotelomer alcohol (6:2 FTOH)
	Perfluoroalkane sulfonamido substances [d]	N-Alkyl FASAs	$-SO_2N(R')$ where $R' = C_mH_{2m+1}$ (m = 1, 2,4)	- N-methyl perfluorooctane sulfonamide (NMePFOSA) - N-ethyl perfluorooctane sulfonamide (NEtPFOSA)

Table 2. Nonpolymer PFAS Included in Collected Discharge Data and EPA's Cross-Agency PFAS Research List

Subclass	Group	Subgroup	General Chemical Structure [a] $C_nF_{2n+1}R$, where R =	PFAS Chemicals [a]
		Perfluoroalkane sulfonamido ethanols (FASEs) and N-Alkyl FASEs	$-SO_2N(R')CH_2CH_2OH$ where $R' = C_mH_{2m+1}$ (m = 0, 1, 2, 4)	- N-methyl perfluorooctane sulfonamido ethanol (NMeFOSE) - N-ethyl perfluorooctane sulfonamido ethanol (NEtFOSE)
		Perfluoroalkane sulfonamido acetic acids (FASAAs) and N-Alkyl FASAAs	$-SO_2N(R')CH_2COOH$ where $R' = C_mH_{2m+1}$ (m = 0, 1, 2, 4)	- N-methyl perfluorooctane sulfonamido acetic acid (NMeFOSAA) - N-ethyl perfluorooctane sulfonamido acetic acid (NEtFOSAA)
	Per- and polyfluoroalkyl ether acids (PFEAs)	Per- and polyfluoroalkyl ether carboxylic acids (PFECAs)	*Varies by Chemical*	- Hexafluoropropylene oxide dimer acid (HFPO-DA) - 4,8-dioxa-3H-perfluorononanoic acid (DONA) - Ammonium 4,8-dioxa-3H-perfluorononanoate (ADONA) - Sodium dodecafluoro-3H-4, 8-dioxanonanoate (NaDONA) - Perfluoromethoxypropyl carboxylic acid (PMPA) - Perfluoro-2-methoxyacetic acid (PFMOAA) - Perfluoro-3,6-dioxaheptanoic acid (NFDHA) - Perfluoro-3,5-dioxahexanoic acid (PFO2HxA) - Perfluoro-3,5,7-trioxaoctanoic acid (PFO3OA) - Perfluoro-3,5,7,9-tetraoxadecanoic acid (PFO4DA) - Perfluoro-3,5,7,9,11-pentaoxadodecanoic acid (PF05DA) - Perfluoro-4-isopropoxybutanoic acid (PFECA-G)
		Per- and polyfluoroalkyl ether sulfonic acids (PFESAs)	*Varies by Chemical*	- 9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS) - Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate (K-9Cl-PF3ONS) - 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) - Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate (K-11Cl-PF3OUdS) - Perfluoro-3,6-dioxa-4-methyl-7-octene-1-sulfonic acid (PFESA-BP1) - Perfluoro-2-{[perfluoro-3-(perfluoroethoxy)-2-propanyl]oxy}ethane sulfonic acid (PFESA-BP2)

Chemical Structure Abbreviations: C – carbon; H – hydrogen; N – nitrogen; O – oxygen; S – sulfur.

a – For purposes of this report, EPA presents all PFAS names and chemical structures as the neutral/acid form. Under typical environmental conditions, many PFAS are present in the anionic form.

b – PFCAs and PFSAs are denoted using the structural shorthand PFXY where: PF = perfluoro, X = length of the carbon chain (e.g., O for octane or 8 carbons), and Y = the functional group (e.g., A for carboxylic acids and S for sulfonic acids) (ITRC, 2020). The number of carbons in the chain is presented in parentheses.

c – The table identifies short-chain PFCAs (≤7 carbons) and short-chain PFSAs (≤5 carbons) in blue text, while long-chain PFCAs (≥8 carbons) and long-chain PFSAs (≥6 carbons) are designated in red text.

d – Potential PFCA and PFSA precursor.

e – Potential PFCA precursor.

3.1.2 Polymer PFAS

Polymer PFAS are large molecules formed by combining many identical smaller molecules (monomers) in a repeating pattern. Nonpolymer PFAS may be used in the manufacture of some polymer PFAS (either as raw materials or processing aids), included in polymer products as impurities, or released during incineration or degradation. This preliminary report discusses three subclasses of polymer PFAS: fluoropolymers, perfluoropolyethers (PFPEs), and side-chain fluorinated polymers.

Fluoropolymers contain a carbon-only polymer backbone with fluorine directly attached to the backbone. They are not typically made from nonpolymer PFAS raw materials; however, nonpolymer PFAS have been used as processing aids in the polymerization of certain fluoropolymers. Certain high-molecular weight fluoropolymers, including polytetrafluoroethylene (PTFE) and ethylene tetrafluoroethylene (ETFE), are chemically and thermally stable, insoluble in water, and less bioavailable. Based on current information, the molecules of these fluoropolymers are believed to be too large to cross cell membranes and are therefore believed to pose less risk to human and ecological health relative to nonpolymer PFAS (Chemours, 2021; Henry et al., 2018).

Perfluoropolyethers (PFPEs) contain carbon and oxygen polymer backbones with fluorine directly attached to the carbon. PFPEs are believed to have thermal and chemical stability and are not typically soluble in water. PFPEs are not made from long-chain PFAAs or their potential precursors, nor are long chain PFAAs involved in their manufacture.

Side-chain fluorinated polymers contain a nonfluorinated polymer backbone from which fluorinated side chains branch. Some may degrade to PFAAs when the point of connection of a fluorinated side-chain to the polymer is broken (OECD, 2013).

3.2 Phase Out and Replacement of Certain Long-Chain PFAS with Short-Chain PFAS

Until recently (early 2000s), industry primarily used long-chain PFAS, such as PFOS and PFOA, in the manufacture of commercial products. Due to evidence of long-term persistence and adverse health outcomes associated with long-chain PFAS, EPA's Office of Chemical Safety and Pollution Prevention, Office of Pollution Prevention and Toxics (EPA OPPT) has taken a range of regulatory actions under the Toxic Substances Control Act (TSCA) to gather health and exposure information on, require testing of, and control PFAS in manufacturing and consumer products. EPA's efforts to address PFAS through TSCA include, but are not limited to, the following:

- In 2000, EPA worked with the 3M Company (3M) to support the company's voluntary phase out and elimination of PFOA, PFOS, and other specific long-chain PFAAs from production and use. 3M reported that it had completed most of the phase out by 2002, with full completion by 2008.
- In 2006, EPA launched the PFOA Stewardship Program which resulted in the voluntary phase out of long-chain PFCAs and their precursors (i.e., PFOA, higher homologues of PFOA, and their precursors) by eight major chemical manufacturers and processors by year-end 2015. Companies participating in the PFOA Stewardship Program were Arkema, Asahi Glass Company (AGC), Ciba/BASF Corporation, Clariant Corporation, Daikin Industries (Daikin), 3M/Dyneon, DuPont du Nemours (DuPont), and Solvay (formerly, Solvay Solexis).
- Between 2002 and 2020, EPA issued Significant New Use Rules (SNURs) to require manufacturers (including importers) and processors of certain long-chain PFAS to notify EPA at least 90 days before starting or resuming significant new uses of these chemicals. These SNURs prohibit companies from manufacturing, importing, or using certain long-chain PFAS in the United States without prior EPA review and approval.
- EPA's July 2020 SNUR closed an important loophole that previously allowed products containing certain PFAS that have been phased out in the United States to be imported into the nation. The SNUR leveled the playing field for companies that had already voluntarily phased-out the use of long-chain PFAS under EPA's PFOA Stewardship Program by preventing new uses of these phased-out chemicals.

Although manufacture and import of certain long-chain PFAS and precursors effectively ceased as result of EPA's actions under TSCA, products containing these chemicals that were manufactured or imported before 2020 may still be in use. While manufacture of long-chain PFAS is restricted in the United States, Europe, and Japan, their manufacture continues in China, India, Russia, and other countries.

EPA's TSCA Chemical Substance Inventory lists over 1,000 PFAS, approximately half of which are known to be commercially active within the United States in the last decade. As of February 2020, EPA reviewed more than 300 of the commercially active PFAS under the New Chemicals Program, and regulated about 200 PFAS with consent orders and/or new chemical SNURs (EPA, 2020b).

The phase out and increasing concerns regarding persistence, bioaccumulation, and health effects of certain long-chain PFAAs has led many manufacturers to develop replacement technologies. Manufacturers have developed alternative processes and chemistries to substitute for these long-chain PFAS, including nonfluorinated chemicals, short-chain PFCAs and PFSAs, and PFAS chemistries that do not degrade to long-chain PFAAs. The list below presents several examples of alternative short-chain PFAS that manufacturers have developed and used to replace long-chain PFAS:

- HFPO-DA (one chemical used in the DuPont/Chemours GenX technology) and ADONA (one chemical used in a 3M technology) are replacements for PFOA as a polymerization aid in the production of fluoropolymers and PFPEs.[5] Transition to the GenX- and ADONA-based processing aid technologies began in 2009 as part of industry's commitment under the PFOA Stewardship Program to work toward the elimination of certain long-chain PFAAs and precursors from emissions and products by 2015.
- Short-chain PFCAs (PFBA, PFPeA, PFHxA, PFHpA) and short-chain PFSAs (PFBS, PFPeS) are replacements for PFOA and PFOS in chemical coatings, additives, and surface treatments. For example, PFBS (a four-carbon homologue of the eight-carbon PFOS) replaced PFOS in 3M's Scotchgard™ stain repellent.
- Fluorotelomer-based substances with six or less fully fluorinated carbons (e.g., 6:2 FTSA, 6:2 FTOH) are replacements for long-chain PFAAs and their precursors in aqueous film-forming foam (AFFF) and food contact materials requiring water and oil resistance or nonstick properties.
- Fluorotelomer-based substances with six or less fully fluorinated carbons (e.g., 6:2 FTSA) and chlorinated PFESAs (e.g., 9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid and 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid) are replacements of PFOS used as metal plating mist and fume suppressants. The substances 9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS), 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS), and their potassium salts are also known as "F-53B" chemicals.[6]

Many short-chain PFAS-based replacement chemicals are structurally similar to their predecessors and manufactured by the same companies. Replacement short-chain PFAS may be used in higher quantities than long-chain PFAS to achieve the same desired properties (Blepp et al., 2017; Blum et al., 2015).

Chemical property information is publicly available for only a few alternative PFAS chemistries; very few health, toxicity, and hazard assessments have been performed for these chemicals (Blum et al., 2015). As part of this study, EPA conducted a preliminary review of four short-chain PFAS adopted by industry to replace PFOA and PFOS. Section 3.4 summarizes current information and data on advisory standards, toxicity, bioaccumulation and persistence, and degradation of PFOA, PFOS, PFBS, 6:2 FTSA, 6:2 FTOH, and HFPO-DA.

3.3 Environmental Fate and Transport of PFAS

Short- and long-chain PFAS enter the environment through manufacturing and during use and disposal of consumer items. According to ATSDR, PFAS have been found worldwide in surface water, groundwater, finished drinking water, rainwater, soils, sediments, ice caps, outdoor and indoor air, plants, animal tissue, and human blood serum. The highest environmental concentrations of long- and short-chain PFAS are found in surface water, groundwater, soils, and sediments around facilities that have produced or used PFAS (ATSDR, 2021). According to the Association of State and Territorial Solid Waste Management Officials (ASTSWMO), fresh waters near

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[5] The Chemours Company (Chemours) is a July 2015 spinoff of the former DuPont performance chemicals business unit. GenX is the trade name for a fluoropolymer processing aid technology that is associated with two chemicals, HFPO-DA and its ammonium salt, also referred to as "GenX" chemicals.

[6] The trade name "F-53B" refers specifically to a single chemical, 9Cl-PF3ONS, but the name is often used to encompass 9Cl-PF3ONS, minor impurities such as the homologue 11Cl-PF3OUdS, and their potassium salts. The major and minor components of F-53B are sometimes referred to as "F-53B major" (9Cl-PF3ONS) and "F-53B minor" (11Cl-PF3OUdS).

industrial sites have documented PFAS concentrations ranging up to 1,000 nanograms per liter (ng/L). Oceanic concentrations of PFAS are several orders of magnitude lower, ranging from 0.01 to 0.1 ng/L (ASTSWMO, 2015).

EPA used the Unregulated Contaminants Monitoring Rule 3 (UCMR3) to collect data for contaminants suspected to be present in drinking water, but that do not have health-based standards set under the Safe Drinking Water Act. As part of UCMR3, EPA sampled drinking water for six PFAS (PFOS, PFOA, PFNA, PFHxS, PFHpA, and PFBS) between 2013 and 2015 (EPA, 2017a). EPA's UCMR3 monitoring indicated that public water systems in 33 states serving 16.5 million residents had detectable levels of long- and short-chain PFAS. Sixty-six public water systems serving more than 6 million people were found to have at least one sample above 70 ng/L, EPA's lifetime health advisory (LHA) value for the sum of PFOA and PFOS in drinking water (Hu et al., 2016). See Section 3.4 for more information regarding EPA's LHA values for PFAS.

Owing to their chemical and thermal stability, some long- and short-chain PFAS can withstand heat, acids, bases, reducing agents, and oxidants and, as a result, are not readily degradable by most natural processes. As discussed in Section 3.2, manufacturers that have phased out certain long-chain PFAS have replaced them with alternative PFAS chemistries, including short-chain PFCAs and PFSAs, and PFAS that do not degrade to long-chain PFAAs. Some short-chain PFAS are as persistent in the environment as their long-chain homologues (Wang et al., 2013) although other short-chains degrade much faster.

3.4 PFAS Exposure and Health Effects

This section summarizes information on exposure and adverse human health effects of certain PFAS. Research in this field is ongoing and information presented in this section represents the current state of knowledge based on EPA's review of technical literature, EPA toxicity assessments, ATSDR toxicological profiles, the United States Department of Commerce National Oceanic and Atmospheric Administration (NOAA) Chemical Aquatic Fate and Effects (CAFE) database (NOAA, 2019), and the United States Department of Health and Human Services National Institutes of Health (NIH) Toxicology Data Network (TOXNET) (NIH, 2019).

There are a variety of ways that individuals may be exposed to PFAS. Known exposure routes for PFAS include (ATSDR, 2021; EPA, 2016a, 2016b):

- Consumption of drinking water from contaminated public water systems or private wells.
- Consumption of contaminated fish.
- Consumption of crops grown in contaminated soils, particularly in agricultural areas that receive amendments of biosolids from POTWs.
- In utero exposure.
- Consumption of contaminated breast milk by infants.
- Inhalation and ingestion of contaminated indoor dust.
- Direct contact with products treated with PFAS, such as food papers/packaging and treated carpets.

For the general population, contaminated drinking water and food are the most frequently documented routes of exposure to long- and short-chain PFAS. There is evidence that exposure to certain PFAS can lead to adverse health outcomes in animals and humans. If animals or humans ingest PFAS-contaminated food or water, the PFAS are absorbed, and can accumulate in the body. Certain PFAS, such as PFOA and PFOS, may stay in the human body for longer than 10 years. As individuals become exposed to PFAS from different sources over time, the level of PFAS in their bodies may increase to the point where they suffer from adverse health effects (ATSDR, 2021).

In May 2016, EPA established an LHA value at 70 ng/L for the sum of PFOA and PFOS to protect the public from these potential adverse health effects resulting from exposure to PFOA and PFOS in drinking water. EPA's LHA values are based on the best available peer-reviewed studies of the effects of PFOA and PFOS on laboratory animals (rats and mice) and were also informed by epidemiological studies of human populations that have been exposed to PFAS (EPA, 2021d). EPA's LHA values are not legally enforceable; they provide technical information on drinking water contaminants to federal, state, and local officials, and managers of public or community water systems to assist them with protecting public health (EPA, 2018b). In 2021, EPA initiated a proposal to establish enforceable National Primary Drinking Water Regulations for PFOS and PFOA. This process will include evaluating the need for enforceable maximum contaminant levels (MCLs) for PFOA and PFOS.

As discussed in Section 3.2, industry has effectively ceased manufacturing and using certain long-chain PFAS and is substituting with short-chain PFAS. Less information about the toxicity and bioaccumulation of short-chain PFAS is available compared to long-chain PFAS. EPA reviewed information on the toxicity, bioaccumulation, and degradation potential for four short-chain PFAS (6:2 FTSA, 6:2 FTOH, PFBS, and HFPO-DA) adopted by industry to replace long-chain PFAS. Table 3 summarizes available information for these four short-chain PFAS, as well as for PFOA and PFOS for comparison. EPA notes that complete toxicity, bioaccumulation, and human half-life information is not available for all substances; EPA presents draft values where final values are not yet available (e.g., HFPO-DA toxicity values). See the *Short-Chain PFAS Review: Fact Sheet for 6:2 FTSA, 6:2 FTOH, PFBS, and HFPO-DA* for additional information (ERG, 2021d).

The informational categories in the first column of Table 3 are defined below:

- **Current Industrial Applications.** Describes use of the PFAS by the five point source categories assessed.
- **Chronic Reference Dose (RfD).** An estimate of the daily oral exposure for a chronic duration to the human population that is likely to be without an appreciable risk of deleterious effects during a lifetime. Generally used in EPA's noncancer health assessments and expressed in weight of substance per unit weight of organism per day (e.g., mg/kg-day). The lower the chronic RfD, the more toxic the substance.
- **Oral Median Lethal Dose (LD50).** A statistically derived single dose that can be expected to cause death in 50 percent of the test animals when administered by the route indicated (oral, dermal, inhalation). It is expressed as a weight of substance per unit weight of animal (e.g., mg/kg). The lower the LD50, the more acutely toxic the substance.
- **Toxicity Effects.** Describes types of adverse health effects observed in humans or test animals following exposure to the substance.
- **Bioaccumulation Factors (BAF).** The ratio of the concentration of a contaminant in an organism to the concentration in the ambient environment at steady state, where the organism can take in the contaminant through ingestion with its food as well as through direct contact. EPA OPPT characterizes a chemical as bioaccumulative if it has a bioconcentration factor (BCF) or BAF greater than or equal to 1,000. EPA OPPT characterizes a chemical as very bioaccumulative if it has a BCF or BAF greater than or equal to 5,000 (EPA, 2017b).
- **Human Half-Life.** The time required for human biological processes to naturally eliminate half the amount of a substance initially measured in blood serum.
- **Degradation Products.** Terminal products observed following degradation of the organic substance.

While information on human health effects of the four short-chain PFAS is limited, studies current when this preliminary report was written suggest 6:2 FTSA, 6:2 FTOH, PFBS, and HFPO-DA demonstrate less risk to overall human health and less potential for bioaccumulation, relative to PFOA and PFOS. However, EPA has documented these short-chain PFAS are present in industrial discharges, are environmentally persistent, and do demonstrate potential for adverse impacts to ecological and human health receptors. Additional findings from EPA's preliminary review of these four short-chain PFAS, PFOA, and PFOS are listed below:

- PFBS, PFOA, and PFOS have lower reported minimum chronic RfD and oral LD50 values than 6:2 FTSA, 6:2 FTOH, and HFPO-DA. This suggests PFBS, PFOA, and PFOS may have higher chronic and acute toxicity.
- PFOA and PFOS meet EPA OPPT's criteria for designation as "very bioaccumulative" and "bioaccumulative," respectively. The four short-chain PFAS do not meet these criteria.
- Human half-life identified for PFBS is estimated as 43.8 days as compared to more than 2 years for PFOA and PFOS, which suggests PFBS is less bioaccumulative.
- Fluorotelomers readily degrade and transform through multiple complex mechanisms. Terminal end products for 6:2 FTSA and 6:2 FTOH include short-chain PFCAs and FTCAs (do not degrade to PFOA or PFOS); some of these degradation products may be environmentally and biologically persistent (Kabadi et al., 2018, 2020).
- Available information suggests PFBS and HFPO-DA are stable under ambient environmental conditions.

Table 3. Summary of Draft and Final Health Effects Information for Short-Chain PFAS, PFOA, and PFOS

	6:2 FTSA	6:2 FTOH	PFBS	HFPO-DA	PFOA	PFOS
Current Industrial Applications	Modern AFFF, food contact substances, and metal finishing mist and fume suppressants	Modern AFFF, food contact substances, and intermediate in chemical/resin manufacturing	Chemical coatings, additives and surface treatments (e.g., 3M Scotchgard™)	Polymerization aid in production of fluoropolymers and PFPEs	Manufacture, use, and import restricted in the United States	Manufacture, use, and import restricted in the United States
Chronic RfD (mg/kg-day)	*None identified*	*None identified*	0.0003	0.00008 [a]	0.00002	0.00002
Oral LD50 (mg/kg)	300 – 2,000	1,750 – 2,000	430	1,730 – 1,750 [b]	430 – 680	251 - 579
Toxicity Effects	Skin irritation, kidney and liver effects	Kidney, liver, immune system, and developmental effects	Thyroid, liver, kidney, developmental, and reproductive effects	Liver, kidney, immune system, hematological, developmental, and carcinogenic effects	Liver, kidney, reproductive, developmental, and carcinogenic effects	Liver, kidney, thyroid, immune system, developmental, cardiovascular, and carcinogenic effects
Bioaccumulation Factors (BAF)	*None identified*	*None identified*	< 10	< 10 (tissue value) [a]	7,670	1,900
Human Half-Life	*None identified*	*None identified*	43.8 days	*None identified*	2.1 – 10.1 years	3.3 – 27 years
Degradation Products	5:3 FTCA, PFPeA, PFHxA	5:3 FTCA, PFPeA, PFHxA	Environmentally stable (no natural degradation)	Environmentally stable (no natural degradation)	Environmentally stable (no natural degradation)	Environmentally stable (no natural degradation)
References	NASF, 2019a EPA 2021v	Kabadi et al., 2018, 2020 Rice et al., 2020 EPA, 2021v	AECOM, 2019 ASTDR, 2021 EPA, 2021u, 2021v	Dupont, 2008 EPA, 2018c	ATSDR, 2021 EPA, 2016a, 2018a, 2021v	ATSDR, 2021 EPA, 2016b, 2018a, 2021v

a – Draft values from EPA's 2018 draft *Human Health Toxicity Values for Hexafluoropropylene Oxide (HFPO) Dimer Acid and Its Ammonium Salt (CASRN 13252-13-6 and CASRN 62037-80-3)* (EPA, 2018c). Values subject to change; final GenX values anticipated in late 2021.

b – The oral median lethal doses (LD50s) were 1,730 mg/kg and 1,750 mg/kg in male rats and female rats, respectively. In these rat and mouse studies, animals received a single dose in the dose range of 175–5,000 mg/kg HFPO-DA and were assessed for effects for 14 days (DuPont, 2008; EPA, 2018c).

4. Data Collection Activities

This section describes the data sources EPA collected and evaluated as part of the Multi-Industry PFAS Study, provides information on data quality, and describes how EPA handles and safeguards confidential business information (CBI).

4.1 Data Sources and Quality

EPA gathered available data and reached out to stakeholders to identify facilities producing or using PFAS, determine wastewater characteristics, estimate PFAS in wastewater discharges, and identify effective PFAS control practices and treatment technologies. Table 4 describes each data source EPA consulted as part of the study and summarizes how each was used. EPA considered the accuracy, reliability, and representativeness of the data sources listed in Table 4 to assess their usability for the Multi-Industry PFAS Study, as described below.

Accuracy. EPA assumed that data and information contained in supporting government publications or databases, peer-reviewed journal articles, and other technical literature are sufficiently accurate to support the general characterization of industries, sources, wastewater discharges, and treatment associated with PFAS, as well as human health impacts and environmental fate, transport, and exposure pathways of PFAS. EPA considered the data and information obtained from direct correspondence with individual companies, industry trade associations, and state government representatives and regulators as sufficiently accurate to characterize and quantify specific PFAS wastewater discharges or related process operations from individual facilities.

Reliability. EPA used the following criteria to evaluate the reliability of available data and other information collected and used in its analyses:

- The work is clearly written, so that all assumptions and methodologies can be identified.
- The variability and uncertainty (quantitative and qualitative) of the information, or the procedures, measures, methods, or models used to compile the information, are evaluated and characterized.
- The assumptions and methods are consistently applied throughout the analysis, as reported in the source.
- Wastestreams, analytes, units, and analytical limitations (when appropriate) are clearly characterized.
- The contact is reputable and has knowledge of the industry, facility, processes, and/or wastestreams of interest.

EPA considered data sources that met these criteria sufficiently reliable to characterize and understand industries, sources, and wastewater discharges associated with PFAS.

Representativeness. EPA evaluated whether data and information were characteristic of PFAS discharges and impacts across industries or sources and were relevant to and representative of typical operations relevant to PFAS.

EPA considered data sources that met these criteria of being sufficiently accurate, reliable, and representative to characterize industries, sources, and wastewater discharges and treatment associated with PFAS, as well as human health impacts and environmental fate, transport, and exposure pathways of PFAS.

Table 4. Multi-Industry PFAS Study Data Sources

Data Source	Description	Use in Study
EPA Data Sets and Coordination		
2016 Chemical Data Reporting (CDR) Database	The CDR rule, under TSCA, requires manufacturers (including importers) to provide EPA with information on the production and use of chemicals in commerce. The information is collected every four years from manufacturers and importers of certain chemicals when production volumes exceed specified thresholds for a specific reporting year. EPA accessed CBI CDR data reported in 2016 for chemicals listed in EPA's Cross-Agency PFAS Research List, including facility names and locations and volumes of chemicals for production, import, and use of PFAS. The CDR information reported in 2016 reflects the most recent data set available and reflect production volumes from 2012 through 2015 (EPA, 2021b).	Estimate PFAS production volumes, identify companies and facilities manufacturing or importing PFAS, and identify industrial and commercial uses of PFAS.
2019 and 2020 Discharge Monitoring Reports (DMR)	EPA downloaded DMR data for PFAS from the Integrated Compliance Information System National Pollutant Discharge Elimination System for 2019 and 2020 using the online Water Pollutant Loading Tool. The data include pollutant discharge information (e.g., types and concentrations) and discharge flow rate data for direct dischargers with PFAS effluent limitations or monitoring requirements in their NPDES permits (EPA, 2020c, 2021c).	Evaluate wastewater characteristics, estimate facility and industry PFAS concentrations, and identify facilities with NPDES permit requirements for PFAS.
Industrial Wastewater Treatment Technology (IWTT) Database	EPA's IWTT database contains information on treatment technology advances identified through EPA's Annual Reviews. As part of its screening of industrial wastewater discharges, EPA reviews literature regarding the performance of new and improved industrial wastewater treatment technologies and enters the data into its IWTT database (EPA, 2021e).	Identify technologies used to remove PFAS from wastewaters.
Drinking Water Treatability Database (DWTD)	EPA's DWTD is a compilation of research articles on contaminants found in drinking water sources and treatment technologies for drinking water treatment plants. The DWTD includes PFAS removal performance data for an assortment of treatment technologies and 37 PFAS (EPA, 2021f).	Identify PFAS treatment technologies and assess PFAS removal performance.
EPA's Office of Enforcement and Compliance Assurance	EPA met with EPA's Office of Enforcement and Compliance Assurance and received industry data submittals, reports, and sampling data for three companies that manufacture or process PFAS (EPA, 2020d).	Identify unit operations, PFAS effluent limitations or monitoring requirements, and wastewater treatment in place. Used to determine wastewater characteristics and estimate PFAS in wastewater discharges.
EPA's Office of Chemical Safety and Pollution Prevention, Office of Pollution Prevention and Toxics (EPA OPPT)	EPA met and coordinated with EPA's OPPT to discuss and collect PFAS data available in the TSCA Chemical Substance Inventory and 2016 CDR database (EPA, 2020f, 2021b).	Collect information on PFAS discussed in this report. Identify commercially active PFAS and companies and facilities manufacturing or importing these PFAS, as well as associated production volumes.

Table 4. Multi-Industry PFAS Study Data Sources

Data Source	Description	Use in Study
Previous Rulemaking Materials and EPA Publications	EPA obtained supporting documentation from previous EPA actions and rulemakings associated with PFAS and the industrial categories included in the Multi-Industry PFAS Study. These development documents contain findings, conclusions, and data on industry profiles, PFAS use and restrictions, and PFAS control and treatment technologies. Materials collected included, but are not limited to, existing ELGs, National Emission Standards for Hazardous Air Pollutants (NESHAP), SNURs, PFOA Stewardship Program status reports, EPA PFAS toxicology assessments, EPA's Interim Guidance on PFAS Disposal and Destruction, and technology technical briefs prepared by EPA's PFAS Innovative Treatment Team (PITT).	Background information on the population and processes of the five industrial point source categories and on the impacts of current government programs and regulations related to PFAS.
Information from Other Federal Agencies		
United States Department of Transportation, Federal Aviation Administration (FAA)	EPA met with the FAA to discuss PFAS-containing AFFF use at airports. EPA collected materials related to the military specifications for AFFF used at commercial airports and FAA guidance for AFFF use and control (ERG, 2020a).	Background on PFAS use by commercial airports and FAA activities to control PFAS.
United States Department of Health and Human Services, Food and Drug Administration (FDA)	EPA collected and reviewed FDA-funded studies and food contact substance notifications (FCNs) for PFAS approved for used in food contact materials (FDA, 2020, 2021; Kabadi et al., 2018, 2020; Rice et al., 2020).	Background information on the use of PFAS in food-contact materials.
Information from States and Regions		
National Pollutant Discharge Elimination System (NPDES) Permits, Permit Applications, and Fact Sheets	The CWA requires direct dischargers to control their discharges according to limitations, monitoring, and requirements included in NPDES permits. EPA obtained and reviewed copies of NPDES permits and, where available, accompanying permit applications and fact sheets for facilities discharging PFAS in the five industrial point source categories. Information contained in permit materials includes onsite wastewater treatment processes, outfall descriptions, and destinations of wastewater discharges.	Identify unit operations, PFAS effluent limitations or monitoring requirements, and wastewater treatment in place.
Michigan Department of Environment, Great Lakes, and Energy (MI EGLE)	EPA met with MI EGLE and local wastewater authorities on several occasions between 2019 and 2021. EPA received or downloaded multiple documents and data sets including PFAS survey materials, reports summarizing MI EGLE's efforts to identify sources of and address PFAS, and effluent analytical data for direct and indirect discharges (ERG, 2019a, 2019b, 2019c; MI EGLE, 2020a, 2020b, 2020c, 2020d; MI GLWA, 2019).	Identify unit operations, PFAS effluent limitations or monitoring requirements, and wastewater treatment in place. Used to determine wastewater characteristics and estimate PFAS in wastewater discharges.
EPA Region 3	On February 26, 2020, EPA Region 3 submitted a field sampling investigation report containing effluent data for a Sartomer (a division of Arkema) PFAS production plant in West Chester, Pennsylvania. The sampling was performed to confirm PFAS levels in a surface water and determine if the Sartomer facility was the source of PFAS (EPA Region 3, 2019).	Determine wastewater characteristics and estimate PFAS in wastewater discharges.

Table 4. Multi-Industry PFAS Study Data Sources

Data Source	Description	Use in Study
New Jersey Department of Environmental Protection (NJ DEP)	EPA met with NJ DEP and received effluent analytical data and/or the NPDES permit materials for two facilities that manufacture or process PFAS (NJ DEP, 2015, 2018, 2020).	Identify unit operations, PFAS effluent limitations or monitoring requirements, and wastewater treatment in place. Used to determine wastewater characteristics and estimate PFAS in wastewater discharges.
Wisconsin Department of Natural Resources (WI DNR)	EPA met with the WI DNR to discuss sources of PFAS in the state of Wisconsin (ERG, 2020b).	Identify companies and facilities discharging PFAS in Wisconsin.
Information from Industry		
American Chemistry Council (ACC) and ACC FluoroCouncil	EPA met with the ACC and members of the ACC FluoroCouncil and collected materials relevant to the production and use of PFAS in the United States as well as assessments of certain PFAS (ERG, 2019d).	Background on PFAS manufacture and processing in the United States, to identify PFAS currently in the domestic market, and to identify specific companies and facilities that manufacture or process PFAS.
The Chemours Company (Chemours)	EPA met with Chemours (a July 2015 spinoff of the former DuPont performance chemicals business unit) and received materials from the company including presentations, technical papers, materials associated with a consent order for one Chemours PFAS manufacturing facility, and effluent analytical data and the NPDES permit for another Chemours PFAS manufacturing facility (ERG, 2019e; Chemours, 2020a; NJ DEP, 2018).	Identify unit operations, PFAS effluent limitations or monitoring requirements, and wastewater treatment in place. Used to determine wastewater characteristics and estimate PFAS in wastewater discharges.
3M	EPA met with 3M and received materials from the company, including an industry report on fluorochemical production, procedures for a direct injection analytical method for PFAS, wastewater treatment diagrams, NPDES permits, and effluent analytical data for three 3M facilities that manufacture or process PFAS (ERG, 2019f; 3M, 2020a, 2020b).	Identify unit operations, PFAS effluent limitations or monitoring requirements, and wastewater treatment in place. Used to determine wastewater characteristics and estimate PFAS in wastewater discharges.
Daikin	EPA met with Daikin America, a subsidiary of Daikin, to discuss manufacture, formulation, and discharge of PFAS (ERG, 2019g).	Identify unit operations, PFAS effluent limitations or monitoring requirements, and wastewater treatment in place.
AGC	EPA met with AGC Chemicals Americas, a wholly owned subsidiary of AGC, to discuss manufacture, formulation, and discharge of PFAS. AGC Chemicals Americas stated that they do not operate any facilities domestically that manufacture PFAS; however, they do have at least one facility that processes PFAS (ERG, 2019h).	Identify companies and facilities manufacturing or importing PFAS.
National Association for Surface Finishing (NASF)	EPA met with NASF and collected materials related to the use and toxicity of PFAS in metal finishing operations (ERG, 2020c; NASF, 2019a, 2019b, 2019c).	Background on PFAS use by the metal finishing industry and to assess toxicity, bioaccumulation, and persistence of PFAS.

Table 4. Multi-Industry PFAS Study Data Sources

Data Source	Description	Use in Study
American Forest and Paper Association (AF&PA)	EPA met with the AF&PA and several member companies to discuss sources and classifications of PFAS, including AF&PA member companies that use PFAS and the potential to discharge PFAS into the environment. AF&PA submitted information on the use and discharge of PFAS in four subsequent data submissions and facilitated outreach with specific member companies (ERG, 2020d; AF&PA, 2020a, 2020b, 2020c, 2020d).	Background on PFAS use by the pulp, paper, and paperboard industry, to identify unit operations and products associated with PFAS, and determine wastewater characteristics.
Ahlstrom-Munksjö USA Inc. (Ahlstrom-Munksjö)	EPA met with Ahlstrom-Munksjö to discuss use and discharge of PFAS by the company's pulp, paper, and paperboard facilities. Ahlstrom-Munksjö currently uses PFAS in the manufacture of food contact paper and packaging for five facilities in the United States. Ahlstrom-Munksjö is transitioning all production to PFAS-free formulations in the next few years (EPA, 2021g).	Background on PFAS use by the pulp, paper, and paperboard industry and to identify facilities using PFAS.
Georgia Pacific, LLC (Georgia-Pacific)	EPA met with Georgia-Pacific to discuss use and discharge of PFAS by the company's pulp, paper, and paperboard facilities. Georgia-Pacific discontinued application of PFAS to paper and packaging products more than a decade ago. In 2021, Georgia-Pacific completely discontinued purchase, conversion, and distribution of PFAS-treated paper and packaging products (EPA, 2021h).	Background on PFAS use by the pulp, paper, and paperboard industry and to identify facilities using PFAS.
Graphic Packaging International	EPA met with Graphic Packaging International to discuss use and discharge of PFAS by the company's pulp, paper, and paperboard facilities. Graphic Packaging International currently uses PFAS in the manufacture of food contact paper and packaging at a single facility in the United States, but will discontinue PFAS use at this facility by end of 2021 (EPA, 2021i).	Background on PFAS use by the pulp, paper, and paperboard industry and to identify facilities using PFAS.
WestRock Company	EPA met with WestRock Company to discuss use and discharge of PFAS by the company's pulp, paper, and paperboard facilities. WestRock Company discontinued application of PFAS to paper and packaging products across all United States mills in 2020 (EPA, 2021j).	Background on PFAS use by the pulp, paper, and paperboard industry and to identify facilities using PFAS.
Sappi North America, Inc. (Sappi)	EPA met with Sappi to discuss use and discharge of PFAS by the company's pulp, paper, and paperboard facilities. Sappi currently uses PFAS in the manufacture of food contact packaging at a single facility in the United States, but will discontinue use at this facility by 2024 (EPA, 2021m).	Background on PFAS use in the pulp, paper, and paperboard industry and to identify facilities using PFAS.
Domtar Corporation	Domtar Corporation submitted a letter to EPA with information on the use of PFAS by the company's pulp, paper, and paperboard facilities. Domtar Corporation notified EPA that one mill was using PFAS in the manufacture of food contact paper and packaging in 2021; however, this mill has closed and none of the remaining United States facilities use PFAS (Domtar Corporation, 2021).	Identify companies and facilities manufacturing or importing PFAS.

Table 4. Multi-Industry PFAS Study Data Sources

Data Source	Description	Use in Study
International Paper Company	Domtar Corporation submitted a letter to EPA confirming the company does not use PFAS in manufacturing products at United States mills and does not sell or import into the United States products with PFAS intentionally added (International Paper Company, 2021).	Identify companies and facilities manufacturing or importing PFAS.
Airport Council International – North America	EPA met with the Airport Council International – North America to discuss use and composition of PFAS-containing AFFF and efforts to reduce/control releases (ERG, 2020e).	Background on AFFF use at commercial airports and potential release of PFAS.
National Association of Clean Water Agencies (NACWA)	EPA met with the NACWA to discuss industrial producers and users of PFAS, treatment technologies, and POTW concerns related to PFAS (ERG, 2019i).	Background on organizations or companies collecting PFAS effluent and treatment technology data.
Other Sources		
Scientific and Academic Literature	As part of targeted literature reviews, EPA collected peer-reviewed and technical literature relevant to PFAS manufacture, release, sampling/analysis, treatment, toxicity, degradation, bioaccumulation, persistence, and other topics.	Obtain information on the five industrial point source categories, PFAS manufacture and use, and PFAS characteristics.
Conferences/Webinars	EPA participated in and obtained information from multiple conferences and virtual webinars relevant to PFAS manufacture, release, sampling/analysis, treatment, toxicity, degradation, bioaccumulation, persistence, and other topics.	Obtain information on the five industrial point source categories, PFAS manufacture and use, and PFAS characteristics.

4.2 Protection of Confidential Business Information

Certain data in the study record have been claimed as CBI or enforcement sensitive materials. As required by federal regulations at 40 CFR Part 2, EPA has taken precautions to prevent the inadvertent disclosure of this CBI. The agency has withheld CBI from the public docket in the Federal Docket Management System and available on www.regulations.gov. In addition, EPA has found it necessary to withhold from disclosure some data not claimed as CBI because the release of these data could indirectly reveal CBI. Where necessary, EPA has aggregated certain data in the public docket, masked plant identities, or used other strategies to prevent the disclosure of CBI. The agency's approach to protecting CBI ensures that the data in the public docket explain the basis for the study and provide the opportunity for public comment without compromising data confidentiality.

5. Review of the OCPSF Point Source Category

This section describes the OCPSF point source category, information and data EPA collected on its production and use of PFAS, and EPA's estimates of types and concentrations of PFAS discharged by OCPSF facilities that manufacture or formulate PFAS. EPA focused on PFAS manufacturers and PFAS formulators for its review of the OCPSF point source category in this study. EPA collected and reviewed information on PFAS manufacture, use, and discharge by this subset of OCPSF facilities from the sources below.

- Outreach with chemical manufacturers, industry trade associations, and state and local wastewater authorities.
- The agency's Chemical Data Reporting (CDR) database.
- Discharge Monitoring Report (DMR) data available in EPA's Water Pollutant Loading Tool.
- Reports and data collected from industry and state and local wastewater authorities.
- Wastewater discharge permits.
- Industry submissions associated with PFAS consent orders and enforcement activities.
- Publicly available technical literature.

5.1 Industry Description, Manufacture, and Use of PFAS

The OCPSF point source category includes more than 1,000 facilities that manufacture certain organic chemicals, plastics, and synthetic fibers and related products. The industry is diverse with many complex unit operations and specialized manufacturing facilities that process raw materials into thousands of different products. OCPSF facilities operate continuous processes as well as batch operations with a wide range of production volumes.

EPA promulgated the OCPSF Effluent Guidelines (40 CFR Part 414) in 1987, with technical amendments in 1989, 1990, 1992, and 1993. The OCPSF ELGs apply to process wastewater discharges resulting from the manufacture of seven products or product groups (40 CFR Part 414 Subparts B to H) at facilities included within specified United States Department of Commerce Bureau of the Census Standard Industrial Classification system (SIC) groups. Table 5 summarizes the manufactured products and SIC groups applicable to each subpart. The OCPSF ELGs apply to facilities that manufacture PFAS and may apply to facilities that use PFAS in production of applicable products or product groups; however, these regulations do not establish effluent limitations or pretreatment standards for any PFAS. See EPA's *Product and Product Group Discharges Subject to Effluent Limitations and Standards for the OCPSF Point Source Category – 40 CFR 414* report for additional information on applicability, effluent limitations and pretreatment standards, and wastewaters subject to the OCPSF ELGs (EPA, 2005).

Table 5. Applicability of 40 CFR Part 414 Subparts to Manufacture of Products and Product Groups

40 CFR Part 414 Subpart	Manufactured Products and Applicable SIC Groups [a]
B – Rayon Fibers	Applies only to cellulosic manmade fiber (Rayon) manufactured by the Viscose® process, generally classified and reported under SIC group 2823.
C – Other Fibers	Applies to all other synthetic fibers (except Rayon) generally classified and reported under SIC groups 2823 or 2824.
D – Thermoplastic Resins	Applies to any plastic generally classified and reported under SIC group 28213.
E – Thermosetting Resins	Applies to any plastic generally classified and reported under SIC group 28214.
F – Commodity Organic Chemicals	Applies to commodity organic chemicals and commodity organic chemical groups generally classified and reported under SIC groups 2865, 2869, or 2899.
G – Bulk Organic Chemicals	Applies to bulk organic chemicals and bulk organic chemical groups generally classified and reported under SIC groups 2865, 2869, or 2899.
H – Specialty Organic Chemicals	Applies to all other organic chemicals and groups not specifically listed in Subparts F or G that are classified and reported under SIC groups 2865, 2869, or 2899.

SIC group key: 28213 (Thermoplastic Resins); 28214 (Thermosetting Resins); 2823 (Cellulosic Man-Made Fibers); 2824 (Synthetic Organic Fibers, Except Cellulosic); 2865 (Cyclic Crudes and Intermediates, Dyes, and Organic Pigments); 2869 (Industrial Organic Chemicals, Not Elsewhere Classified); 2899 (Miscellaneous Chemicals).

a – This SIC group listing is provided as a guide. See 40 CFR Part 414 for precise applicability and definitions of the OCPSF regulations.

The OCPSF point source category includes a broad range of sectors, raw materials, and unit operations that may manufacture or use PFAS. EPA identified that some OCPSF facilities manufacture PFAS through electrochemical fluorination, fluorotelomerization, or other processes. EPA identified six facilities in the OCPSF point source category that manufacture PFAS. For purposes of the Multi-Industry PFAS Study, EPA refers to these facilities as "PFAS manufacturers." The PFAS feedstocks may be further processed on site or transferred to other facilities where they are blended, converted, or integrated with other materials to produce new commercial or intermediate products. EPA identified that some OCPSF facilities use PFAS feedstocks as polymerization processing aids or in the production of plastic, rubber, resin, coatings, and commercial cleaning products. For the purpose of this study, EPA refers to facilities that are the primary customers of PFAS manufacturers and that use PFAS feedstocks to produce commercial goods or intermediary products as "PFAS formulators."

EPA focused on PFAS manufacturers and PFAS formulators for its review of the OCPSF point source category in this study. Table 6 lists domestic OCPSF facilities that EPA identified as PFAS manufacturers or PFAS formulators through outreach and information collected from chemical manufacturers, industry trade associations, and state and local wastewater authorities (described in Section 5.2) and describes PFAS manufacturing operations for each. These facilities produce products that have broad application in the industrial and consumer market. EPA notes that Table 6 includes major PFAS manufacturing sites EPA has identified thus far and is not a comprehensive list of all OCPSF facilities manufacturing or using PFAS in the United States.

EPA evaluated available information and data on the number, type, and volume of PFAS that are manufactured or imported to the United States using data reported by industry to EPA under the TSCA and industry literature. As of February 2020, EPA's TSCA Chemical Substance Inventory of active chemicals contains a total of 606 active PFAS (EPA, 2020b, 2020g). However, the inventory reflects substances in commerce between 2006 and 2016 so it includes long-chain PFAS now restricted in the United States and does not include any PFAS that have entered commerce since. The CDR rule, under the TSCA, requires manufacturers (including importers) to provide EPA with information on the production, import, and use of chemicals in commerce.[7] EPA evaluated the 2016 CDR database for PFAS in EPA's Cross-Agency PFAS Research List and determined that 118 of these PFAS were reported between 2012 and 2015 (EPA, 2021a, 2021b). The six PFAS manufacturers presented in Table 6 reported domestic manufacture or import of 76 individual PFAS. EPA notes that the 2016 CDR database does not reflect a holistic view of the total United States volume of PFAS manufactured and used because these data are reported as ranges in the database and facilities are exempt from reporting requirements if they have annual sales below $4 million, do not meet the reporting threshold of 25,000 pounds, imported the chemical as part of an article, or manufactured the chemical in a manner described in 40 CFR 720.30(g) or (h).

EPA also evaluated industry literature to assess the number of PFAS that are currently manufactured. A recent study (Buck et al., 2021) conducted by representatives of three global fluorochemical producers – AGC, Chemours, and Daikin – concluded that 256 PFAS were offered for sale as commercial products, ingredients, or degradation products (including components and impurities) in December 2019. The study authors classified the majority of PFAS reported as PFEAs or PFPEs (34 percent); short-chain fluorotelomers or fluorotelomer-based side-chain fluorinated polymers (28 percent); or fluoropolymers (15 percent) (Buck et al., 2021). EPA determined the study did not provide a comprehensive account of the number, type, and volume of PFAS manufactured in or imported into the United States due to the following limitations:

- The results reflect only a subset of manufacturers which do not practice electrochemical fluorination (does not account for PFAS produced by other companies or by electrochemical fluorination).
- The list of 256 PFAS reported were not identified in the study and may include PFAS only in commerce outside of the United States.
- No production volume, commercial product names, use/functionality, or alternatives were assessed.

EPA contacted industry to seek more information on the list of 256 PFAS reported but did not receive additional information. However, the industry did provide EPA with an economic assessment of the United States

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[7] Manufacturers and importers must report to the CDR database if they meet certain annual volume thresholds, typically 25,000 pounds, but 2,500 pounds for chemicals subject to certain TSCA actions. The information is collected every four years. The CDR information reported in 2016 (2016 CDR) reflects the most recent data set available and includes production volumes from 2012 to 2015.

fluoropolymer industry, which estimated 85,000 tons of fluoropolymers are produced and 77,500 tons are sold in the United States each year (Wood, 2020a).

Based on information and data derived from the data sets described above, EPA estimates that at least 118 PFAS are active in the United States market and 85,000 tons of PFAS are produced domestically each year.

Table 6. OCPSF Facilities Identified as PFAS Manufacturers or PFAS Formulators

Facility Name	Location	Description of PFAS Manufacture
PFAS Manufacturers		
3M Cordova Plant	Cordova, Illinois	Manufactures specialty fluorochemicals used in electronics, cleaning supplies, lubricant depositions, and antistatic polymers.
3M Decatur Plant	Decatur, Alabama	Manufactures fluoropolymers, fluoroelastomers, fluoroplastics, and flame-retardant polymers.
Chemours Chambers Works	Deepwater, New Jersey	Manufactures fluoropolymers, fluoroelastomers, fluoromonomers, PFPEs, fluorotelomers, and PFAS intermediates.
Chemours Fayetteville Works	Fayetteville, North Carolina	Manufactures PFPEs, fluoropolymers, fluoromonomers, and polymerization aids used for the GenX technology.
Chemours Washington Works	Parkersburg, West Virginia	Manufactures fluoropolymers and fluorotelomers.
Daikin Decatur Plant	Decatur, Alabama	Manufactures fluoropolymers and fluorotelomer-based substances.
PFAS Formulators		
3M Cottage Grove Plant	Cottage Grove, Minnesota	Processes PFAS feedstocks from 3M's Decatur, Alabama and Cordova, Illinois plants.
AGC Chemicals Americas	Thorndale, Pennsylvania	Processes fluoropolymers and fluorinated solvents that are manufactured internationally (no polymerization occurs in the United States).
DuPont Circleville Plant	Circleville, Ohio	Converts PFAS intermediates into fluoropolymer resin and film products.
DuPont Spruance Plant	Richmond, Virginia	Converts PFAS intermediates to produce PTFE fiber.
DuPont/Chemours Montague Plant	Montague, Michigan	*Unknown.*
Arkema/Sartomer Production Plant	West Chester, Pennsylvania	Processes fluoropolymers, such as PVDF.
Solvay Specialty Polymers USA, LLC	West Deptford, New Jersey	Processes fluoroelastomers and perfluoroelastomers.

5.2 Stakeholder Outreach

EPA met with industry stakeholders and state and local wastewater authorities, who voluntarily provided information on the manufacture, use, and discharge of PFAS by OCPSF facilities. EPA used this information to assess the volume and types of commercially produced PFAS and to understand better how to quantify and control PFAS discharges.

Outreach to the OCPSF industry included meeting with the American Chemistry Council (ACC) FluoroCouncil, a former subsidiary organization within the ACC that represented the world's leading manufacturers of fluorinated chemistries, and member companies AGC, Chemours, Daikin, and Solvay (ERG, 2019d). ACC provided EPA with technical literature concerning PFAS terminology and classification, a list of short-chain fluorotelomers studies, an economic assessment of the United States fluoropolymer industry, and contacts at 3M, Chemours, and Daikin, which they identified as the only PFAS manufacturing companies in the United States. The ACC FluoroCouncil disbanded in April 2020 and was superseded by two new groups:

- The Performance Fluoropolymer Partnership, which represents the world's leading companies that manufacture, formulate, or process fluoropolymers, fluoroelastomers, and polymeric perfluoropolyethers. Members include AGC, Chemours, Daikin, and Gujarat Fluorochemicals.
- The Alliance for Telomer Chemistry Stewardship, which represents leading manufacturers of six-carbon fluorotelomer-based products in North America, Europe, and Japan. Members include AGC, Daikin, Dynax Corporation, and Johnson Controls, Inc.

EPA met with representatives of 3M, AGC, Chemours, and Daikin and each company provided EPA with information on PFAS manufacture as well as operations and wastewater treatment data for their United States facilities (ERG, 2019e, 2019f, 2019g, 2019h). EPA also contacted other chemical manufacturers that voluntarily participated in the phased elimination of specific long-chain PFAS through the PFOA Stewardship Program: Arkema, BASF Corporation, Clariant Corporation, and Solvay. Clariant Corporation notified EPA that in 2013 it divested its fluorotelomer business to an independent entity now known as Archroma and that Clariant Corporation no longer manufactures PFAS (ERG, 2020f). EPA did not receive any additional information from Archroma, Arkema, BASF Corporation, or Solvay.

EPA met with the wastewater permitting authorities from the Wisconsin Department of Natural Resources (WI DNR); New Jersey Department of Environmental Protection (NJ DEP); Michigan Department of Environment, Great Lakes, and Energy (MI EGLE); and two large wastewater authorities in Michigan with POTWs that are investigating potential sources of PFAS wastewater. All provided EPA with wastewater permit materials and/or PFAS sampling results from PFAS manufacturers and PFAS formulators operating in their jurisdictions (ERG, 2019a, 2019b, 2019c, 2020b; NJ DEP 2015, 2018, 2020).

5.3 PFAS Wastewater Regulatory Requirements and Controls

EPA collected and reviewed NPDES wastewater discharge permits for the PFAS manufacturers and PFAS formulators listed in Table 6. EPA also collected publicly available materials associated with PFAS-related consent orders for the Chemours Fayetteville Works and 3M Decatur facilities. EPA reviewed these materials to identify effluent limitations or monitoring requirements for PFAS and assess current practices for managing PFAS-containing wastewaters. Table 7 summarizes PFAS wastewater regulatory requirements and existing PFAS control technologies and practices.

The National Defense Authorization Act for Fiscal Year 2020 established reporting requirements for 172 PFAS under the Toxics Release Inventory (TRI) program. An additional three PFAS were added for reporting year 2021. By July 1, 2021, facilities will have to report PFAS releases, including discharges to water, of any of 175 PFAS that they manufacture, process, or use above a 100-pound reporting threshold. EPA will review 2020 TRI data reported for PFAS once this information is publicly available.

Table 7. PFAS Wastewater Regulatory Requirements and Controls for PFAS Manufacturers and PFAS Formulators

Facility Name	Location	PFAS Wastewater Regulatory Requirements	Existing PFAS Wastewater Controls [a]
PFAS Manufacturers			
3M Cordova Plant	Cordova, Illinois	- NPDES permit IL0003140 establishes quarterly monitoring requirements for 14 PFAS. [b] - No effluent limitations for PFAS.	- *No known PFAS wastewater controls.*
3M Decatur Plant	Decatur, Alabama	- NPDES permit AL0000205 establishes quarterly monitoring requirements for 11 PFAS. [b] - 2020 consent order will require monitoring requirements for 33 PFAS. - No effluent limitations for PFAS.	- Granular activated carbon (GAC) treatment for directly discharged process wastewater and indirectly discharged fluoroelastomer washing water, as required by 2020 consent order. - Developing wastewater minimization plan and evaluating PFAS control technologies to further reduce PFAS discharged to wastewater treatment plant, as required by 2020 consent order.
Chemours Chambers Works	Deepwater, New Jersey	- NPDES permit NJ0005100 establishes weekly monitoring requirements for 16 PFAS. - No effluent limitations for PFAS.	- Powdered activated carbon (PAC) systems are in place, but not actively used to treat directly discharged process wastewater.
Chemours Fayetteville Works	Fayetteville, North Carolina	- NPDES permit NC0003573 establishes monitoring requirements for PFOA. - 2019 consent order prohibits discharge of Chemours' process wastewaters. - NPDES permit NC0089915 establishes biweekly monitoring requirements for HFPO-DA, PFMOAA, PMPA, and 56 additional PFAS for nonprocess wastewaters discharged via an old process wastewater outfall. - NPDES permit NC0089915 establishes effluent limitations for HFPO-DA, PFMOAA, and PMPA for nonprocess wastewaters discharged via an old process wastewater outfall.	- Chemours' process wastewater is captured and disposed off site via deep well injection or incineration. The only process wastewater discharged comes from Chemours' tenants DuPont and Kuraray. - As of November 2020, the facility was conducting a pilot study to evaluate PFAS removal from process wastewater using a treatment train consisting of two-stage reverse osmosis (RO), GAC, and ion exchange (IX) (Chemours, 2020b). - GAC treatment for contaminated nonprocess wastewaters to achieve 99% reduction of certain PFAS, as required by 2019 consent order. - Thermal oxidizer and thermolysis reactor to control PFAS air emissions.
Chemours Washington Works	Parkersburg, West Virginia	- NPDES permit WV0001279 establishes effluent limitations for APFO and HFPO-DA. - NPDES permit WV0001279 establishes monitoring requirements for 8 PFAS.	- Thermal oxidizer to control PFAS air emissions. - GAC treatment for directly discharged process wastewater. - Plans to install additional treatment units to meet future PFAS effluent limitations.

Table 7. PFAS Wastewater Regulatory Requirements and Controls for PFAS Manufacturers and PFAS Formulators

Facility Name	Location	PFAS Wastewater Regulatory Requirements	Existing PFAS Wastewater Controls [a]
Daikin Decatur Plant	Decatur, Alabama	- NPDES permit AL0064351 establishes quarterly monitoring requirements for 7 PFAS. [b] - No effluent limitations for PFAS.	- GAC treatment for directly discharged fluoropolymer/polymerization process wastewater. - Fluorotelomerization process wastewater is incinerated off site (no discharge).
PFAS Formulators			
3M Cottage Grove Plant	Cottage Grove, Minnesota	- NPDES permit MN0001449 establishes monthly monitoring requirements for 14 PFAS. - No effluent limitations for PFAS.	- Hazardous waste incinerator. - Regenerative thermal oxidizer and scrubber to control PFAS air emissions. - GAC treatment for process wastewaters.
AGC Chemicals Americas	Thorndale, Pennsylvania	- *Unknown.*	- *No known PFAS wastewater controls.*
DuPont Circleville Plant	Circleville, Ohio	- NPDES permit OH0006327 establishes monitoring requirements for PFOA. - No effluent limitations for PFAS.	- *No known PFAS wastewater controls.*
DuPont Spruance Plant	Richmond, Virginia	- NPDES permit VA0004669 establishes quarterly monitoring requirements for PFOA. - No effluent limitations for PFAS.	- *No known PFAS wastewater controls.*
DuPont/Chemours Montague Plant	Montague, Michigan	- NPDES permit MI0000884 establishes quarterly monitoring requirements for 4 PFAS. - No effluent limitations for PFAS.	- *No known PFAS wastewater controls.*
Arkema/Sartomer Production Plant	West Chester, Pennsylvania	- No monitoring requirements or effluent limitations for PFAS.	- *No known PFAS wastewater controls.*
Solvay Specialty Polymers USA, LLC	West Deptford, New Jersey	- NPDES permit NJ0005185 establishes weekly monitoring requirements for 12 PFAS. - No effluent limitations for PFAS.	- *No known PFAS wastewater controls.*

a – Conventional wastewater treatment methods (e.g., primary settling, physical-chemical treatment, neutralization, biological treatment, clarification) are not demonstrated to be effective controls for PFAS and are not presented in this table. See Section 10 for additional information on performance of wastewater treatment technologies.

b – The referenced NPDES permit has an expiration date that has passed but the permit is administratively extended.

5.4 Wastewater Characteristics

EPA evaluated available data on types and concentrations of PFAS in wastewater discharged from PFAS manufacturers and PFAS formulators. EPA summarized the limited information available and calculated average PFAS concentrations in effluent from these facilities based on 2019 DMR, 2020 DMR, industry-submitted, enforcement, and state and regional permitting authority data.

EPA identified analytical data that meet EPA's acceptance criteria for inclusion in analyses for characterizing PFAS in industrial wastewater discharges. EPA's acceptance criteria are presented in the memorandum "Development of the Multi-Industry PFAS Study Analytical Database" (ERG, 2021a). EPA identified seven sources of effluent sampling and monitoring data from PFAS manufacturers and OCPSF PFAS formulators that meet EPA's acceptance criteria:

- PFAS monitoring results reported in 2019 and 2020 DMRs (EPA, 2020c, 2021c).
- 2018 – 2019 PFAS monitoring results for three 3M facilities (3M, 2020a).
- 2020 PFAS monitoring results for Chemours Chambers Works (Chemours, 2020a).
- MI ELGE 2020 PFAS monitoring results for direct discharge facilities (MI EGLE, 2020b).
- NJ DEP PFAS monitoring data for Solvay Specialty Polymers USA, LLC in West Deptford, New Jersey (NJ DEP, 2020).
- An EPA Region 3 field sampling investigation report titled *PFAS Screening of Goose Creek & Goose Creek Industrial Users* (EPA Region 3, 2019).
- PFAS effluent sampling results submitted by PFAS manufacturers to EPA's Office of Enforcement and Compliance (EPA, 2020d).

EPA included 6,006 PFAS sample results representing all six PFAS manufacturers and 735 PFAS sample results representing six of seven PFAS formulators in its analysis characterizing PFAS in effluent from PFAS manufacturers and PFAS formulators. EPA calculated facility-level average, minimum, and maximum concentrations for each PFAS with available data using the following assumptions and limitations:

- EPA assumed all nondetection results and results below the level of quantification (i.e., the reporting or quantification limitation) were zero.[8]
- EPA did not have information on analytical method sensitivity and level of quantification for all data.
- EPA did not know the operations or treatment processes online at the facilities at the time of sampling.
- EPA does not have information on whether the observed PFAS concentrations are from legacy use of PFAS, current use of PFAS, or formation from degradation of more complex PFAS.

EPA calculated an overall average, minimum, and maximum concentration for each PFAS based on the facility-level results. EPA did not estimate average concentrations for any PFAS that were not detected at or above the level of quantification across all facilities or did not have any data.

Table 8 presents the average, minimum, and maximum concentrations for each PFAS in PFAS manufacturer and PFAS formulator effluent. As illustrated in the table, average PFAS concentrations in PFAS manufacturer effluent were higher than in PFAS formulator effluent for all PFAS except PFNA and PFUnA. Average concentrations for short-chain PFCAs and PFSAs were generally higher relative to long-chain PFCAs and PFSAs for both PFAS manufacturers and formulators.

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[8] The lower level of quantification is the lowest concentration that the analytical method being used can measure accurately.

Table 8. PFAS Manufacturer and PFAS Formulator PFAS Wastewater Concentrations

PFAS Subgroup	Analyte[a b]	PFAS Manufacturers			PFAS Formulators		
		Quantified Detections/ Total Sample Results	Concentration Range (µg/L)[c]	Average Concentration (µg/L)[c]	Quantified Detections/ Total Sample Results	Concentration Range (µg/L)[c]	Average Concentration (µg/L)[c]
PFCAs	PFBA	68/71	ND – 2,390	153	24/25	ND – 177	41.7
	PFPeA	78/95	ND – 20	2.49	24/24	0.169 – 4.08	0.829
	PFHxA	234/255	ND – 45	6.93	50/50	0.0022 – 0.519	0.111
	PFHpA	220/246	ND – 26	1.50	34/62	ND – 0.112	0.0167
	PFOA	1,235/1,367	ND – 430	3.77	60/73	ND – 1.6	0.116
	APFO	709/747	ND – 450	3.27	1/2	ND – 0.013	0.0065
	PFNA	145/164	ND – 1.19	0.224	26/62	ND – 14	0.883
	PFDA	120/125	ND – 1.5	0.271	25/50	ND – 0.088	0.0112
	PFUnA	32/74	ND – 0.09	0.0129	26/49	ND – 0.27	0.0401
	PFDoA	96/125	ND – 0.14	0.0182	0/50	ND	ND
	PFTrA	39/74	ND – 0.039	0.00416	2/50	ND – 0.0011	0.0000404
	PFTeA	31/58	ND – 0.04	0.00315	0/26	ND	ND
	PFHxDA	5/13	ND – 0.012	0.00218	*No Data*		
	PFODA	4/13	ND – 0.0044	0.000892	*No Data*		
PFSAs	PFBS	188/194	ND – 777	6.49	26/62	ND – 17.6	2.77
	PFPeS	4/14	ND – 0.013	0.000901	*No Data*		
	PFHxS	214/245	ND – 28.6	0.510	26/62	ND – 0.466	0.057
	PFHpS	1/14	ND – 0.0022	0.0000917	*No Data*		
	PFOS	58/76	ND – 21.2	3.37	49/63	ND – 0.153	0.034
	PFNS	0/14	ND	ND	*No Data*		
	PFDS	0/14	ND	ND	*No Data*		
FASAs	PFOSA	97/169	ND – 76.3	0.756	0/24	ND	ND
FTSAs	4:2 FTSA	0/14	ND	ND	*No Data*		
	6:2 FTSA	3/14	ND – 0.022	0.00177	*No Data*		
	8:2 FTSA	0/14	ND	ND	*No Data*		
N-Alkyl FASAs	NMePFOSA	0/13	ND	ND	*No Data*		
	NEtPFOSA	0/13	ND	ND	*No Data*		
FASEs and N-Alkyl FASEs	NMeFOSE	0/13	ND	ND	*No Data*		
	NEtFOSE	0/13	ND	ND	*No Data*		
FASAAs and N-Alkyl FASAAs	NMeFOSAA	70/107	ND – 112	2.92	*No Data*		
	NEtFOSAA	70/107	ND – 118	3.21	*No Data*		

Table 8. PFAS Manufacturer and PFAS Formulator PFAS Wastewater Concentrations

PFAS Subgroup	Analyte[a,b]	PFAS Manufacturers			PFAS Formulators		
		Quantified Detections/ Total Sample Results	Concentration Range (µg/L)[c]	Average Concentration (µg/L)[c]	Quantified Detections/ Total Sample Results	Concentration Range (µg/L)[c]	Average Concentration (µg/L)[c]
PFECAs	HFPO-DA	1,098/1,180	ND – 530	8.00	*No Data*		
	NaDONA	0/13	ND	ND	*No Data*		
	PMPA	0/3	ND	ND	*No Data*		
	PFMOAA	5/31	ND – 0.34	0.123	*No Data*		
	NFDHA	0/3	ND	ND	*No Data*		
	PFO2HxA	0/31	ND	ND	*No Data*		
	PFO3OA	0/31	ND	ND	*No Data*		
	PFO4DA	0/31	ND	ND	*No Data*		
	PFO5DA	0/31	ND	ND	*No Data*		
	PFECA-G	0/31	ND	ND	*No Data*		
PFESAs	K-9Cl-PF3ONS	0/13	ND	ND	*No Data*		
	K-11Cl-PF3OUdS	0/13	ND	ND	*No Data*		
	PFESA-BP1	0/31	ND	ND	*No Data*		
	PFESA-BP2	0/31	ND	ND	*No Data*		

Sources: ERG, 2021b.

Abbreviations: ND – nondetection; µg/L – micrograms per liter.

a – This table presents data for all PFAS listed in Table 2 for which sample results are available and meet EPA's acceptance criteria. See the PFAS Analytical Database for additional results for the following 11 PFAS not discussed in this preliminary report: 10:2 FTSA, PFEESA, FBSA, PFDoS, Hydro-EVE Acid, EVE Acid, PEPA, NVHOS, PFESA Byproduct 4, PFESA Byproduct 5, PFESA Byproduct 6.

b –The table identifies short-chain PFCAs (≤7 carbons) and short-chain PFSAs (≤5 carbons) in blue text, while long-chain PFCAs (≥8 carbons) and long-chain PFSAs (≥6 carbons) are designated in red text.

c – In this analysis, EPA treated all nondetection results as zero for the purpose of estimating concentrations. All concentration values are rounded to three significant figures.

5.5 OCPSF Point Source Category Summary

Based on information and data EPA collected for the Multi-Industry PFAS Study, EPA documented that PFAS have been, and continue to be, manufactured and used by OCPSF facilities in the United States. The type and quantity of PFAS manufactured and used vary by facility and have changed over time. Through outreach and data collected from industry, EPA identified six OCPSF facilities that manufacture PFAS in the United States through electrochemical fluorination, fluorotelomerization, or other processes. The PFAS feedstocks may be further processed on site or transferred to other facilities where they are blended, converted, or integrated with other materials to produce new commercial or intermediate products such as plastic, rubber, resins, coatings, and cleaning products. EPA identified seven additional OCPSF facilities that use PFAS feedstocks to formulate other products. EPA has not developed a comprehensive list of all PFAS manufacturers and formulators in the United States and considers it probable that there are more OCPSF facilities using PFAS that EPA has not yet identified. Based on limited information available, EPA estimates that the OCPSF facilities in the United States manufacture or use at least 118 PFAS and produce 85,000 tons of fluoropolymers annually.

EPA documented that the manufacture or formulation of PFAS by OCPSF facilities may generate PFAS-containing wastewaters. EPA verified that PFAS, including legacy long-chain PFAS and replacement PFAS, are present in wastewater discharges from PFAS manufacturers and PFAS formulators to surface waters and POTWs. Using available PFAS monitoring data, EPA estimated the average PFAS concentrations in PFAS manufacturer effluent were higher than average PFAS concentrations in effluent from PFAS formulators. Average concentrations for short-chain PFCAs and PFSAs were generally higher than the average concentrations of long-chain PFCAs and PFSAs; this was true for both PFAS manufacturers and formulators.

PFAS manufacturers and formulators have few monitoring requirements, effluent limitations, or pretreatment standards for PFAS in their wastewater discharge permits and may continue to discharge PFAS to POTWs or surface waters unless effective controls are in place. EPA identified some PFAS manufacturers and formulators successfully controlling PFAS in wastewater using granular activated carbon (GAC), ion exchange (IX), reverse osmosis (RO), and thermal treatment systems. Based on EPA’s Drinking Water Treatability Database (DWTD), these technologies are able to remove more than 99 percent on some PFAS in industrial wastewater, or completely eliminate the discharge of wastewater containing PFAS (EPA, 2021f). See Section 10 for more information on PFAS discharge control technologies.

6. Review of the Metal Finishing Point Source Category

This section describes the metal finishing point source category, information and data EPA collected on its PFAS use and discharge, and EPA's estimates of types and concentrations of PFAS discharged by facilities within the category. EPA collected and reviewed information on PFAS use and discharge by metal finishing facilities from the following sources:

- Outreach with an industry trade association and state and local wastewater authorities.
- Reports and data collected from industry and state and local wastewater authorities.
- Publicly available technical literature.

6.1 Industry Description and Use of PFAS

Metal finishing refers to changing the surface of an object to improve its appearance and/or durability. EPA promulgated the Metal Finishing Effluent Guidelines (40 CFR Part 433) in 1983, with technical amendments in 1984 and 1986. The regulations cover wastewater discharges from facilities that perform one or more of the following six metal finishing operations on any basis material:

- Electroplating.[9]
- Electroless plating.
- Anodizing.
- Coating (phosphating, chromating, and coloring).
- Chemical etchings and milling.
- Printed circuit board manufacture.

If a facility performs any of these six core operations, then discharges from the 46 operations listed in 40 CFR Part 433.10(a) are covered by the Metal Finishing ELGs. EPA estimates about 44,000 facilities perform metal finishing operations and discharge process wastewater directly to United States surface waters or indirectly to surface waters through POTWs.

The metal finishing point source category includes a broad range of sectors, raw materials, and unit operations that may use PFAS. EPA identified that some metal finishing facilities have used, and continue to use, nonpolymer PFAS and related products as wetting agents, mist and fume suppressants (to prevent emissions of toxic metal fumes to air), agents to reduce mechanical wear, and surface coatings to impart specific characteristics (e.g., reduced corrosion, enhanced aesthetic appearance). EPA also identified that some polymer PFAS, such as PTFE, may be used in electroless nickel plating operations (NASF, 2019b; MI EGLE, 2020d; ITRC, 2020; Glüge et al., 2020). PFAS used by metal finishing facilities may be transferred to wastewater streams generated by the facility and ultimately discharged to surface waters or POTWs.

Based on studies conducted by EPA and states since 2007, the agency identified chromium electroplating and chromium anodizing operations (collectively referred to as "chromium electroplating facilities") as the most significant source of PFAS, particularly PFOS, in the metal finishing point source category (MPCA, 2006; EPA Region 5, 2009; MI EGLE, 2020d). Thus, EPA focused on chromium electroplating facilities for its review of the metal finishing point source category in this study.

Since the 1980s, mist and fume suppressants containing 5 to 10 percent PFOS by weight were frequently used by United States chromium electroplating facilities to control hexavalent chromium emissions (a known human carcinogen and inhalation hazard), as required under the Clean Air Act by the National Emission Standards for

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[9] Metal finishing is related to electroplating, which is the production of a thin surface coating of the metal upon another by electrodeposition. Certain electroplating processes are covered by the ELGs for the Electroplating Category (40 CFR Part 413), rather than the ELGs for the Metal Finishing Category. These include job shop electroplaters, independent printed circuit board manufacturers, indirect discharge electroplating facilities, and electroplating facilities in operation before July 15, 1983.

Hazardous Air Pollutants (NESHAP) for Chromium Emissions from Hard and Decorative Chromium Electroplating and Chromium Anodizing (40 CFR Part 63, Subpart N). The surfactant properties of PFOS reduce the surface tension of the electrolyte solution, which limits the release of hexavalent chromium vapors to the air and thereby reduces worker exposure. Due to concerns about human health and environmental impacts of PFOS, in 2012 EPA amended the NESHAP to phase out the use of mist and fume suppressants containing more than 1 percent PFOS by weight in chromium electroplating by September 21, 2015 (ITRC, 2020; NASF, 2019b; EPA, 2012a). EPA identified the following alternative technologies adopted by industry following the restriction on PFOS-based agents:

- Replacing PFOS-based mist and fume suppressants with fluorotelomer-based substances (e.g., 6:2 FTSA) and chlorinated PFESAs (e.g., F-53B).[10]
- Replacing PFOS-based mist and fume suppressants with nonfluorinated mist and fume suppressants.
- Using mechanical controls such as enclosed lines, air jet systems, air pollution scrubbers, and closed-loop systems to manage hexavalent chromium emissions.
- Transitioning to trivalent chromium rather than hexavalent chromium for decorative chromium electroplating. Trivalent chromium is considered less toxic and less bioavailable relative to hexavalent chromium. However, trivalent chromium generally cannot be used for hard chromium electroplating because of quality differences and cost (MI ELGE, 2020a).

Once the electroplating bath liquid can no longer be used, it may be treated to remove chromium and other metals, but PFAS may remain present and be discharged in effluent from chromium electroplating facilities still using PFAS-based mist and fume suppressants. Most metal finishing facilities, including those that perform chromium electroplating, discharge wastewater to their local sewer system rather than to surface waters.

In developing the 2012 NESHAP, EPA developed a profile of the 1,339 chromium electroplating facilities operating in the United States (652 hard chromium electroplating, 517 decorative chromium electroplating, 170 chromium anodizing) (EPA, 2012a). Approximately 50 percent of chromium electroplating facilities apply PFAS-based mist and fume suppressants, based on information from the 2020 MI EGLE report titled *Identified Industrial Sources of PFOS to Municipal Wastewater Treatment Plants* (MI EGLE, 2020d).

6.2 Stakeholder Outreach

EPA met with the National Association for Surface Finishing (NASF) and Michigan wastewater authorities, who voluntarily provided information on the use and discharge of PFAS by metal finishing facilities. NASF is a trade association that represents the United States surface coating industry and provided EPA with information to categorize PFAS historically and currently used in the industry. NASF estimates that 30 to 40 percent of surface finishing facilities have chromium electroplating processes, but not all facilities have used or currently use PFAS-based mist and fume suppressants (NASF, 2019c). Representatives from MI EGLE and two large Michigan wastewater authorities investigating potential sources of PFAS wastewater provided EPA with information on current efforts to regulate and control PFAS discharges and/or PFAS sample results for metal finishing facilities operating in those jurisdictions. As part of ongoing investigations, MI EGLE (2020d) determined that approximately two-thirds of sampled chromium electroplating facilities discharged PFOS exceed the Michigan water quality standard of 12 ng/L even though mist and fume suppressants containing more than 1 percent PFOS are no longer in use. MI EGLE also found no detectable amounts of PFOS precursors or PFOA precursors in mist and fume suppressant samples from 11 chromium electroplating facilities. The presence of PFOS may be due to legacy issues, trace levels (less than 1 percent) of PFOS in modern suppressants, or, to a lesser degree, due to degradation of other more complex PFAS (MI EGLE, 2020a, 2020d).

6.3 PFAS Wastewater Regulatory Requirements and Controls

EPA did not identify any chromium electroplating facilities with PFAS effluent limitations or pretreatment standards in their wastewater discharge permits. As part of Michigan's Industrial Pretreatment Program PFAS Initiative, MI EGLE required 95 POTWs to evaluate their industrial users as potential sources of PFOS and PFOA. These local POTWs implemented requirements for industrial users discharging to their system to monitor for

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[10] EPA and MI EGLE analysis of modern mist and fume suppressants used by 11 chromium electroplaters in Michigan showed that 6:2 FTSA was the only detectable PFAS; PFOS and PFOS precursors were not detected in any suppressant samples evaluated (MI EGLE, 2020a).

PFOA and PFOS and, if effluent PFOS concentrations exceeded Michigan's screening value of 12 ng/L, implement PFOS reduction programs. MI EGLE reports that numerous POTWs identified chromium electroplating facilities as sources of PFOS discharges and have required these sources to install PFAS pretreatment, such as GAC. MI EGLE identified six Michigan chromium electroplating facilities have lowered effluent concentrations of PFAS using GAC (MI EGLE, 2020d).

6.4 Wastewater Characteristics

EPA evaluated available data on types and concentrations of PFAS in wastewater discharged from chromium electroplating facilities. EPA has not identified any facilities with PFAS monitoring requirements or effluent limitations; therefore, no DMR data are available for PFAS. EPA summarized the limited information available and calculated average PFAS concentrations in effluent from chromium electroplating facilities based on Michigan permitting authority data.

EPA identified analytical data that meet EPA's acceptance criteria for inclusion in analyses for characterizing PFAS discharges in industrial wastewater discharges. EPA's acceptance criteria are presented in the memorandum "Development of the Multi-Industry PFAS Study Analytical Database" (ERG, 2021a). EPA identified two sources of analytical data for chromium electroplating, both of which meet EPA's acceptance criteria: *Targeted and Nontargeted Analysis of PFAS in Fume Suppressant Products at Chrome Plating Facilities* (2020 chromium electroplating report) (MI EGLE, 2020a) and MI EGLE PFAS monitoring results for indirect discharge facilities (MI EGLE, 2020c). EPA included 1,137 PFAS sample results representing 47 chromium electroplating facilities in its analysis characterizing PFAS in chromium electroplating wastewater.

Some wastewater sample data from the 2020 chromium electroplating report (MI EGLE, 2020a) were collected before adsorption treatment processes that target PFAS. EPA included these data in this preliminary analysis because most chromium electroplating facilities do not have dedicated treatment for PFAS in place and, thus, the samples are representative of effluent from most facilities. During discussions with EPA, NASF representatives stated that the MI EGLE (2020a) data are a fair representation of the industry as a whole (ERG, 2020c).

EPA calculated facility-level average, minimum, and maximum concentrations for each PFAS with available data using the following assumptions and limitations:

- EPA assumed all nondetection results and results below the level of quantification (i.e., the reporting or quantification limitation) were zero.[11]
- EPA did not have information on analytical method sensitivity and level of quantification for all data.
- EPA did not know the operations or treatment processes online at the facilities at the time of sampling.
- EPA does not have information on whether the observed PFAS concentrations are from legacy use of PFAS, current use of PFAS, or form from degradation of more complex PFAS.

EPA calculated an overall average, minimum, and maximum concentration for each PFAS based on the facility-level results. EPA did not estimate average concentrations for any PFAS that were not detected at or above the level of quantification across all facilities or did not have any data.

Table 9 presents the average, minimum, and maximum concentrations for each PFAS observed in chromium electroplating facility effluent. As illustrated in the table, EPA estimated the average wastewater concentration of 6:2 FTSA was more than 100 times greater than any other PFAS detected in chromium electroplating wastewater. Despite the phase out of PFOS-based mist and fume suppressants, some chromium electroplating facilities still report detectable levels of PFOS in their wastewater.

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[11] The lower level of quantification is the lowest concentration that the analytical method being used can measure accurately.

Table 9. Chromium Electroplating Wastewater PFAS Concentrations

PFAS Subgroup	Analyte [a b]	Facilities with Data	Quantified Detections/Total Sample Results	Concentration Range (µg/L) [c]	Average Concentration (µg/L) [c]
PFCAs	PFBA	11	5/12	ND – 4.71	0.492
	PFPeA	11	5/12	ND – 0.93	0.108
	PFHxA	11	8/12	ND – 0.34	0.0703
	PFHpA	11	8/12	ND – 0.49	0.0687
	PFOA	47	51/406	ND – 0.74	0.00770
	PFNA	11	0/12	ND	ND
	PFDA	11	0/12	ND	ND
	PFUnA	11	0/12	ND	ND
	PFDoA	11	0/12	ND	ND
	PFTrA	11	0/12	ND	ND
	PFTeA	11	0/12	ND	ND
PFSAs	PFBS	10	7/11	ND – 19.8	2.09
	PFPeS	11	2/12	ND – 0.088	0.015
	PFHxS	11	8/12	ND – 1.22	0.147
	PFHpS	11	7/12	ND – 0.323	0.0595
	PFOS	47	412/456	ND – 240	4.86
	PFNS	11	3/12	ND – 0.043	0.00636
	PFDS	11	1/12	ND – 0.014	0.00127
FASAs	PFOSA	11	0/12	ND	ND
FTSAs	4:2 FTSA	11	8/12	ND – 1.39	0.229
	6:2 FTSA	11	11/12	ND – 3,140	532
	8:2 FTSA	11	7/12	ND – 0.237	0.0633
FASAAs and N-Alkyl FASAAs	NMeFOSAA	11	0/12	ND	ND
	NEtFOSAA	11	0/12	ND	ND
PFECAs	HFPO-DA	11	5/12	ND – 0.065	0.0124

Sources: ERG, 2021b.

Abbreviations: ND – nondetection; µg/L – micrograms per liter.

a – This table presents data for all PFAS listed in Table 2 for which sample results are available and meet EPA's acceptance criteria. EPA does not have any sample results PFAS note listed.

b –The table identifies short-chain PFCAs (≤7 carbons) and short-chain PFSAs (≤5 carbons) in blue text, while long-chain PFCAs (≥8 carbons) and long-chain PFSAs (≥6 carbons) are designated in red text.

c – In this analysis, EPA treated all nondetection results as zero for the purpose of estimating concentrations. All concentration values are rounded to three significant figures.

6.5 Metal Finishing Point Source Category Summary

Based on information and data EPA collected for the Multi-Industry PFAS Study, EPA documented that PFAS have been, and continue to be, used by metal finishing facilities in the United States. EPA identified chromium electroplating facilities as the most significant source of PFAS in the metal finishing point source category. Chromium electroplating facilities use PFAS-based mist and fume suppressants to control toxic hexavalent chromium emissions. PFOS-based mist and fume suppressants were frequently used until 2015, when EPA's revisions to the chromium electroplating NESHAP required chromium electroplating facilities to phase out mist and fume suppressants containing more than 1 percent PFOS by weight. Chromium electroplating facilities have adopted alternative technologies, including mist and fume suppressants containing fluorotelomer-based substances (e.g., 6:2 FTSA) and chlorinated PFESAs (e.g., F-53B), to replace use of PFOS-based products. EPA estimates that approximately half of the 1,339 chromium electroplating facilities in the United States still apply some type of PFAS-based mist and fume suppressant.

EPA documented that the use of PFAS-based mist and fume suppressants may generate wastewaters containing PFAS. EPA verified that PFAS, including legacy long-chain PFAS and replacement PFAS, are present in wastewater discharges from chromium electroplating facilities to surface waters and POTWs. Using available sampling data, EPA estimated the average wastewater concentration of 6:2 FTSA was more than 100 times greater than any other PFAS evaluated. 6:2 FTSA was the only PFAS detected in a 2020 MI EGLE targeted analysis of PFAS in mist and fume suppressants used by several facilities included in this analysis. Despite the phase out of PFOS-based mist and fume suppressants, some chromium electroplating facilities still report detectable levels of PFOS in their wastewater. MI EGLE (2020d) determined that approximately two-thirds of the evaluated chromium electroplating facilities discharged PFOS exceed the Michigan water quality standard of 12 ng/L even though mist and fume suppressants containing more than 1 percent PFOS are no longer in use. As part of a separate study, EPA and MI EGLE found no detectable amounts of PFOS precursors or PFOA precursors in mist and fume suppressant samples from 11 facilities. The presence of PFOS may be due to legacy issues, trace levels (less than 1 percent) of PFOS in modern suppressants, or, to a lesser degree, due to degradation of other more complex PFAS (MI EGLE, 2020a, 2020d).

EPA did not identify any chromium electroplating facilities with PFAS effluent limitations or pretreatment standards in their wastewater discharge permit and estimates that less than 5 percent of chromium electroplating facilities monitor for PFAS. Most chromium electroplating facilities are not monitoring for PFAS and are likely to continue to discharge PFAS to POTWs or surface waters unless effective controls are in place. EPA identified that at least six Michigan chromium electroplating facilities have lowered effluent concentrations of PFAS using GAC.

7. Review of the Pulp, Paper, and Paperboard Point Source Category

This section describes the pulp, paper, and paperboard point source category; information and data EPA collected on the category's PFAS use and discharge; and EPA's estimates of types and concentrations of PFAS discharged by the category. EPA collected and reviewed information on PFAS use and discharge by pulp, paper, and paperboard facilities from the following sources:

- Outreach with pulp and paper companies, an industry trade association, and state and local wastewater authorities.
- Reports and data collected from industry and state and local wastewater authorities.
- Publicly available technical literature.

7.1 Industry Description and Use of PFAS

The pulp, paper, and paperboard point source category includes companies and facilities that convert wood into pulp, paper, paperboard, and other cellulose-based products. Facilities that convert wood to pulp, paper, or paperboard are generally classified as integrated mills or nonintegrated mills. Integrated mills have the onsite capability to convert wood into pulp, and then into paper, while nonintegrated mills only manufacture paper or paperboard from purchased pulp. There are additional types of mills that manufacture only pulp or recycled fiber for manufacture of various good elsewhere (e.g., market pulp mills, dissolving pulp mills, fluff pulp mills, recycled fiber mills). There are also converting facilities which cut, fold, or otherwise convert manufactured paper into commercial products.

EPA promulgated the Pulp, Paper, and Paperboard Effluent Guidelines (40 CFR Part 430) in 1974 and 1977, with numerous technical amendments and revisions between 1982 and 2007. EPA estimated approximately 565 pulp, paper, and paperboard mills operated in the United States in 1997, the time of the most recent major ELGs rulemaking. The ELGs cover wastewater discharges from facilities which perform specified pulping processes or manufacture specified paper products and are typically classified under four SIC groups: 2621, 2631, 2641, and 2661. The existing ELGs do not establish effluent limitations or pretreatment standards for PFAS.

PFAS have been used by pulp, paper, and paperboard facilities as an additive or coating to impart certain surfactant qualities to finished paper products. Some facilities have manufactured high-performance paper products, such as those requiring oil, grease, and/or moisture resistance, using additives mixed with the pulp before it is formed into paper. Other facilities have applied coatings containing PFAS to the finished paper. PFAS are primarily used by facilities that manufacture food contact papers and packaging (e.g., fast food wrappers, take-out containers, bakery bags, popcorn bags, pizza boxes), but also have limited applications for specialty paper products (e.g., carbonless forms, masking paper) (AF&PA, 2020a; WA DEC, 2021).

Chemicals used in food contact paper and packaging are regulated by the United States Department of Health and Human Services, Food and Drug Administration (FDA) because of their potential to migrate to food. All food contact substances (FCSs) must be approved through the food contact substance notification (FCN) program, under which the FDA will review available migration, exposure, and human health risk data to ensure an FCS is safe for its intended use prior to approving it for use in market. Manufacturers of chemicals approved to be used as an FCS are permitted to market and sell these chemicals to food contact paper and packaging producers who will use them in their products (FDA, 2020).

Since the 1960s, the FDA has authorized several broad classes of PFAS for use as FCSs, including long-chain PFAS such as PFOS and PFOA, and more recently short-chain, fluorotelomer, and side-chain polymer PFAS. As of December 2020, there are 17 distinct PFAS approved by the FDA for use to provide oil and grease resistance in food contact applications. All approved PFAS are for polyfluorinated polymers, with the majority being six-carbon side-chain fluorinated polymers and the rest being perfluoropolyethers (FDA, 2021; WA DEC, 2021).

When the FDA identifies potential safety concerns with an FCS, the agency may work with industry to reach voluntary market phase out agreements or revoke food contact authorizations. In 2011, the FDA worked with three manufacturers (DuPont, Clariant Corporation, and BASF Corporation) to voluntarily end sale of several long-chain PFAS for food contact applications. In 2016, the FDA revoked authorization for the remaining uses of long-chain PFAS and these chemicals are no longer approved for use in food contact applications in the United States. In July 2020, the FDA announced that four manufacturers will voluntarily phase out the use of PFAS containing or degrading to 6:2 FTOH. The market phase out is a response to FDA research that raised questions about human health risks for 6:2 FTOH. The four manufacturers (AGC, Chemours, Daikin, and Archroma) hold FCNs for approximately 11 PFAS containing or degrading to 6:2 FTOH. AGC, Daikin, and Archroma agreed to a complete market phase out of PFAS containing 6:2 FTOH by December 31, 2023; Chemours has already stopped sales of 6:2 FTOH-containing products in the United States (FDA, 2020; WA DEC, 2021).

In recent years, an increasing number of major food distributers and retailers (e.g., Sweetgreen, Chipotle, McDonald's, Taco Bell, Whole Foods Market, Trader Joes, Kroger, Panera Bread) have announced plans to phase out all PFAS in food contact papers and packaging in response to consumer and regulatory pressures. Several states, including California, Maine, New York, Vermont, and Washington, are also acting to restrict use of PFAS in these materials and identify alternative substances that provide comparable performance without use of PFAS. Washington Department of Ecology identified the following FDA-approved alternatives to PFAS coated food contact paper (WA DEC, 2021):

- Uncoated paper.
- Wax coated paper.
- Clay coated paper.
- Siloxane coated paper.
- Polyvinyl alcohol (PVOH) or ethylene vinyl alcohol (EVOH) copolymer coated paper.
- Polylactic acid (PLA) and PLA coated paper.
- Polyethylene (PE) or polyethylene terephthalate (PET) coated paper.

7.2 Stakeholder Outreach

EPA met with industry stakeholders and state and local wastewater authorities, who voluntarily provided information on the use and discharge of PFAS by pulp, paper, and paperboard facilities. EPA used this information to categorize PFAS being used in the industry, quantify PFAS in discharges, and to learn how the industry controls PFAS discharges.

Outreach to the industry included contact with the American Forest and Paper Association (AF&PA) and companies that historically or currently manufacture food contact paper/packing or specialty paper products. AF&PA is a national trade association for the forest, pulp, and paper industry; its 38 member companies represent approximately 85 percent of the pulp, paper, tissue, and paper-based packaging products manufactured in the United States. EPA met with the AF&PA and 10 member companies in March 2020 (ERG, 2020d). AF&PA provided four letters to EPA containing information on the use and discharge of PFAS in the pulp and paper industry (AF&PA, 2020a, 2020b, 2020c, 2020d).

AF&PA reported that the pulp, paper, and paperboard industry phased out the use of PFOA and PFOS approximately 10 years ago, but continues to use FDA-approved short-chain PFAS in the manufacture of food contact packaging and specialty paper products to enhance resistance to water, oil, and grease. According to AF&PA, PFAS is not integral to or used in the pulping process (including pulp brightening or bleaching), recycling of paper and packaging, or manufacture of products requiring absorbency, publishing papers, newsprint, and conventional packaging (e.g., standard corrugated boxes). AF&PA states certain PFAS may be present in process equipment components (e.g., PTFE piping or valves), cleaners, and firefighting foam used at pulp, paper, and paperboard facilities; however, these substances are used infrequently and in small volumes compared to raw materials used in pulp and paper manufacturing. Additionally, AF&PA reported that some PFAS may enter the facility through intake water or from recycled paper products that are used to create recovered pulp and fiber.

Whether PFAS are in an additive mixed with the pulp prior to forming it into paper, or in a coating applied to the surface of the paper after the paper is formed, AF&PA reports the majority of PFAS would remain in the final product and not be transferred to a wastewater stream. AF&PA stated that the PFAS chemistries may be expensive, so there is economic incentive for facilities to minimize PFAS use, minimize loss to process wastewater, and retain as much PFAS as possible in the final product.

In 2020, AF&PA conducted a survey of their member companies regarding their use of PFAS. Nineteen AF&PA member companies responded to the survey, representing 146 of the 171 mills operated by members. Five of the 146 mills covered by the respondents reported they intentionally use PFAS in the manufacture of pulp and paper products as of July 2020. The five mills are operated by different companies and all reported that PFAS would be phased out of production processes in the next three to four years. Table 10 summarizes the PFAS-containing products produced by these mills and company plans to reduce or alter PFAS use.

Table 10. Summary of AF&PA Member Company Mills Using PFAS

Mill ID	Products and Processes Using PFAS	PFAS Phase Out Steps
A	Uses FDA-approved PFAS in products with food service applications.	Products to be phased out in 2021, so no alternatives to PFAS under consideration.
B	Uses FDA-approved PFAS in food packaging.	Commercial alternatives under evaluation. Other coatings in development.
C	Uses FDA-approved PFAS in paper for food packaging and other specialty packaging.	Production-scale trials expected for two PFAS-free product designs.
D	Uses FDA-approved PFAS in coatings on bleached kraft paper for specialty business forms.	Alternative materials have been evaluated in lab, with plans to move forward on pilot testing and manufacturing trials.
E	Uses FDA-approved PFAS in food packaging.	Facility closed in 2021 with no plans to move manufacture to another site in the United States.

Source: AF&PA, 2020a, 2020b, 2020c, 2020d.

The total production of paper containing PFAS for the five mills (85,000 tons) accounts for 4.3 percent of these five mills' total paper production and roughly 0.14 percent of the mill production of all AF&PA members. AF&PA stated that one mill, Mill E, closed in 2021 and the company no longer uses PFAS at any United States mills. AF&PA also confirmed that no PFOA, PFOS, or GenX chemicals (including HFPO-DA) are intentionally added to the products at the four mills that remain active. The four active mills indicated that they are either actively seeking nonPFAS alternatives for their coatings or ending production of PFAS-containing product lines, a decision driven by public opinion, market pressure, and regulatory measures.

AF&PA identified major producers of food contact paper and packaging or specialty paper products and provided EPA with contact information for representatives of these companies. EPA met separately with representatives of Ahlstrom-Munksjö USA, Inc. (Ahlstrom-Munksjö); Georgia-Pacific, LLC (Georgia-Pacific); Graphic Packaging International, Inc. (Graphic Packaging); WestRock Company; and Sappi North America, Inc. (Sappi). Each company provided EPA with information on their historical and current PFAS use, as well as operations and wastewater treatment data for their United States facilities.

EPA and representatives from Ahlstrom-Munksjö discussed the company's operations at two pulp and paper mills and two specialty paper manufacturing facilities in Wisconsin. Ahlstrom-Munksjö became a member of AF&PA after the trade association's PFAS survey was conducted and their facilities are not included the survey results. As of July 2021, the four Ahlstrom-Munksjö Wisconsin facilities are applying coatings containing PFAS to impart oil and grease resistance to food service products. Two copolymer coatings, supplied by Daikin and Solvay, contain FDA-approved PFAS and are applied to finished sheets in a closed-loop, recirculating system (excess coating is captured and reused). To Ahlstrom-Munksjö's knowledge, no wastewaters are generated during the coating process. Ahlstrom-Munksjö estimated that approximately 10 percent of production at the four Wisconsin plants is manufactured using PFAS; however, Ahlstrom-Munksjö is transitioning all Wisconsin facilities to FluoroFree® technology and 100 percent PFAS-free products, with a goal to eliminate PFAS use by end of 2023. Ahlstrom-Munksjö stated the company also operates five additional pulp, paper, or paperboard manufacturing facilities in other states. Of these five facilities, only one site, in Windsor Locks, Connecticut is using significant volumes of

FDA-approved PFAS. Ahlstrom-Munksjö did not provide additional information on PFAS use at the Windsor Locks, Connecticut facility or other facilities in the United States (Ahlstrom-Munksjö, 2021; EPA, 2021g).

Georgia-Pacific has not directly purchased or applied PFAS to paper products since 3M phased out production of PFOA, PFOS, and other long-chain PFAS in the 2000s. Until April 2021, Georgia-Pacific continued to purchase, cut, fold, and otherwise convert paper treated with FDA-approved PFAS into food contact products, including sandwich wraps, take-out containers, and food trays. In April 2021, Georgia-Pacific discontinued purchasing PFAS-treated paper, and switched to paper treated with a nonPFAS polymer blend. Based on the company's estimate at the time, the inventory of PFAS-treated paper in the supply chain would be distributed by July 2021. Georgia-Pacific indicated that the transition to nonPFAS alternatives was influenced by customer interest in PFAS-free products and the company's forecast of market demand for such products. Georgia-Pacific confirmed that the company does not own or operate any of the five mills presented in Table 10; however, the company reported that their Packerland Plant in Green Bay, Wisconsin, purchased and converted PFAS-treated paper in 2020. Georgia-Pacific provided EPA with PFAS monitoring data, representing effluent from two pulp, paper, and paperboard mills in Green Bay, Wisconsin (not the Packerland Plant) and also confirmed that the company operated two facilities represented in a sampling data set previously provided by AF&PA (EPA, 2021h).

As of June 2021, Graphic Packaging was intentionally applying PFAS-based coatings in the manufacture of food contact paper and packaging at one facility, the Texarkana Mill in Queen City, Texas. Graphic Packaging estimates 15,000 tons of PFAS-treated product is manufactured at the Texarkana Mill annually, accounting for less than 2 percent of the mill's total annual production and less than 1 percent of the total annual production across all Graphic Packaging facilities. Wastewater generated during the papermaking process and washing equipment is treated at an onsite wastewater treatment plant before it is discharged to the Sulphur River. Graphic Packaging reported that the Battle Creek Mill in Battle Creek, Michigan also intentionally applied PFAS until 2018 (zero PFAS use since 2018). Graphic Packaging further reported that all PFAS use will be discontinued by January 1, 2022 (this inventory will be exhausted by end of March 2022) and PFAS-based coatings will be substituted with alternative technologies such as polymer resins and proprietary nonfluorinated coatings (EPA, 2021i).

WestRock Company historically used FDA-approved PFAS in production of food contact papers and packaging. As of 2021, WestRock Company no longer intentionally uses PFAS in pulp, paper, and paperboard production at any of the company's 28 mills and more than 200 converting facilities in the United States. WestRock Company completed transition of the last facility using PFAS to nonfluorinated technologies in 2020 (EPA, 2021j).

As of July 2021, Sappi was intentionally using PFAS-based additives in food packaging applications at their Somerset Mill, one of the company's three United States mills. The Somerset Mill is an integrated pulping and papermaking mill in Skowhegan, Maine that uses FDA-approved PFAS to enhance grease-resistance of specialty food packaging products. The company reported that less than 2 percent of the Somerset Mill's annual production is manufactured using PFAS-based additives and that they account for less than 1 percent of the weight of the food packaging products in which they are used. The PFAS additives are applied at the wet-end of the papermaking process in one product line of grease-proof food packaging products. Wastewater generated during the process is captured and treated at the Somerset Mill's onsite wastewater treatment plant prior to being discharged to the Kennebec River. This treatment includes primary settling, activated sludge, and a polishing pond prior to discharge, and does not include any treatment for PFAS chemicals specifically. Sappi reports that the company is developing nonfluorinated additive alternatives and will eliminate PFAS use across all product lines by 2024 (EPA, 2021m).

EPA also contacted Kimberly-Clark Corporation, Domtar Corporation, and International Paper Company to discuss potential use of PFAS at pulp, paper, and paperboard facilities. Kimberly-Clark Corporation notified EPA that PFAS would inhibit the high absorbency of their products and that the specialty paper business unit was spun off as a separate entity known as Neenah Paper in 2004 (EPA, 2021l). EPA attempted to contact Neenah Paper to discuss potential use of PFAS and received no response (EPA, 2021n). Neither Kimberly-Clark Corporation or Neenah Paper are members of AF&PA. In a June 2021 letter, Domtar Corporation notified EPA that the company's Port Huron Mill, in Port Huron, Michigan, used FDA-approved short-chain PFAS to manufacture food contact paper and packaging until the facility closed in March 2021. None of Domtar Corporation's remaining nine pulp, paper, and paperboard manufacturing facilities use PFAS in production of paper-based products (Domtar Corporation, 2021). In a July 2021 letter, International Paper Company confirmed the company does not use PFAS in manufacturing products at United States mills and does not sell into the United States products with PFAS intentionally added (International Paper Company, 2021).

7.3 PFAS Wastewater Regulatory Requirements and Controls

As part of the Multi-Industry PFAS Study, EPA did not identify any pulp, paper, and paperboard facilities with PFAS effluent limitations or pretreatment standards in their wastewater discharge permits. As part of Michigan's Industrial Pretreatment Program PFAS Initiative, MI EGLE required 95 POTWs to evaluate their industrial users as potential sources of PFOS and PFOA. These local POTWs implemented requirements for industrial users discharging to their system to monitor for PFOA and PFOS and, if effluent PFOS concentrations exceeded Michigan's screening value of 12 ng/L, implement PFOS reduction programs. MI EGLE reports that POTWs identified approximately ten pulp, paper, or paperboard manufacturing facilities as sources of PFOS discharges and are collaborating with these sources to further investigate and reduce PFOS concentrations (MI EGLE, 2020d).

7.4 Wastewater Characterization

EPA evaluated the available data on types and concentrations of PFAS in wastewater discharged from pulp, paper, and paperboard facilities. EPA has not identified any facilities with PFAS monitoring requirements or effluent limitations; therefore, no DMR data are available for PFAS. EPA summarized the information available and calculated average PFAS concentrations in effluent from pulp, paper, and paperboard facilities based on industry and state permitting authority data.

EPA identified analytical data that meet EPA's acceptance criteria for inclusion in analyses for characterizing PFAS discharges in industrial wastewater discharges. EPA's acceptance criteria are presented in the memorandum "Development of the Multi-Industry PFAS Study Analytical Database" (ERG, 2021a). EPA identified four sources of PFAS analytical data for pulp, paper, and paperboard effluent:

- MI ELGE 2020 PFAS monitoring results for direct and indirect discharge facilities (MI EGLE, 2020b, 2020c).
- 2019 study of PFAS in industrial, municipal, and landfill leachate discharges commissioned by the Vermont Department of Environmental Conservation (VT DEC, 2020).[12]
- PFOA and PFOS sampling results for Georgia-Pacific facilities located in Green Bay, Wisconsin (EPA, 2021h).
- AF&PA summary of PFAS concentrations in effluent from six unidentified pulp and paper mills, originally collected by the National Council for Air and Stream Improvement (AF&PA, 2020c).

EPA determined that the AF&PA submission did not meet all of EPA's acceptance criteria for inclusion in analyses for characterizing PFAS in pulp, paper, and paperboard facility discharges because results were not reported as individual or average concentration results. EPA determined that all individual sample results in the other three data sources did meet EPA's acceptance criteria. EPA included 358 PFAS sample results representing 23 facilities from these sources in its analysis characterizing PFAS in pulp, paper, and paperboard effluent.

EPA calculated facility-level average, minimum, and maximum concentrations for each PFAS with available data using the following assumptions and limitations:

- EPA assumed all nondetection results and results below the level of quantification (i.e., the reporting or quantification limitation) were zero.[13]
- EPA did not have information on analytical method sensitivity and level of quantification for all data.
- EPA did not know the operations or treatment processes online at the facilities at the time of sampling.

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[12] AF&PA excluded the PFAS sample results from this 2019 study from its data submittal to EPA. AF&PA and the National Council for Air and Stream Improvement asserted the data in the report "may be imprecise as evidenced by the high degree of variability" and "split samples collected by one of the facilities and analyzed at a separate laboratory showed much lower concentrations" (AF&PA, 2020c). Based on discussions with WestRock Company and the Vermont Department of Environmental Conservation, EPA determined the effluent data for the two pulp, paper, and paperboard facilities in the report met EPA's acceptance criteria and were of sufficient quality for a preliminary review of PFAS concentrations in industry discharges (EPA, 2021j; VT DEC, 2021).

[13] The lower level of quantification is the lowest concentration that the analytical method being used can measure accurately.

- EPA does not have information on whether the observed PFAS concentrations are from legacy use of PFAS, current use of PFAS, or form from degradation of more complex PFAS.

EPA calculated an overall average, minimum, and maximum concentration for each PFAS based on facility-level results. EPA did not estimate average concentrations for any PFAS that were not detected at or above the level of quantification across all facilities or did not have any data.

Table 11 presents the average, minimum, and maximum concentrations for each PFAS observed in effluent from the 23 pulp, paper, and paperboard facilities. As illustrated in the table, EPA estimated the average concentrations for 6:2 FTSA and short-chain PFCAs (both degradation products of FDA-approved PFAS used in food packaging) were generally higher relative to PFSAs and long-chain PFCAs. Despite the phase out of long-chain PFAAs, some pulp, paper, and paperboard facilities still report detectable levels of PFOA and PFOS in their wastewater.

Table 11. Pulp, Paper, and Paperboard Wastewater PFAS Concentrations

PFAS Subgroup	Analyte [a b]	Facilities with Data	Quantified Detections/Total Sample Results	Concentration Range (µg/L) [c]	Average Concentration (µg/L) [c]
PFCAs	PFBA	2	8/8	0.149 – 0.638	0.377
	PFPeA	2	8/8	0.0308 – 0.246	0.145
	PFHxA	2	8/8	0.0841 – 0.64	0.250
	PFHpA	2	8/8	0.0235 – 0.206	0.118
	PFOA	23	55/79	ND – 0.68	0.0377
	PFNA	2	8/8	0.00592 – 0.0526	0.0235
	PFDA	2	4/8	ND – 0.0197	0.00501
	PFUnA	2	4/8	ND – 0.0153	0.00441
	PFDoA	2	4/8	ND – 0.0203	0.00496
	PFTrA	2	4/8	ND – 0.0249	0.00579
	PFTeA	2	4/8	ND – 0.023	0.00493
PFSAs	PFBS	2	4/8	ND – 0.254	0.0343
	PFPeS	2	0/8	ND	ND
	PFHxS	2	0/8	ND	ND
	PFHpS	2	0/8	ND	ND
	PFOS	23	56/80	ND – 0.41	0.0318
	PFNS	2	1/8	ND – 0.00217	0.000271
	PFDS	2	1/8	ND – 0.00517	0.000646
FASAs	PFOSA	2	0/8	ND	ND
FTSAs	4:2 FTSA	2	0/8	ND	ND
	6:2 FTSA	2	7/8	ND – 0.284	0.0691
	8:2 FTSA	2	0/8	ND	ND
FASAAs and N-Alkyl FASAAs	NMeFOSAA	2	0/8	ND	ND
	NEtFOSAA	2	0/8	ND	ND

Sources: ERG, 2021b.

Abbreviations: ND – nondetection; µg/L – micrograms per liter.

a – This table presents data for all PFAS listed in Table 2 for which sample results are available and meet EPA's acceptance criteria. EPA does not have any sample results for PFAS not listed.

b –The table identifies short-chain PFCAs (≤7 carbons) and short-chain PFSAs (≤5 carbons) in blue text, while long-chain PFCAs (≥8 carbons) and long-chain PFSAs (≥6 carbons) are designated in red text.

c – In this analysis, EPA treated all nondetection results as zero for the purpose of estimating concentrations. All concentration values are rounded to three significant figures.

7.5 Pulp, Paper, and Paperboard Point Source Category Summary

Based on information and data EPA collected for the Multi-Industry PFAS Study, EPA documented that PFAS have been, and continue to be, used by pulp, paper, and paperboard facilities in the United States; however, only a small subset of facilities are actively applying PFAS and it appears the production of paper products containing PFAS at these facilities is a small percentage of the overall production. Additionally, the industry has indicated they plan to eliminate PFAS use by 2024. Information collected from one trade association and eight major companies indicates the industry phased out the use of PFOA and PFOS approximately 10 years ago, but continues to use FDA-approved short-chain PFAS in limited quantities in the manufacture of food contact packaging and specialty paper products. PFAS may be in additives mixed with the pulp prior to forming it into paper or in coatings applied to the surface of the paper after the paper is formed to enhance resistance to water, oil, and grease. Based on outreach and a trade association survey of companies representing 85 percent of United States production, EPA identified 10 pulp, paper, and paperboard facilities operated by six companies that have applied PFAS since 2020. The PFAS-based production volume of these 10 mills represents less than 10 percent of their total production and less than 1 percent of the total produced by this industry in the United States. The companies operating all 10 facilities reported to EPA that they will transition to PFAS-free technologies and eliminate all application of PFAS in their United States pulp and papermaking operations by 2024. This schedule coincides with an FDA agreement with chemical manufacturers to voluntarily phase out use of PFAS that contain or may degrade to 6:2 FTOH in food contact applications by 2024.

EPA did not identify any pulp, paper, and paperboard facilities with PFAS effluent limitations or pretreatment standards in their wastewater discharge permit and estimates that only a small fraction of pulp, paper and paperboard facilities monitor for PFAS. EPA did not identify any pulp, paper, and paperboard facilities employing wastewater treatment systems known to effectively reduce PFAS in industrial wastewater. Although industry reports the application of PFAS to pulp, paper, and paperboard products is typically a dry or closed-loop process and may not generate a wastewater stream, EPA documented PFAS, including legacy long-chain PFAS and replacement PFAS, are present in wastewater discharges from pulp, paper, and paperboard facilities to surface waters and POTWs. Using available sampling data, EPA estimated average concentrations for 6:2 fluorotelomers and short-chain PFCAs (both degradation products of FDA-approved PFAS used in food packaging) were generally higher relative to PFSAs and long-chain PFCAs. The presence of PFOA, PFOS, and other long-chain PFAAs may be due to legacy issues or degradation of other more complex PFAS.

8. Review of the Textile Mills Point Source Category

This section describes the textile mills point source category and information and data that EPA collected on its PFAS use and discharge. EPA collected and reviewed information on PFAS use and discharge by textile mills from the following sources:

- Outreach to textile manufacturing companies, carpet manufacturing companies (a subset of the category), industry trade associations, and state and local wastewater authorities.
- Publicly available technical literature.

8.1 Industry Description and Use of PFAS

Textile mills receive and prepare fibers; transform fibers into yarn, thread, or webbing; convert yarn and webbing into fabric or related products; or finish these materials. Many facilities produce a final consumer product such as thread, yarn, fabric, hosiery, towels, sheets, and carpet while the rest produce an intermediate product for use by other establishments in the industry. As part of EPA's 1996 *Preliminary Study of the Textile Mills Category*, EPA estimated that approximately 6,000 establishments in the United States manufactured textile products. The majority of United States textile mills, including carpet manufacturers, are concentrated in the southeastern United States (EPA, 1996). The city of Dalton, Georgia contains over 150 carpet manufacturing plants, and more than 90 percent of the world's carpeting is produced within a 65-mile radius of the city (Town of Centre AL v. Dalton GA Manufacturers, 2017).

EPA promulgated the Textile Mills Effluent Guidelines (40 CFR Part 410) in 1974, with technical amendments in 1977 and 1982. The regulations cover wastewater discharges generated from textile mills using the following processes:

- Wool scouring, topmaking, and general cleaning of raw wool.
- Wool finishing, including carbonizing, fulling, dyeing, bleaching, rinsing, and fireproofing.
- Yarn manufacture, unfinished fabric manufacture, fabric coating, fabric laminating, tire cord and fabric dipping, carpet tufting, and carpet backing.
- Woven fabric finishing, including desizing, bleaching, mercerizing, dyeing, printing, resin treatment, waterproofing, flameproofing, application of soil repellent, and other special finishes.
- Knit fabric finishing, including bleaching, mercerizing, dyeing, printing, resin treatment, waterproofing, flameproofing, application of soil repellent, and other special finishes.
- Carpet finishing, including bleaching, scouring, carbonizing, fulling, dyeing, printing, resin treatment, waterproofing, flameproofing, application of soil repellent, looping, and backing with latex and jute.
- Stock and yarn finishing, including cleaning, scouring, bleaching, mercerizing, dyeing, and finishing.
- Manufacturing of nonwoven textile products of wool, cotton, synthetics, or blends of such fabrics.

The current ELGs do not establish effluent limitations or pretreatment standards for PFAS.

Textile mills use PFAS to impart outdoor gear, clothing, household fabrics, carpets, and other textile products with water, oil, soil, and heat resistance; improve cleanability of oil- and water-based stains; as a wetting or antifoaming agent when dyeing and bleaching, and as a breathable moisture barrier to wind and rain (NCTO, 2016; Wood, 2020b; Glüge et al., 2020). Some textile products that may contain PFAS include consumer apparel and accessories, professional apparel (including medical and firefighter uniforms and personal protection equipment), sportswear, outdoor gear, heat-resistant gloves, footwear, carpeting and rugs, backpacks, swimwear, and upholstery (NRDC, 2021; SAICM, 2021). Fluoropolymer PFAS are most commonly used as breathable membranes, while side-chain fluorinated polymers are used as long-lasting durable water repellent finishes (Wood, 2020b; Glüge et al., 2020). During fabric and carpet manufacturing, PFAS can either be incorporated as an additive mixed into the individual fibers or sprayed as a coating onto finished fabrics, either during manufacturing or after sale (GSPI, 2021).

A National Resources Defense Council (NRDC) analysis of a 2016 fluorotelomer market study concluded the global textile industry was the largest user of fluorotelomers (relative to the volumes used in firefighting foams, food packaging, stain resistance chemicals, and other products), making up approximately 36 percent of the total market (NRDC, 2021; Ahuja and Mamtani, 2016).

Some retailers and textile companies have committed to eliminating the sale or manufacture of PFAS-containing textile products in the coming years, including Interface, Tarkett, IKEA, Herman Miller, Crate and Barrel, Room and Board, Engineered Floors, Lowes, and Home Depot. Fashion, apparel, and home textile brands are currently the most common adopters of PFAS-free commitments. Sports and outdoor brands face more challenges to phase out PFAS while keeping the current level of product performance and functionality because there are no technologies which can repel oil-based materials to the same levels that PFAS achieve, and which are acceptable to the industry. (NRDC, 2021; NCTO, 2018; GSPI, 2021). At least one state, California, is in the process of regulating PFAS in carpets, rugs, and after-market treatments. The major categories of nonfluorinated water-repellant alternatives available on the market include hydrocarbons, silicones, dendrimers, polyurethanes, and nanomaterials (Wood, 2020b).

8.2 Stakeholder Outreach

EPA attempted to meet with industry stakeholders and state and local wastewater authorities to collect, on a voluntary basis, information on the use and discharge of PFAS for textile and carpet mills.

EPA reached out to two trade associations and three textile or carpet manufacturing companies that the agency considered possible users of PFAS. The trade associations and companies that EPA contacted are listed below:

- **National Council of Textile Organizations (NCTO).** A national trade association representing more than 150 companies across the entire spectrum of the United States textile industry and comprising four councils: the fiber council, the yarn council, fabric and home furnishing council, and the industry support council.
- **Carpet and Rug Institute (CRI).** A trade association for the North American carpet industry, providing carpeting-related informational tools, programs, and research resources. CRI represents manufacturers producing 94 percent of carpet in the United States, suppliers of raw materials, and services to the industry.
- **Milliken and Company (Milliken).** Global manufacturer and supplier of household textiles (including carpets), performance and protective textiles, specialty chemicals, and other industrial textiles.
- **Shaw Industries Group, Inc. (Shaw).** Global manufacturer of carpets, rugs, and household textiles.
- **W.L. Gore and Associates (Gore).** Global manufacturer of industrial and commercial fluoropolymer-based products, including waterproof, breathable fabrics (e.g., GORE-TEX) used in apparel, footwear, workwear/technical wear, and outdoor textiles.

NCTO, CRI, and their member companies declined to meet with EPA and did not provide any information on PFAS use in the industry (EPA, 2021o, 2021p). Milliken informed EPA that they are not in a position to discuss PFAS due to ongoing litigation involving claims against multiple defendants related to alleged discharge of PFAS from carpet manufacturing facilities in and around Dalton, Georgia.[14] Milliken explained that the Milliken facility at issue in that litigation was operated by Milliken for only a short time, from October 2009 to November 2012. Milliken also stated that none of the current NPDES permits held by the company’s textile mills have any monitoring requirements for PFAS (EPA, 2021q). EPA did not receive responses or any additional information from Shaw or Gore (EPA, 2021r, 2021s).

8.3 PFAS Wastewater Regulatory Requirements and Controls

As part of the Multi-Industry PFAS Study, EPA did not identify any textile mills with PFAS effluent limitations, pretreatment standards, or monitoring requirements in their wastewater discharge permits. EPA identified a draft

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[14] The plaintiff of this litigation, the Water Works and Sewer Board of the City of Centre, brought a complaint against owners, operators, and/or chemical suppliers to manufacturing facilities (including textile and carpet manufacturers) located in and around Dalton, Georgia. The plaintiff claims that they have and continue to be damaged due to the past and present release of toxic chemicals, including PFOA, PFOS, precursors to PFOA and PFOS and related chemicals released during manufacturing operations (Town of Centre AL v. Dalton GA Manufacturers, 2017).

NPDES permit for one textile mill in Georgia which, if finalized, would require the company to determine if the facility has potential to release PFAS to the environment through discharge of wastewater effluent or industrial sludge disposal (GA DNR, 2020).

8.4 Wastewater Characteristics

EPA evaluated the available data on types and concentrations of PFAS in wastewater discharged from textile mills. As of July 2021, EPA has not identified any textile mills with PFAS monitoring requirements or effluent limitations; therefore, no DMR data is available for PFAS. EPA summarized the information available and calculated average PFAS concentrations in effluent from textile mills based on Michigan permitting authority data.

EPA identified analytical data that meet EPA's acceptance criteria for inclusion in analyses for characterizing PFAS discharges in industrial wastewater discharges. EPA's acceptance criteria are presented in the memorandum "Development of the Multi-Industry PFAS Study Analytical Database" (ERG, 2021a). EPA identified one source of analytical data of textile mill effluent and determined that all individual sample results in the source met EPA's acceptance criteria, MI EGLE PFAS monitoring results for indirect discharge facilities (MI EGLE, 2020c). EPA included 16 PFAS sample results representing three indirect discharge mills in its analysis characterizing PFAS in textile mill effluent.

EPA calculated facility-level average, minimum, and maximum concentrations for each PFAS with available data using the following assumptions and limitations:

- EPA assumed all nondetection results and results below the level of quantification (i.e., the reporting or quantification limitation) were zero.[15]
- EPA did not have information on analytical method sensitivity and level of quantification for all data.
- EPA did not know the operations or treatment processes online at the facilities at the time of sampling.
- EPA does not have information on whether the observed PFAS concentrations are from legacy use of PFAS, current use of PFAS, or formation from degradation of more complex PFAS.

EPA calculated an overall average, minimum, and maximum concentration for each PFAS based on the facility-level results. Table 12 presents the average, minimum, and maximum concentrations for each PFAS observed in effluent the three indirect discharge textile mills.

Table 12. Textile Mill Wastewater PFAS Concentrations

PFAS Subgroup	Analyte [a b]	Facilities with Data	Quantified Detections/Total Sample Results	Concentration Range (µg/L) [c]	Average Concentration (µg/L) [c]
PFCAs	PFOA	3	4/8	ND – 0.114	0.00807
PFSAs	PFOS	3	4/8	ND – 0.0361	0.00249

Source: ERG, 2021b.

Abbreviations: ND – nondetection; µg/L – micrograms per liter.

a – This table presents data for all PFAS listed in Table 2 for which sample results are available and meet EPA's acceptance criteria. EPA does not have any sample results for PFAS not listed.

b –The table identifies long-chain PFCAs (≥8 carbons) and long-chain PFSAs (≥6 carbons) in red text.

c – In this analysis, EPA treated all nondetection results as zero for the purpose of estimating concentrations. All concentration values are rounded to three significant figures.

8.5 Textile Mills Point Source Category Summary

Based on information and data EPA collected for the Multi-Industry PFAS Study, EPA documented that PFAS have been, and continue to be, used by textile mills in the United States. Textile mills use PFAS to enhance resistance to water, oil, soil, and heat; improve cleanability of oil- and water-based stains; as a wetting and antifoaming agency; and in the breathable moisture barrier to wind and rain. PFAS have been applied in a wide range of textiles

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[15] The lower level of quantification is the lowest concentration that the analytical method being used can measure accurately.

including but not limited to clothing, footwear, carpets and rugs, household fabrics, upholstery, medical garments, firefighting gear, luggage, and outdoor gear (e.g., jackets, hats, gloves, tents). During fabric and carpet manufacturing, PFAS can either be incorporated as an additive mixed into the individual fibers or sprayed as a coating onto finished fabrics during manufacturing or after sale.

EPA's review of PFAS use and discharge by the textile mills point source category is largely based on publicly available information and literature. EPA attempted to meet with representatives of industry trade associations and companies to collect, on a voluntary basis, information on the use and discharge of PFAS at textile and carpet mills. Ultimately, EPA did not meet or receive additional information from these entities.

EPA did not identify any textile mills with PFAS effluent limitations or pretreatment standards in their wastewater discharge permit and estimates that only a small fraction of textile mills monitor for PFAS. EPA did not identify any textile mills employing wastewater treatment systems known to effectively reduce PFAS in industrial wastewater. Based on a small number of sample results, EPA has observed that PFAS, including legacy long-chain PFAS, are present in wastewater discharges from textile mills to POTWs. Most textile mills are not monitoring for PFAS and may continue to discharge PFAS to POTWs or surface waters unless effective controls are in place.

9. Review of the Commercial Airport Point Source Category

This section describes the commercial airport point source category, the use of PFAS-containing firefighting foam (i.e., AFFF) for firefighting activities and certification exercises, and mechanisms for PFAS release to the environment. EPA collected and reviewed information on PFAS use and discharge by commercial airports from outreach with the United States Department of Transportation, Federal Aviation Administration (FAA) and an industry trade association; government reports and databases; a survey and report published by the Airport Cooperative Research Program (ACRP); and publicly available technical literature.

9.1 Industry Description and Use of PFAS

The FAA and Airport and Airway Improvement Act (AAIA) (49 U.S.C. Chapter 471) classify commercial airports by size based on volume of commercial traffic. Commercial airports are defined by the FAA and AAIA as publicly owned airports that have at least 2,500 passenger boardings (number of passengers boarding a plane for departure) each calendar year and receive scheduled passenger service (EPA, 2012b). As of April 2021, the FAA has certified 519 commercial airports (FAA, 2021a). Military installations and other facilities operated by the United States Department of Defense (DOD) are not considered commercial airports; therefore, PFAS use and discharge by DOD facilities are outside the scope of this study.

14 CFR Part 139 contains the regulations pertaining to certification of airports and requires commercial airports to conduct periodic testing of certain equipment and train personnel to perform aircraft rescue and firefighting (ARFF) operations. In 2006, the FAA required that commercial airports certified under 14 CFR Part 139 purchase only firefighting foams that conform to military specification (Mil-Spec) MIL-PRF-24385 for performance and procurement, which required that AFFF liquid concentrates contain fluorocarbon (i.e., PFAS) surfactants (FAA, 2006). In May 2019, the DOD amended Mil-Spec MIL-PRF-24385 to remove the Mil-Spec's requirement that AFFF contain fluorocarbon surfactants (DOD, 2019). However, as of June 2021, all firefighting foam formulations that meet MIL-PRF-24385 contain PFAS in concentrations less than 800 parts-per-billion (ACRP, 2017; ERG, 2020a).

AFFF is produced by mixing PFAS-containing concentrate with water at the specified proportion, typically 3 or 6 percent ratio to water. AFFF has been, and continues to be, stored and used at military installations, industrial facilities, petroleum refineries, and airports to prevent, extinguish, or control Class B flammable fuel fires and for firefighter training. When mixed with water, AFFF concentrate generates an aqueous film and foam solution that spreads across the surface of a hydrocarbon (e.g., grease, oil, gasoline, solvent) fire to extinguish the flames and form a vapor barrier separating fuel and atmospheric oxygen to prevent reignition. Military and commercial airport AFFF applications subject to the Mil-Spec MIL-PRF-24385 account for more than 75 percent of AFFF used in the United States (ACRP, 2017).

The FAA requires periodic testing of foam proportioning system performance in ARFF vehicles, as prescribed in 14 CFR Parts 139.315 to 139.319. The number of ARFF vehicles and amount of AFFF present at each airport is based on the length of aircraft and average number of daily departures from the airport. Until recently, foam proportioning system testing required airports to perform output-based testing, in which AFFF is sprayed from the ARFF vehicle for at least 30 seconds to demonstrate that the firefighting equipment operates correctly. During output-based testing, safeguards, such as capture containers, containment basins, absorbent pads, and use separator/scrubbing systems may be used to prevent the release of PFAS to the environment (ACRP, 2017; FAA, 2021b). 14 CFR Part 139 also permits input-based testing, a method that requires additional equipment but allows for a substitute (typically water) to be sprayed instead of AFFF. Input-based testing requires the establishment of a baseline by spraying foam from the ARFF vehicles. Once a baseline is established, the test can be run using water rather than foam. As of June 2021, the FAA has approved and encourages use of four different types of AFFF testing equipment that do not require dispensing AFFF. The FAA and some states are providing funding for the purchase of input-based testing equipment that does not require foam to be dispensed onto the ground (FAA, 2019a, 2019b, 2021b). Commercial airports that use input-based testing equipment will eliminate potential discharges of wastewater containing PFAS during periodic testing of foam proportioning system performance in ARFF vehicles.

The FAA also requires all airport firefighting personnel to complete an annual live-fire fighting training as dictated in 14 CFR Parts 139.315 to 139.319. Live firefighting training involves extinguishing a pit fire with an aircraft mockup using enough fuel to simulate the type of conditions that could be encountered during a rescue situation. If training of airport firefighting personnel does not occur within a 12-month period, an airport will be considered out of compliance with 14 CFR Part 139. Commercial airports are not required to use AFFF during live firefighting testing (water solutions or alternative methods may be used). Some airports have a designated firefighting training area to perform training, while others do not (ACRP, 2017).

Until application, AFFF is managed as a concentrated product containing less than 2 percent PFAS by weight. PFAS account for less than 1 percent of AFFF after the concentrate is mixed with water to create the firefighting solution. AFFF formulations are generally categorized into three groups, based on the PFAS type included:

- **Legacy PFOS-based AFFF.** First-generation AFFF formulations where PFOS is an active ingredient. Manufactured by 3M via electrochemical fluorination and sold under the brand name Light Water™ in the United States from 1970s to 2002.
- **Legacy fluorotelomer-based AFFF.** Second-generation AFFF formulations containing precursors to long-chain PFCAs (e.g., PFOA) and manufactured via fluorotelomerization in the United States from 1970s to 2016.
- **Modern fluorotelomer-based AFFF.** Modern AFFF formulations containing four- and six-carbon fluorotelomer chemistries (e.g., 6:2 FTSA) and developed in response to the PFOA Stewardship Program. These AFFF formulations are currently being commercially sold in the United States market.

While modern fluorotelomer-based AFFF formulations have the potential to be less harmful to human health and the environment than legacy formulations, much remains unknown about the short-chain PFAS used. Four- and six-carbon chain fluorotelomers degrade into short-chain PFCAs and other short-chain PFAS, as discussed in Section 3.4. Since certain short-chain PFAS are less effective surfactants than their long-chain counterparts, greater quantities of short-chain PFAS may be required to provide equivalent performance (ACRP, 2017).

PFAS contamination has been observed in surface water, groundwater and drinking water in proximity to airports that use AFFF (Hu et al., 2016; Gewurtz et al., 2014; ITRC, 2020). Due to growing concerns related to PFAS use and release at airports, the FAA Reauthorization Act of 2018 (enacted October 5, 2018) mandates that the FAA can no longer require the use of AFFF by 14 Part 139 airports no later than three years from the date of enactment (October 4, 2021). As a result, the FAA has approved, encourages use of, and, in some cases, funds technologies that do not require dispensing AFFF when airports conduct periodic equipment testing and training. While the FAA Reauthorization Act of 2018 will end the requirement for use of AFFF, it does not prohibit its use by commercial airports (FAA, 2018, 2019c, 2021b).

The National Defense Authorization Act of fiscal year 2020 (enacted December 20, 2019) requires the DOD to phase out its use of AFFF at all military installations by October 2024, with limited exceptions, and immediately stop military training exercises with AFFF. The Secretary of the Navy must publish specifications for PFAS-free firefighting foam at all military installations and ensure that the foam is available for use by October 2023 (ITRC, 2020). These mandates do not apply to commercial airports and the FAA has not yet announced plans to require exclusive use of PFAS-free formulations at commercial airports.

Despite discontinued manufacture of legacy AFFF formulations with long-chain PFAS chemistries, many airports have AFFF in service or in stockpiles and are not prohibited from using legacy or modern AFFF. AFFF has a long shelf life; some manufacturers claim PFAS-containing AFFF can remain viable up to 25 years if stored properly (ACRP, 2017). This means that PFAS-containing AFFF, including legacy PFOS-based products, still exist in United States inventories and ongoing permitted use of AFFF at commercial airports can still result in PFAS releases. The current volume of AFFF in commercial airport stockpiles or used annually is not known.

The DOD, states, and other organizations recommend the complete replacement of legacy AFFF and have launched proper disposal and take-back programs. As of January 2020, EPA has identified eleven states with AFFF procurement, use, storage, and/or disposal regulations: Arizona, Colorado, Georgia, Kentucky, Michigan, Minnesota, New Hampshire, New York, Virginia, Washington, and Wisconsin (ACRP, 2017; ERG, 2020a; Bloomberg Law, 2020).

Many firefighting foam manufacturers now offer Class B fluorine-free foam products. A 2020 literature review and market study found that fluorine-free alternative foams, including hydrocarbon- and detergent-based foams, are generally available and technically feasible, and have been successfully implemented in many industrial sectors in Europe (Wood, 2020c).

As of June 2021, the FAA has not identified any fluorine-free foams on the market that provide the same level of fire suppression, flexibility, and scope of usage as MIL-PRF-24385 AFFF and therefore they are not used at DOD- and FAA-regulated facilities. To aid in the transition to nonfluorinated AFFF, the FAA, the DOD, Airports Council International – North America, firefighting foam manufacturers/developers, and other organizations are researching and testing at least 15 commercially available fluorine-free AFFF alternatives to identify formulations that are environmentally friendly and provide the same level of safety currently offered by the Mil-Spec MIL-PRF-24385. The FAA has built a research testing facility and conducted over 400 tests in an effort to find a new fluorine-free alternative firefighting extinguishing agent (ERG, 2020a; FAA, 2019a, 2021b; SERDP, 2020).

9.2 Stakeholder Outreach

EPA met with the FAA and the Airports Council International – North America to collect, on a voluntary basis, information on the use and release of PFAS-containing AFFF by commercial airports. FAA representatives provided EPA with an overview of firefighting requirements for commercial airports to maintain 14 CFR Part 139 certification. EPA collected information on the Mil-Spec MIL-PRF-24385, FAA development and approval of input-based testing systems, and the FAA's guidance to commercial airports to control release of AFFF. EPA continues to coordinate with the FAA to understand the status of research on fluorine-free formulations and actions taken to address AFFF. The Airports Council International – North America is a trade association for the North American airport industry and provided EPA with information on its members' practices for using and capturing AFFF.

9.3 PFAS Wastewater Regulatory Requirements and Controls

As part of the Multi-Industry PFAS Study, EPA did not identify any commercial airports with PFAS effluent limitations, pretreatment standards, or monitoring requirements in their wastewater discharge permits.[16]

9.4 PFAS Releases Associated with AFFF Use

Commercial airports have historically generated PFAS-containing wastewater during periodic testing of ARFF equipment, live-fire firefighting training, emergency response activities, rinsing ARFF equipment, and accidental leaks. The volume of PFAS released to the environment can vary depending on the activity and types of controls employed by the airport. A 2016 survey of 167 airports across the United States and Canada indicated that nearly 80 percent of respondents sprayed AFFF directly on the ground rather than an engineered containment system. Most airports which reported directly spraying AFFF onto the ground also reported that AFFF was left to evaporate, dissipate, dilute, or infiltrate into the ground. Most airports that reported capturing AFFF in a containment system or cleaning up AFFF sprayed onto the ground ultimately discharged the solution to POTW via a sewer or to a surface water (ACRP, 2017). Most on-site airport wastewater treatment systems and POTWs are not capable of effectively removing PFAS. Once released, AFFF foam can contaminate soil, surface water, and groundwater.

To minimize AFFF releases, the FAA and Airports Council International – North America are working with airports to enhance education of AFFF issues; reduce AFFF release during periodic testing and training activities; and promote practices and technologies used for the capture of AFFF, and subsequent treatment at municipal wastewater treatment plants (ERG, 2020a; FAA, 2021b). Since 2019, the implementation of input-based testing and PFAS control processes and technologies have reduced AFFF releases by commercial airports. Releases to the environment will be further reduced based on federal actions to review and revise mandates to use PFAS-containing AFFF and proliferation of fluorine-free firefighting foams alternatives. The FAA states that approval of input-based testing and updated guidance has eliminated the need for commercial airports to discharge

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[16] The FAA requires commercial airports and air carriers to conduct deicing and anti-icing of aircraft and airfield pavement to ensure safety of flights. In 2012, EPA promulgated the Airport Deicing ELGs (40 CFR Part 449), which address control of wastewater generated by deicing. The ELGs do not apply to wastewater generated by AFFF use at commercial airports, nor do they establish PFAS requirements or effluent limitations (EPA, 2012b).

wastewater contaminated with AFFF except during actual emergency response situations (i.e., AFFF should not be released during periodic testing of ARFF equipment .

9.5 Commercial Airports Point Source Category Summary

The FAA Reauthorization Act of 2018 (enacted October 5, 2018) mandates that the FAA can no longer require the use of PFAS-based AFFF by 14 CFR Part 139 airports no later than three years from the date of enactment (October 4, 2021). As a result, the FAA has approved, encourages use of, and in some cases funds four different types of AFFF testing equipment that do not require dispensing AFFF when airports conduct periodic equipment testing and training (FAA, 2021b). The FAA has also built a research testing facility and has conducted over 400 tests in an effort to find a new fluorine-free alternative firefighting extinguishing agent (FAA, 2019b).

Historically, the FAA required that commercial airports certified under 14 CFR Part 139 purchase only firefighting foams that conform to Mil-Spec MIL-PRF-24385 for performance and procurement (FAA, 2006). In May 2019, the DOD amended Mil-Spec MIL-PRF-24385 to remove the requirement that AFFF must contain PFAS. As of July 2021, all firefighting foam formulations that meet MIL-PRF-24385 contain less than 800 parts-per-billion of PFAS. The FAA and the DOD are continuing to collaborate on research and to test fluorine-free alternatives that provide the same level of safety currently offered by Mil-Spec MIL-PRF-24385.

Based on this information, EPA determined that commercial airports may generate PFAS-containing wastewater from live-fire firefighting training, emergency response activities, and accidental leaks from stockpiles of AFFF. The volume of PFAS released to the environment can vary depending on the activity, types of controls employed by the airport, and type and volume of AFFF released.

10. Review of PFAS Treatment Technologies

This section summarizes information and performance data EPA collected on treatment technologies capable of removing or destroying PFAS in water streams. For the purposes of this preliminary report, PFAS destruction (sometimes referred to as mineralization) is the complete chemical degradation of PFAS molecules into base elements or compounds such as carbon dioxide (CO_2), water (H_2O), and fluorine ions (F^-). Incomplete destruction leaves behind partially degraded PFAS, resulting in increased concentrations of PFAAs or precursors. Removal is the physical separation of PFAS from an influent wastestream, but does not imply chemical transformation. Removal technologies result in PFAS being concentrated into another wastewater stream or solid waste.

EPA reviewed technical literature, Industrial Wastewater Treatment Technology (IWTT) database (EPA, 2021e), and EPA's Drinking Water Treatability Database (DWTD) (EPA, 2021f) to identify technologies capable of removing or destroying PFAS in industrial wastewater, drinking water, and municipal wastewater. The following treatment types are presented in this preliminary report:

- **Conventional Water Treatment.** Physical, biological, and chemical processes which are commonly applied in drinking water treatment plants (DWTPs) or POTWs to remove organic pollutants, solids, nutrients, and provide disinfection (see Section 10.1).
- **Adsorption.** Removal by transfer of contaminants from a liquid phase onto the surface of a solid adsorbent through hydrophobic partitioning or electrostatic interactions with active sites (see Section 10.2).
- **Membrane Filtration.** Removal of contaminants from a solution into a concentrated liquid wastestream using a selective barrier (see Section 10.3).
- **Incineration/Thermal Treatment.** Destruction by application of heat to break down the chemical structure of contaminants (i.e., breaking chemical bonds of PFAS molecules using extremely high temperatures) (see Section 10.4).
- **Advanced Oxidation and Reduction Processes.** Destruction using oxidizing or reducing agents and processes to break down the chemical structure of contaminants (i.e., breaking chemical bonds of PFAS molecules through a series of oxidation-reduction reactions) (see Section 10.5).
- **Emerging Technologies for PFAS Treatment.** Additional technologies being studied for PFAS removal or destruction but not yet widely implemented or demonstrated (see Section 10.6).

Table 13 summarizes demonstrated PFAS technologies identified through EPA's review. Where available, treatment capabilities reported in literature or EPA's DWTD are provided for six PFAS representing a range of chain length, functional group, and level of fluorination (PFOA, PFOS, PFBA, PFBS, 6:2 FTSA, HFPO-DA). See EPA's 2021 *Evaluation of Industrial Wastewater Treatment Technologies* report for additional information and data on these and additional PFAS treatment technologies (ERG, 2021c).

Table 13. Summary of Available PFAS Treatment Technologies

Treatment Technology	Treatment Description	Observed PFAS Removal Level [a]	Considerations for Use
Conventional Drinking Water and Wastewater Treatment	Water treatment processes commonly used by DWTPs or POTWs including filtration, coagulation, sedimentation, biological treatment, clarification, and disinfection.	Marginal reduction (< 25%) in concentration for most PFAS.	• PFAS removal limited to compounds adsorbed onto solids (i.e., dissolved PFAS are not removed). • May increase effluent concentrations of PFCAs and PFSAs through transformation of precursors.
Activated Carbon	Transfers PFAS from a liquid wastestream onto a solid powdered or granulated carbon-based adsorbent. Includes granular activated carbon (GAC) and powdered activated carbon (PAC).	PFOA: Up to 99% [a] PFOS: Up to 99% [a] PFBA: Up to 99% [a] PFBS: Up to 99% [a] HFPO-DA: Up to 93% [a] 6:2 FTSA: Up to 88% [a]	• Short-chain PFAS have lower removal rates than long-chain PFAS. • PFCAs have lower removal rates than PFSAs. • Sorption rates sensitive to water solution chemistry (e.g., greater pH or higher organic content of wastewater is linked to lower sorption rates). • Requires thermal regeneration or disposal of spent adsorbent media. • GAC is commercially available and has been implemented at OCPSF and chromium plating facilities to capture PFAS.
Ion Exchange Resin	Synthetic resins used to remove charged PFAS. Can be used in batch or flow-through reactors.	PFOA: Up to 99% [a] PFOS: 90-99% [a] PFBA: Up to 99% [a] PFBS: Up to 99% [a] HFPO-DA: Up to 99% [a] 6:2 FTSA: Up to 99% [a]	• Can be tailored to target electrostatically charged PFAS. • PFAS selective resins are more expensive but demonstrate higher removal capacities than activated carbon treatment for certain PFAS. • Rate of exchange depends on PFAS type, influent PFAS concentration, resin properties, and solution ionic strength. • Requires chemical generation or disposal of spent resin. Single-use resins create a solid waste stream onto which PFAS is absorbed. Regeneration of a reusable resin with a chemical solution generates a concentrated PFAS liquid wastestream. Regenerable resin cannot be infinitely regenerated and will create a solid wastestream onto which PFAS is adsorbed. • Commercially available for wastewater treatment.
Membrane Separation	Separation treatment that pushes water molecules through a semi-permeable membrane while rejecting larger PFAS molecules. Includes nanofiltration (NF) and reverse osmosis (RO).	PFOA: Up to 99% [a] PFOS: Up to 99% [a] PFBA: Up to 99% [a] PFBS: Up to 99% [a] HFPO-DA: Up to 99% [a] 6:2 FTSA: Up to 99% [a]	• Higher capital cost and energy demand than conventional treatments or adsorption. • Effective in removing most PFAS from water solutions. • Susceptible to fouling without pretreatment. • Generates a concentrated PFAS wastestream that must be treated or disposed.

Table 13. Summary of Available PFAS Treatment Technologies

Treatment Technology	Treatment Description	Observed PFAS Removal Level [a]	Considerations for Use
Incineration/ Thermal Treatment	Process of applying high temperatures to chemically break down PFAS molecules.	Complete PFAS destruction at temperatures ranging 200 – 1,400°C (varies from one PFAS to another).	• Can be used to regenerate solid adsorbents, such as GAC, while also destroying PFAS. However, high heat required to break carbon-fluorine bond can destroy adsorbent as well. • Incomplete destruction of PFAS may result in increased PFAA and precursor concentrations.
Advanced Oxidation/ Reduction Processes	Use of chemical or electrochemical catalyst to break down PFAS molecules.	Up to 99% PFAS destruction.	• Requires high energy or chemical catalyst input to initiate reactions. • Pretreatment to create a concentrated PFAS influent will reduce energy demand. • Incomplete destruction of PFAS may result in increased PFAA and precursor concentrations. • Advanced reduction requires strong alkaline systems.

a – Potential removal rates are based on reported data from EPA’s DWTD for PFAS. See the DWTD for removal rates for additional PFAS (EPA, 2021f).

10.1 Conventional Treatment Technologies

Treatment methods commonly found in DWTPs include coagulation, sand or multimedia filtration, and disinfection involving ultraviolet light or chemicals. POTWs typically treat wastewater using primary screening, sedimentation, secondary biological treatment (e.g., suspended growth or fixed-film biological processes), clarification, filtration, and/or disinfection. These conventional treatment processes used in POTWs and DWTPs do not degrade the carbon-fluorine bond and are ineffective at removing PFAS. No or inconsistent removal of PFAS has been observed, with most studies reporting less than 25 percent removal of total PFAS. PFAS removal for these treatments is limited to physical removal of PFAS bound to filtered solids, leaving behind dissolved PFAS (Appleman et al., 2014; Rahman et al., 2014; EPA, 2021f). Nonpolymer polyfluorinated PFAS and polymer PFAS may be partially degraded in drinking water or wastewater treatment processes, leading to increased PFAA detections in effluent and sludge (Pan et al., 2016; Hamid and Li, 2016).

10.2 Adsorption

Adsorption is a demonstrated process for contaminant removal in water and wastewater and is the most common treatment method for PFAS. Adsorption is both a physical and chemical process that removes a compound in an aqueous solution (adsorbate) through association to a solid phase (adsorbent). Adsorption does not chemically alter or destroy PFAS; rather, compounds are transferred from the liquid phase to a solid when they adhere to the solid’s active sites.

PFAS adsorption rates may be affected by pH, organic co-contaminant nature and concentration, and the ionic strength of the solution. Because adsorption processes can remove a wide spectrum of organic contaminants, the presence of nontargeted contaminants can increase competition for sorption sites, thus reducing removal of PFAS (Gagliano et al., 2020). Pretreatment steps may be necessary to optimize the performance of media, including coagulation, precipitation, filtration, pH adjustment, or oxidant removal.

Adsorption technologies require further treatment or disposal of the spent adsorbent media. Once adsorptive media is exhausted and breakthrough (i.e., PFAS is observed at a specific concentration in the effluent) occurs, the adsorbent media is considered spent and must be replaced or reactivated using high temperatures or chemical regenerants to renew adsorptive capabilities. Reactivation can create concentrated PFAS separate wastestreams from regenerant concentrate or through incineration gas emissions. Once an adsorbent can no longer be reactivated, it must be disposed of as a solid waste.

The following sections outline different adsorbents that rely on physical adsorption of PFAS.

10.2.1 Activated Carbon

Activated carbon is a widely used adsorbent for contaminant treatment. Granular activated carbon (GAC) and powdered activated carbon (PAC) are carbonaceous media that can be used to adsorb natural and synthetic organic compounds. Activated carbon treatment is available, relatively inexpensive, and can be scaled to suit treatment requirements.

GAC and PAC differ in the diameters of the activated carbon particles (1.2 to 1.6 millimeters for GAC, approximately 0.1 millimeter for PAC). Because of the small particle size, PAC cannot be used in a flow through bed, but can be added directly to the water and then removed in the clarification stage (conventional water treatment or low-pressure membranes such as microfiltration or ultrafiltration). Used in this way, PAC is not as efficient or economical as GAC at removing PFAS.

The application of GAC as a treatment technology for PFAS removal has been practiced for more than a decade at industrial sites, military installations, and DWTPs. GAC media regeneration requires heating the spent material to temperatures greater than 1,000 °C and regenerated GAC may be less effective than virgin GAC (Watanabe et al., 2016).

GAC and PAC performance for PFAS treatment has been documented with bench, pilot, and full-scale studies reporting up to 99 percent removal of PFAS depending on the compound being treated (Zhao et al., 2011; Ross et al., 2018). Studies have shown that PFSAs are more readily adsorbed than PFCAs, and long-chain PFAS are more readily adsorbed than short-chain PFAS (Appleman et al., 2014; Ross et al., 2018; EPA, 2021f). Increased pH and organic matter content in the adsorbate can decrease PFAS adsorption rates.

10.2.2 Ion Exchange Resins

Ion exchange (IX) technology removes charged contaminant ions using exchange sites on synthetic, highly porous resins in batch or continuous flow reactors. The charged resin sites attract and bind to oppositely charged contaminant ions.

Most PFAAs are present in environmental matrices in their anionic form and may be removed from water by anion exchange resins. Sorption rates will vary based on the resin and porosity. Unlike activated carbon, IX resins can be specialized to selectively target specific PFAS, require less contact time, and remove higher PFAS loads than GAC or PAC.

While IX technology has been used for decades, the development and use of selective resins for PFAS removal is relatively new. IX resin options for removal of PFAS include single-use and regenerable resins. Single-use resins are used until breakthrough occurs at a preestablished threshed and are then removed from the treatment unit. Regenerable IX resins may be regenerated on site using regenerant solution once active sites have been occupied but may not offer the same removal efficiency as single-use resins and cannot be infinitely regenerated. Resin regeneration creates a concentrated PFAS liquid wastestream that must be further treated or disposed. Continuous flow studies of IX resins for PFAS removal report that breakthrough occurs for PFCAs before PFSAs and short-chain PFAS before long-chain PFAS (Boyer et al., 2021). Bench- and pilot-scale studies captured in EPA's DWTD report PFAS removals from 30 to 99 percent (EPA, 2021f).

10.2.3 Other Adsorbents for PFAS Removal

EPA identified several other adsorbents that have demonstrated an ability to remove PFAS, listed below. As with activated carbon and IX resins, the properties of the adsorbent and the wastestream impact the amount of PFAS that can be removed.

- **Polymer adsorbents.** Synthetic materials that can be designed with specific traits suitable for the targeted removal of specific PFAS. Polymer adsorbents may have high hydrophobicity or an electrostatically charged surface to increase PFAS removal. Some polymer adsorbents may be regenerated and studies show regeneration can occur under much lower temperatures compared to GAC, allowing for less damage to the adsorbent. Some polymer adsorbents, such as crosslinked cyclodextrin polymers and cationic hydrogels, demonstrate more than 90 percent removal of long-chain PFAS and more than 80 percent removal of short-chain PFAS (greater sorption of short-chain PFAS than GAC) (Xiao et al., 2019; Ateia et al., 2019; EPA, 2021f).
- **Modified mineral adsorbents.** Mineral sorbents that have been modified using organic additives to increase PFAS sorption, such as organically modified silica and organoclays (Stebel et al., 2019). Bench-scale studies have observed more than 90 percent removal of total PFAS, with higher PFSAs removal rates than PFCAs.
- **Biochar.** Carbonaceous material derived from biomass. Biochar requires less energy to generate than activated carbon but has slower adsorption kinetics and lower observed PFAS removal relative to GAC. Short-chain PFAS are not readily removed using biochar (Xiao et al., 2017; ITRC, 2020).

10.3 Membrane Filtration

Membrane filtration is a physical separation process used for removal of both organic and inorganic compounds in water. A driving force is applied to the influent stream to push pressurized water through a semi-permeable membrane while rejecting larger, undesirable contaminants. Treated water (permeate) passes through the membrane and the rejected water (concentrate) is collected for treatment or disposal. All membrane processes generate a concentrated PFAS liquid wastestream that must be further treated or disposed. Membranes may also need to be replaced or disposed, generating a solid waste product. The following types of membranes are well-studied for PFAS removal:

- **Reverse Osmosis (RO).** RO is a form of membrane filtration in which pressure is applied to transport liquid through a membrane with a pore size of less than 1 nanometer. RO can be run as a continuous flow or batch process. EPA's DWTD reports that RO typically achieves at least 98 percent removal of PFAS regardless of chain length or functional group (EPA, 2021f). One full-scale treatment study has reported between 67 and 97 percent removal of total PFAS using RO (Glover et al., 2018).
- **Nanofiltration (NF).** NF is a membrane process that is lower in pressure than RO in which the membrane has pore sizes between 1 and 10 nanometers. Nanometer-sized membrane pores are used to remove compounds in a process similar to RO, but NF allows smaller PFAAs and salt ions to pass through which would otherwise be captured by the smaller pore sized used in an RO system. Lab-scale studies have shown nanofiltration removal of PFAS up to 90 percent (Boo et al., 2018).
- **Low Pressure Membrane Filters.** Ultrafiltration and microfiltration, two additional types of membrane filters with pore sizes larger than 10 nanometers, are less effective at capturing nonpolymer PFAS and are typically used for particulate removal. Sampling at full scale DWTPs using microfiltration or ultrafiltration has shown no or inconsistent removal (typically less than 50 percent) of PFCAs and PFSAs (EPA, 2021f), unless a powdered adsorbent is used within the system.

Wastestreams may need to go through a pretreatment step to reduce the risk of membrane damage or fouling (loss of production capacity) due to accumulation of material on the membrane surface.

10.4 Incineration/Thermal Treatment

Thermal treatment or incineration is using high temperatures to chemically break down PFAS. Incineration has been used to destroy other halogenated organic chemicals such as polychlorinated biphenyls (PCBs) and ozone-depleting substances, where sufficiently long exposures to sufficiently high temperatures break the carbon-halogen bond, after which the halogen can be scrubbed from the flue gas, typically as an alkali-halogen (EPA, 2019c). These treatments can be used for AFFF and solid wastes onto which PFAS has adsorbed, such as spent GAC or sludge, but may also be applied to PFAS-containing wastewater. However, PFAS are more difficult to break down than other halogenated organic chemicals due to fluorine's electronegativity and the chemical stability of fluorinated compounds.

Incinerators or combustors that are already in place for hazardous or municipal waste destruction may be used to destroy PFAS (Watanabe et al., 2016; EPA, 2020e). Incomplete destruction of PFAS during combustion can result in the formation of smaller PFAS or mixed halogenated organic byproducts, referred to as products of incomplete combustion (PICs) (EPA, 2019c, 2020f).

The effectiveness of incineration to destroy PFAS and the tendency for formation of PICs is not currently well understood. Few experiments have been conducted under oxidative and temperature conditions representative of field-scale incineration. Limited studies on the thermal destructibility of fluorotelomer-based polymers found no detectable levels of perfluorooctanoic acid after 2 second residence time at 1,000°C (Yamada et al., 2005; Taylor et al., 2014). Emission studies, particularly for PICs, have been incomplete due to lack of necessary measurement methods suitable for the comprehensive characterization of fluorinated and mixed halogenated organic compounds. EPA is actively researching the effective destruction temperatures and treatment times for PFAS, the potential to generate PICs, and the release and potential land deposition of PFAS-containing stack gases.

10.5 Advanced Oxidation and Reduction Processes

Advanced oxidation and reduction as methods for destruction of PFAS have been studied more in recent years. Through a series of oxidation and reduction reactions, PFAS molecules are defluorinated, decreasing the fluorinated carbon chain length until the PFAS molecules are degraded into base components such as CO_2, H_2O, and F^-. Full destruction is achieved when only the base components remain. Because PFAS are destroyed through these processes, PFAS are eliminated from wastewater rather than being captured via adsorption or membrane filtration. If PFAS molecules are not fully destroyed through these reactions, effluent concentrations of PFAAs and precursors can increase.

Most advanced oxidation and reduction processes do not generate a liquid or solid waste that would need to be managed.

10.5.1 Advanced Oxidation Processes

Advanced oxidation processes (AOPs) treat water by activating an oxidizing agent to react with and degrade contaminants. Chemical oxidation uses chemical catalysts to initiate degradation reactions. Chemical catalysts that have been studied for PFAS destruction include ozone, zero valent iron, and persulfate (Mitchell et al., 2013; Dai et al., 2019; Lee et al., 2020). Electrochemical oxidation uses electrical currents generated by specialty electrodes to catalyze oxidation reactions. Boron doped diamond electrodes, metal-oxide electrodes, and porous electrode membranes have been studied for PFAS destruction and studies report PFAS mass reductions ranging from 71 to 99 percent (Gomez-Ruiz et al., 2017; Le et al., 2019; AECOM, 2020; EPA, 2021w).

EPA's PFAS Innovative Treatment Team (PITT) identified electrochemical oxidation and supercritical water oxidation (SWCO) as two noncombustion PFAS destruction treatments for further research and consideration (EPA, 2021w, 2021x). SWCO catalyzes rapid oxidation reactions by applying heat greater than 705 °C and pressure greater than 221.1 bar to an aqueous solution or solid. In the presence of an oxidizing agent, supercritical water dissolves and oxidizes PFAS. SWCO has demonstrated up to 99 percent destruction of targeted PFAS in diluted AFFF, membrane concentrate, and landfill leachate by three vendors (EPA, 2021x). PITT further reports that SWCO is a potential solution for treatment of spent GAC and IX resin.

AOPs are nonselective, oxidizing all available contaminants. Therefore, the presence of nonPFAS oxidizable compounds in the influent may increase competition and reduce PFAS removal efficiency (particularly with chemical oxidation). Removing nonPFAS oxidizable compounds prior to AOP reduces treatment time and the amount of oxidant needed to destroy PFAS, increasing treatment performance (Ross et al., 2018).

10.5.2 Advanced Reduction Processes

Advanced reduction processes (ARPs) use the same reaction mechanisms as AOPs but use positively charged radicals to initiate reduction reactions to degrade PFAS rather than oxidation reactions. Strong alkaline systems are required to initiate reduction reactions. Lab-scale studies have shown between 70 percent and 99.9 percent destruction of PFAS at pH9 to pH12 using ARPs (Qu et al., 2014; Bentel et al., 2020).

10.6 Emerging PFAS Treatment Technologies

Table 14 presents PFAS treatment technologies that are in earlier stages of research and development. Some of these technologies build on treatment mechanisms outlined in the previous sections. There are limited data available on applicability and scalability of these treatments.

Table 14. Emerging PFAS Destruction Technologies

Treatment	Treatment Description	State of Research	Reference
Aqueous Electrostatic Concentrator	Combined use of IX membrane and electrodes to separate PFAS from solution and initiate oxidation reactions.	Lab-scale study of the patented technology reports 99% removal of both PFOA and PFOS.	Jackson, 2019
Bismuth Oxyhydroxyphosphate (BOHP)	Photocatalytic process in which BOHP ($Bi_3O(OH)(PO_4)_2$) is activated by ultraviolet light to degrade PFAS through oxidation or reduction reactions.	A pilot-scale study from the DOD's Strategic Environmental Research and Development Program (SERDP) reports up to 95% destruction of PFCAs and 90% degradation of fluorotelomers.	Sahu et al., 2018 Cates, 2020
Boron Nitride Oxidation	Use of activated boron nitride and ultraviolet light to degrade compounds.	One lab-scale study reports 99% removal of PFOA and 20% removal of HFPO-DA.	Duan et al., 2020
Electron Beam (E-beam)	Use of an accelerator to generate a stream of highly energetic electrons that are bombarded onto contaminated water, initiating both reduction and oxidation reactions.	Reports from SERDP state E-beam technology reduced PFOA and PFOS concentrations by up to 99.99% in soil samples and up to 87.91% in groundwater samples.	Pillai, 2020
Enhanced Contact Plasma Reactors (ECPR)	Plasma-based water treatment uses electricity to convert water into a mixture of highly reactive species (i.e., plasma) that rapidly and nonselectively degrade PFAS.	Lab-scale studies report up to 99% removal of PFAS for lab-prepared solutions and landfill leachate samples.	Singh et al., 2019, 2021
Mechanochemical Degradation	Destruction method using a high-energy ball-milling device and co-milling reagents to produce localized high temperatures and radicals that break down contaminants.	One lab-scale study reports 99% destruction of target PFAS in AFFF-impacted soil. Identified by EPA's PITT as a potential noncombustion destruction method for PFAS that would not require high temperatures or solvents.	EPA, 2021y
Pyrolysis and Gasification	Thermal treatment that decomposes materials at moderately elevated temperatures in oxygen free or very low oxygen environments. Used to transform biosolids into biochar and hydrogen-rich synthetic gas.	Limited data available on PFAS destruction. Identified by EPA's PITT as a potential noncombustion destruction method for PFAS in biosolids.	EPA, 2021z
Sonochemical Oxidation/ Ultrasound	Use of sound waves to facilitate cavitation in water which in turn releases large amounts of thermal energy and hydroxyl radicals to initiate PFAS degradation reactions.	One lab-scale study reports 90% destruction of PFOS. Identified by EPA's PITT as a potential noncombustion destruction method for PFAS in biosolids.	Wood et al., 2020

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United States
Environmental Protection
Agency

PFAS Strategic Roadmap: EPA's Commitments to Action 2021–2024

A Note from EPA Administrator Michael S. Regan

For far too long, communities across the United States have been suffering from exposure to PFAS pollution. As the science has continued to develop, we know more now than ever about how PFAS build up in our bodies over long periods of time, and how they can cause adverse health effects that can devastate families. As Secretary of the North Carolina Department of Environmental Quality, I saw this devastation firsthand. For years, the Cape Fear River had been contaminated by these persistent "forever" chemicals. As I spoke with families and concerned citizens, I could feel their suffering and frustration with inaction. I knew my job was going to be trying and complex. But we were able to begin to address this pervasive problem by following the science, following the law, and bringing all stakeholders to the table.

As one of my earliest actions as EPA Administrator, I established the EPA Council on PFAS and charged it with developing an ambitious plan of action to further the science and research, to restrict these dangerous chemicals from getting into the environment, and to immediately move to remediate the problem in communities across the country. EPA's PFAS strategic roadmap is our plan to deliver tangible public health benefits to all people who are impacted by these chemicals—regardless of their zip code or the color of their skin.

Since I've been EPA Administrator, I have become acutely aware of the invaluable and central role EPA has in protecting public health in America. For more than 50 years, EPA has implemented and enforced laws that protect people from dangerous pollution in the air they breathe, the water they drink, and the land that forms the foundation of their communities. At the same time, my experience in North Carolina reinforced that EPA cannot solve these challenges alone. We can only make progress if we work in close collaboration with Tribes, states, localities, and stakeholders to enact solutions that follow the science and stand the test of time. To affect meaningful change, engagement, transparency, and accountability will be critical as we move forward.

This roadmap will not solve our PFAS challenges overnight. But it will turn the tide by harnessing the collective resources and authority across federal, Tribal, state, and local governments to empower meaningful action now.

I want to thank the co-chairs of the EPA Council on PFAS—Radhika Fox, Assistant Administrator for Water, and Deb Szaro, Acting Regional Administrator in Region 1—for their leadership in guiding the development of this strategy.

Let's get to work.

Administrator Michael S. Regan

PFAS Council Members

The following policy and technical leaders serve as members of the EPA Council on PFAS. They have been instrumental in working with their respective offices to develop the Agency's strategy. The Council will continue to coordinate across all EPA offices and Regions to accelerate progress on PFAS.

Co-Chairs

Radhika Fox, Assistant Administrator for Water

Deb Szaro, Acting Regional Administrator, Region 1

Office of the Administrator

John Lucey, Special Assistant to the Administrator

Andrea Drinkard, Senior Advisor to the Deputy Administrator

Office of Air and Radiation

John Shoaff, Director, Air Policy and Program Support

Office of Chemical Safety and Pollution Prevention

Jeffrey Dawson, Science Advisor

Tala Henry, Deputy Director, Pollution Prevention and Toxics

Office of Enforcement and Compliance Assurance

Cyndy Mackey, Director, Site Remediation Enforcement

Karin Leff, Director, Federal Facilities Enforcement

Office of General Counsel

Dawn Messier, Deputy Associate General Counsel, Water

Jen Lewis, Deputy Associate General Counsel, Solid Waste and Emergency Response

Office of Land and Emergency Management

Dana Stalcup, Deputy Director, Superfund Remediation and Technology Innovation

Dawn Banks, Director, Policy Analysis and Regulatory Management

Office of Research and Development

Tim Watkins, Acting Director, Center for Public Health and Environmental Assessment

Susan Burden, PFAS Executive Lead

Office of Water

Jennifer McLain, Director, Ground Water and Drinking Water

Deborah Nagle, Director, Science and Technology

Zachary Schafer, Senior Advisor to the Assistant Administrator

EPA Regions

John Blevins, Acting Regional Administrator, Region 4

Tera Fong, Water Division Director, Region 5

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Introduction

Harmful per- and poly-fluoroalkyl substances (PFAS) are an urgent public health and environmental issue facing communities across the United States. PFAS have been manufactured and used in a variety of industries in the United States and around the globe since the 1940s, and they are still being used today. Because of the duration and breadth of use, PFAS can be found in surface water, groundwater, soil, and air—from remote rural areas to densely-populated urban centers. A growing body of scientific evidence shows that exposure at certain levels to specific PFAS can adversely impact human health and other living things. Despite these concerns, PFAS are still used in a wide range of consumer products and industrial applications.

Every level of government—federal, Tribal, state, and local—needs to exercise increased and sustained leadership to accelerate progress to clean up PFAS contamination, prevent new contamination, and make game-changing breakthroughs in the scientific understanding of PFAS. The EPA Council on PFAS developed this strategic roadmap to lay out EPA's whole-of-agency approach to addressing PFAS. To deliver needed protections for the American people, the roadmap sets timelines by which the Agency plans to take specific actions during the first term of the Biden-Harris Administration. The strategic roadmap builds on and accelerates implementation of policy actions identified in the Agency's 2019 action plan and commits to bolder new policies to safeguard public health, protect the environment, and hold polluters accountable.

The risks posed by PFAS demand that the Agency attack the problem on multiple fronts at the same time. EPA must leverage the full range of statutory authorities to confront the human health and ecological risks of PFAS. The actions described in this document each represent important and meaningful steps to safeguard communities from PFAS contamination. Cumulatively, these actions will build upon one another and lead to more enduring and protective solutions.

EPA's integrated approach to PFAS is focused on three central directives:

- **Research.** Invest in research, development, and innovation to increase understanding of PFAS exposures and toxicities, human health and ecological effects, and effective interventions that incorporate the best available science.

- **Restrict.** Pursue a comprehensive approach to proactively prevent PFAS from entering air, land, and water at levels that can adversely impact human health and the environment.

- **Remediate.** Broaden and accelerate the cleanup of PFAS contamination to protect human health and ecological systems.

The Agency's Approach

EPA's approach is shaped by the unique challenges to addressing PFAS contamination. EPA cannot solve the problem of "forever chemicals" by tackling one route of exposure or one use at a time. Rather, EPA needs to take a lifecycle approach to PFAS in order to make meaningful progress. PFAS pollution is not a legacy issue—these chemicals remain in use in U.S. commerce. As such, EPA cannot focus solely on cleaning up the downstream impacts of PFAS pollution. The Agency needs to also look upstream to prevent new PFAS contamination from entering air, land, and water and exposing communities. As the Agency takes tangible actions both upstream and downstream, EPA will continue to pursue a rigorous scientific agenda to better characterize toxicities, understand exposure pathways, and identify new methods to avert and remediate PFAS pollution. As EPA learns more about the family of PFAS chemicals, the Agency can do more to protect public health and the environment. In all this work, EPA will seek to hold polluters accountable for the contamination they cause and ensure disadvantaged communities equitably benefit from solutions.

Consider the Lifecycle of PFAS

EPA will account for the full lifecycle of PFAS, their unique properties, the ubiquity of their uses, and the multiple pathways for exposure.

PFAS are a group of synthetic chemicals that continue to be released into the environment throughout the lifecycle of manufacturing, processing, distribution in commerce, use, and disposal. Each action in this cycle creates environmental contamination and human and ecological exposure. Exacerbating this challenge is that some PFAS persist in the environment. PFAS are synthesized for many different uses, ranging from firefighting foams, to coatings for clothes and furniture, to food contact substances. Many PFAS are also used in industrial processes and applications, such as in the manufacturing of other chemicals and products. PFAS can be released into the environment during manufacturing and processing as well as during industrial and commercial use. Products known to contain PFAS are regularly disposed of in landfills and by incineration, which can also lead to the release of PFAS. Many PFAS have unique properties that prevent their complete breakdown in the environment, which means that even removing PFAS from contaminated areas can create PFAS-contaminated waste. This is currently unregulated in most cases.

Get Upstream of the Problem

EPA will bring deeper focus to preventing PFAS from entering the environment in the first place—a foundational step to reducing the exposure and potential risks of future PFAS contamination.

Intervening at the beginning of the PFAS lifecycle—before they have entered the environment—is a foundational element of EPA's whole-of-agency approach. While hundreds of individual PFAS compounds are in production and use,[i] a relatively modest number of industrial facilities produce PFAS feedstock,[ii] and a relatively narrow set of industries directly discharge PFAS into water or soil or generate air emissions in large quantities.[iii] This context helps to pinpoint clear opportunities to restrict releases into the environment. EPA will use its authorities to impose appropriate limitations on the introduction of new unsafe PFAS into commerce and will, as appropriate, use all available regulatory and permitting authorities to limit emissions and discharges from industrial facilities. This approach does not eliminate the need for remediation where releases and exposures have already occurred, but it is a critical step to preventing ongoing concentrated contamination of soil and surface and groundwaters.

Hold Polluters Accountable

EPA will seek to hold polluters and other responsible parties accountable for their actions and for PFAS remediation efforts.

Many communities and ecosystems are continuously exposed to PFAS in soil, surface water, groundwater, and air. Areas can be exposed due to their proximity to industrial sites, airports, military bases, land where biosolids containing PFAS have been applied, and other sites where PFAS have been produced or used and disposed of for specific and repeated purposes. When EPA becomes aware of a situation that poses a serious threat to human health or the environment, the Agency will take appropriate action. For other sites where contamination may have occurred, the presence of certain PFAS in these environments necessitates coordinated action to understand what specific PFAS have been released, locations where they are found, where they may be transported through air, soil, and water in the future, and what remediation is necessary. EPA will seek to hold polluters and other responsible parties accountable for their actions, ensuring that they assume responsibility for remediation efforts and prevent any future releases.

Ensure Science-Based Decision-Making

EPA will invest in scientific research to fill gaps in understanding of PFAS, to identify which additional PFAS may pose human health and ecological risks at which exposure levels, and to develop methods to test, measure, remove, and destroy them.

EPA's decisions regarding PFAS will be grounded in scientific evidence and analysis. The current body of scientific evidence clearly indicates that there are real, present, and significant hazards associated with specific PFAS, but significant gaps remain related to the impacts of other PFAS on human health and in the environment. Regulatory development, either at the state or federal level, would greatly benefit from a deeper scientific understanding of the exposure pathways, toxicities, and potential health impacts of less-studied PFAS. The federal government, states, industry, academia, and nonprofit organizations—with appropriate coordination and resources—have the capability to conduct this necessary research.

EPA is conducting new research to better understand the similar and different characteristics of specific PFAS and whether and how to address groups and categories of PFAS. The Agency is focused on improving its ability to address multiple chemicals at once, thereby accelerating the effectiveness of regulations, enforcement actions, and the tools and technologies needed to remove PFAS from air, land, and water.

To break the cycle of contamination and exposure from PFAS, additional research is needed to identify and/or develop techniques to permanently dispose of or destroy these durable compounds. Government agencies, industry, and private laboratories need tools and validated methods to measure PFAS in air, land, and water to identify pollution sources, demonstrate facility compliance, hold polluters accountable, and support communities during and after cleanups.

Prioritize Protection of Disadvantaged Communities

When taking action on PFAS, EPA will ensure that disadvantaged communities have equitable access to solutions.

Many known and potential sources of PFAS contamination (including military bases, airports, industrial facilities, and waste management and disposal sites) are near low-income communities and communities of color. EPA needs to ensure these affected populations have an opportunity to participate in and influence the Agency's decision-making. This may call for the Agency to seek out and facilitate the communities' engagement by providing culturally appropriate information and accommodations for people with Limited English Proficiency, facilitating community access to public meetings and comment periods, and offering technical assistance to build community-based capacity for participation. EPA's actions need to consider the unique on-the-ground conditions in these communities, such as outdated infrastructure, to help ensure they benefit equitably from policy solutions.

EPA will also collect more data and develop new methodologies to understand PFAS exposure pathways in disadvantaged communities; to what extent PFAS pollution contributes to the cumulative burden of exposures from multiple sources in these communities; and how non-environmental stressors, such as systemic socioeconomic disparities, can exacerbate the impacts of pollution exposure and vice versa.

Goals and Objectives

EPA's comprehensive approach to addressing PFAS is guided by the following goals and objectives.

RESEARCH

Invest in research, development, and innovation to increase understanding of PFAS exposures and toxicities, human health and ecological effects, and effective interventions that incorporate the best available science.

Objectives

- Build the evidence base on individual PFAS and define categories of PFAS to establish toxicity values and methods.
- Increase scientific understanding on the universe of PFAS, sources of environmental contamination, exposure pathways, and human health and ecological effects.
- Expand research on current and emerging PFAS treatment, remediation, destruction, disposal, and control technologies.
- Conduct research to understand how PFAS contribute to the cumulative burden of pollution in communities with environmental justice concerns.

RESTRICT

Pursue a comprehensive approach to proactively prevent PFAS from entering air, land, and water at levels that can adversely impact human health and the environment.

Objectives

- Use and harmonize actions under all available statutory authorities to control and prevent PFAS contamination and minimize exposure to PFAS during consumer and industrial uses.
- Place responsibility for limiting exposures and addressing hazards of PFAS on manufacturers, processors, distributors, importers, industrial and other significant users, dischargers, and treatment and disposal facilities.
- Establish voluntary programs to reduce PFAS use and release.
- Prevent or minimize PFAS discharges and emissions in all communities, regardless of income, race, or language barriers.

REMEDIATE

Broaden and accelerate the cleanup of PFAS contamination to protect human health and ecological systems.

Objectives

- Harmonize actions under all available statutory authorities to address PFAS contamination to protect people, communities, and the environment.
- Maximize responsible party performance and funding for investigations and cleanup of PFAS contamination.
- Help ensure that communities impacted by PFAS receive resources and assistance to address contamination, regardless of income, race, or language barriers.
- Accelerate the deployment of treatment, remediation, destruction, disposal, and mitigation technologies for PFAS, and ensure that disposal and destruction activities do not create new pollution problems in communities with environmental justice concerns.

Key Actions

This section summarizes the bold actions that EPA plans to take from 2021 through 2024 on PFAS, as well as some ongoing efforts thereafter. The actions described in this roadmap are subject to the availability of appropriations and other resources. Each of these actions—led by EPA's program offices—are significant building blocks in the Agency's comprehensive strategy to protect public health and ecosystems by researching, restricting, and remediating PFAS contamination. As EPA takes each of these actions, it also commits to transparent, equitable, and inclusive engagement with all stakeholders to inform the Agency's work.

These are not the only actions underway at EPA, nor will they be the last. As the Agency does more, it will learn more. And as EPA learns more, it will do more. As EPA continues to build the evidence base, as regulatory work matures, and as EPA learns more from its partnerships across the country, the Agency will deliver additional actions commensurate with the urgency and scale of response that the PFAS problem demands.

Office of Chemical Safety and Pollution Prevention

Publish national PFAS testing strategy

Expected Fall 2021

EPA needs to evaluate a large number of PFAS for potential human health and ecological effects. Most PFAS have limited or no toxicity data. To address this data gap, EPA is developing a national PFAS testing strategy to deepen understanding of the impacts of categories of PFAS, including potential hazards to human health and the environment. This will help EPA identify and select PFAS for which the Agency will require testing using Toxic Substances Control Act (TSCA) authorities. In the 2020 National Defense Authorization Act (NDAA), Congress directed EPA to develop a process for prioritizing which PFAS or classes of PFAS should be subject to additional research efforts based on potential for human exposure to, toxicity of, and other available information. EPA will also identify existing test data for PFAS (both publicly available and submitted to EPA under TSCA) that will be considered prior to requiring further testing to ensure adherence to the TSCA goal of reducing animal testing. EPA will use the testing strategy to identify important gaps in existing data and to select representative chemical(s) within identified categories as priorities for additional studies. EPA expects to exercise its TSCA Section 4 order authority to require PFAS manufacturers to conduct and fund the studies. EPA plans to issue the first round of test orders on the selected PFAS by the end of 2021.

Ensure a robust review process for new PFAS

Efforts Ongoing

EPA's TSCA New Chemicals program plays an important gatekeeper role in ensuring the safety of new chemicals, including new PFAS, prior to their entry in U.S. commerce. Where unreasonable risks are identified as part of the review process, EPA must mitigate those risks before any manufacturing activity can commence. The 2016 TSCA amendments require EPA to review and make a determination regarding the potential risks for each new chemical submission. Since early 2021, EPA has taken steps to ensure that new PFAS are subject to rigorous reviews and appropriate safeguards, including making changes to the policies and processes underpinning reviews and determinations on new chemicals to better align with the 2016 amendments. In addition, EPA has previously allowed some new PFAS to enter the market through low-volume exemptions (LVEs), following an expedited, 30-day review process. In April 2021, the Agency announced that it would generally expect to deny pending and future LVE submissions for PFAS based on the complexity of PFAS chemistry, potential health effects, and their longevity and persistence in the environment. Moving forward, EPA will apply a rigorous premanufacture notice review process for new PFAS to ensure these substances are safe before they enter commerce.

Review previous decisions on PFAS

Efforts Ongoing

EPA is also looking at PFAS that it has previously reviewed through the TSCA New Chemicals program, including those that it reviewed prior to the 2016 TSCA amendments. For example, EPA recently launched a stewardship program to encourage companies to voluntarily withdraw previously granted PFAS LVEs. EPA also plans to revisit past PFAS regulatory decisions and address those that are insufficiently protective. As part of this effort, the Agency could impose additional notice requirements to ensure it can review PFAS before they are used in new ways that might present concerns.

In addition, EPA plans to issue TSCA Section 5(e) orders for existing PFAS for which significant new use notices (e.g., a new manufacturing process for an existing PFAS, or a new use or user) have recently been filed with EPA. The orders would impose rigorous safety requirements as a condition of allowing the significant new use to commence.

More broadly, EPA is planning to improve approaches for overall tracking and enforcement of requirements in new chemical consent orders and significant new use rules (SNURs) to ensure that companies are complying with the terms of those agreements and regulatory notice requirements.

Close the door on abandoned PFAS and uses

Expected Summer 2022

Many existing chemicals (i.e., those that are already in commerce and listed on the TSCA Inventory of chemicals), including PFAS, are currently not subject to any type of restriction under TSCA. In some instances, the chemicals themselves have not been actively manufactured for many years. In others, chemicals may have certain past uses that have been abandoned. Absent restriction, manufacturers are free to begin using those abandoned chemicals or resume those abandoned uses at any time. Under TSCA, by rule, EPA can designate uses of a chemical that are not currently ongoing—and potentially *all* uses associated with an inactive chemical—as “significant new uses.” Doing so ensures that an entity must first submit a notice and certain information to EPA before it can resume use of that chemical or use. TSCA then requires EPA to review and make an affirmative determination on the potential risks to health and the environment and to require safety measures to address unreasonable risks before allowing the PFAS use to resume. EPA is considering how it can apply this authority to help address abandoned uses of PFAS as well as future uses of PFAS on the inactive portion of the TSCA Inventory.

Enhance PFAS reporting under the Toxics Release Inventory

Expected Spring 2022

The Toxics Release Inventory (TRI) helps EPA compile data and information on releases of certain chemicals and supports informed decision-making by companies, government agencies, non-governmental organizations, and the public. Pursuant to the 2020 NDAA, certain industry sectors must report certain PFAS releases to TRI. However, certain exemptions and exclusions remain for those PFAS reporters, which significantly limited the amount of data that EPA received for these chemicals in the first year of reporting.[iv] To enhance the quality and quantity of PFAS information collected through TRI, EPA intends to propose a rulemaking in 2022 to categorize the PFAS on the TRI list as "Chemicals of Special Concern" and to remove the de minimis eligibility from supplier notification requirements for all "Chemicals of Special Concern." EPA will also continue to update the list of PFAS subject to TRI and expects to announce an additional rulemaking to add more PFAS to TRI in 2022, as required by the 2020 NDAA.

Finalize new PFAS reporting under TSCA Section 8

Expected Winter 2022

TSCA Section 8(a)(7) provides authority for EPA to collect existing information on PFAS. In June 2021, EPA published a proposed data-gathering rule that would collect certain information on any PFAS manufactured since 2011, including information on uses, production volumes, disposal, exposures, and hazards. EPA will consider public comments on the proposal and finalize it before January 1, 2023. Ultimately, information received under this rule will enable EPA to better characterize the sources and quantities of manufactured PFAS in the United States and will assist the Agency in its future research, monitoring, and regulatory efforts.

Office of Water

Undertake nationwide monitoring for PFAS in drinking water

Final Rule Expected Fall 2021

The Safe Drinking Water Act (SDWA) establishes a data-driven and risk-based process to assess drinking water contaminants of emerging concern. Under SDWA, EPA requires water systems to conduct sampling for unregulated contaminants every five years. EPA published the proposed Fifth Unregulated Contaminant Monitoring Rule (UCMR 5) in March 2021. As proposed, UCMR 5 would provide new data that is critically needed to improve EPA's understanding of the frequency that 29 PFAS are found in the nation's drinking water systems and at what levels. The proposed UCMR 5 would significantly expand the number of drinking water systems participating in the program, pending sufficient appropriations by Congress. The data gathered from an expanded set of drinking water systems would improve EPA's ability to conduct state and local assessments of contamination, including analyses of potential environmental justice impacts. As proposed, and if funds are appropriated by Congress, all public water systems serving 3,300 or more people and 800 representative public water systems serving fewer than 3,300 would collect samples during a 12-month period from January 2023 through December 2025. EPA is considering comments on the proposed UCMR 5 and preparing a final rule. Going forward, EPA will continue to prioritize additional PFAS for inclusion in UCMR 6 and beyond, as techniques to measure these additional substances in drinking water are developed and validated.

Establish a national primary drinking water regulation for PFOA and PFOS

Proposed Rule Expected Fall 2022,
Final Rule Expected Fall 2023

Under the SDWA, EPA has the authority to set enforceable National Primary Drinking Water Regulations (NPDWRs) for drinking water contaminants and require monitoring of public water supplies. To date, EPA has regulated more than 90 drinking water contaminants but has not established national drinking water regulations for any PFAS. In March 2021, EPA published the Fourth Regulatory Determinations, including a final determination to regulate Perfluorooctanoic acid (PFOA) and Perfluorooctane sulfonic acid (PFOS) in drinking water. The Agency is now developing a proposed NPDWR for these chemicals. As EPA undertakes this action, the Agency is also evaluating additional PFAS and considering regulatory actions to address groups of PFAS. EPA expects to issue a proposed regulation in Fall 2022 (before the Agency's statutory deadline of March 2023). The Agency anticipates issuing a final regulation in Fall 2023 after considering public comments on the proposal. Going forward, EPA will continue to analyze whether NPDWR revisions can improve public health protection as additional PFAS are found in drinking water.

Publish the final toxicity assessment for GenX and five additional PFAS

Expected Fall 2021 and Ongoing

EPA plans to publish the toxicity assessments for two PFAS, hexafluoropropylene oxide dimer acid and its ammonium salt. These two chemicals are known as "GenX chemicals." GenX chemicals have been found in surface water, groundwater, drinking water, rainwater, and air emissions. GenX chemicals are known to impact human health and ecosystems. Scientists have observed liver and kidney toxicity, immune effects, hematological effects, reproductive and developmental effects, and cancer in animals exposed to GenX chemicals. Completing a toxicity assessment for GenX is essential to better understanding its effects on people and the environment. EPA can use this information to develop health advisories that will help communities make informed decisions to better protect human health and ecological wellness. The Office of Research and Development is also currently developing toxicity assessments for five other PFAS—PFBA, PFHxA, PFHxS, PFNA, and PFDA.

Publish health advisories for GenX and PFBS

Expected Spring 2022

PFAS contamination has impacted drinking water quality across the country, including in underserved rural areas and communities of color. SDWA authorizes EPA to develop non-enforceable and non-regulatory drinking water health advisories to help Tribes, states, and local governments inform the public and determine whether local actions are needed to address public health impacts in these communities. Health advisories offer a margin of protection by defining a level of drinking water concentration at or below which lifetime exposure is not anticipated to lead to adverse health effects. They include information on health effects, analytical methodologies, and treatment technologies and are designed to protect all lifestages. EPA will publish health advisories for Perfluorobutane sulfonic acid (PFBS) and GenX chemicals based on final toxicity assessments. The Agency will develop accompanying fact sheets in different languages to facilitate access to information on GenX and other PFAS. Going forward, EPA will develop health advisories as the Agency completes toxicity assessments for additional PFAS.

Restrict PFAS discharges from industrial sources through a multi-faceted Effluent Limitations Guidelines program

Expected 2022 and Ongoing

Effluent Limitations Guidelines (ELGs) are a powerful tool to limit pollutants from entering the nation's waters. ELGs establish national technology-based regulatory limits on the level of specified pollutants in wastewater discharged into surface waters and into municipal sewage treatment facilities. EPA has been conducting a PFAS multi-industry study to inform the extent and nature of PFAS discharges. Based on this study, EPA is taking a proactive approach to restrict PFAS discharges from multiple industrial categories. EPA plans to make significant progress in its ELG regulatory work by the end of 2024. EPA has established timelines for action—whether it is data collection or rulemaking—on the nine industrial categories in the proposed PFAS Action Act of 2021, as well as other industrial categories such as landfills. EPA's multi-faceted approach entails:

- Undertake rulemaking to restrict PFAS discharges from industrial categories where EPA has the data to do so—including the guidelines for organic chemicals, plastics and synthetic fibers (OCPSF), metal finishing, and electroplating. Proposed rule is expected in Summer 2023 for OCPSF and Summer 2024 for metal finishing and electroplating.
- Launch detailed studies on facilities where EPA has preliminary data on PFAS discharges, but the data are currently insufficient to support a potential rulemaking. These include electrical and electronic components, textile mills, and landfills. EPA expects these studies to be complete by Fall 2022 to inform decision making about a future rulemaking by the end of 2022.
- Initiate data reviews for industrial categories for which there is little known information on PFAS discharges, including leather tanning and finishing, plastics molding and forming, and paint formulating. EPA expects to complete these data reviews by Winter 2023 to inform whether there are sufficient data to initiate a potential rulemaking.
- Monitor industrial categories where the phaseout of PFAS is projected by 2024, including pulp, paper, paperboard, and airports. The results of this monitoring, and whether future regulatory action is needed, will be addressed in the Final ELG Plan 15 in Fall 2022.

Leverage NPDES permitting to reduce PFAS discharges to waterways

Expected Winter 2022

The National Pollutant Discharge Elimination System (NPDES) program interfaces with many pathways by which PFAS travel and are released into the environment and ultimately impact people and water quality. EPA will seek to proactively use existing NPDES authorities to reduce discharges of PFAS at the source and obtain more comprehensive information through monitoring on the sources of PFAS and quantity of PFAS discharged by these sources. EPA will use the effluent monitoring data to inform which industrial categories the Agency should study for future ELGs actions to restrict PFAS in wastewater discharges.

- **Leverage federally-issued NPDES permits to reduce PFAS discharges.**[v] EPA will propose monitoring requirements at facilities where PFAS are expected or suspected to be present in wastewater and stormwater discharges, using EPA's recently published analytical method 1633, which covers 40 unique PFAS. In addition, EPA will propose, as appropriate, that NPDES permits: 1) contain conditions based on product elimination and substitution when a reasonable alternative to using PFAS is available in the industrial process; 2) require best management practices to address PFAS-containing firefighting foams for stormwater permits; 3) require enhanced public notification and engagement with downstream communities and public water systems; and 4) require pretreatment programs to include source control and best management practices to protect wastewater treatment plant discharges and biosolid applications.
- **Issue new guidance to state permitting authorities to address PFAS in NPDES permits.** EPA will issue new guidance recommending that state-issued permits that do not already include monitoring requirements for PFAS use EPA's recently published analytical method 1633, which covers 40 unique PFAS, at facilities where PFAS is expected or suspected to be present in wastewater and stormwater discharges. In addition, the new guidance will recommend the full suite of permitting approaches that EPA will use in federally-issued permits. The guidance will enable communities to work closely with their state permitting authorities to suggest monitoring at facilities suspected of containing PFAS.

Publish multi-laboratory validated analytical method for 40 PFAS

Expected Fall 2022

In September 2021, EPA (in collaboration with the Department of Defense) published a single-laboratory validated method to detect PFAS. The method can measure up to 40 specific PFAS compounds in eight environmental matrices (including wastewater, surface water and biosolids) and has numerous applications, including NPDES compliance monitoring. EPA and DOD are continuing this collaboration to complete a multi-laboratory validation of the method. EPA expects to publish the multi-lab validated method online by Fall 2022. Following the publication of the method, EPA will initiate a rulemaking to propose the promulgation of this method under the Clean Water Act (CWA).

Publish updates to PFAS analytical methods to monitor drinking water

Expected Fall 2024

SDWA requires EPA to use scientifically robust and validated analytical methods to assess the occurrence of contaminants of emerging concern, such as an unidentified or newly detected PFAS chemical. EPA will update and validate analytical methods to monitor additional PFAS. First, EPA will review reports of PFAS of concern and seek to procure certified reference standards that are essential for accurate and selective quantitation of emerging PFAS of concern in drinking water samples. EPA will evaluate analytical methods previously published for monitoring PFAS in drinking water (EPA Methods 533 and 537.1) to determine the efficacy of expanding the established target PFAS analyte list to include any emerging PFAS. Upon conclusion of this evaluation, EPA will complete multi-laboratory validation studies and peer review and publish updated EPA PFAS analytical methods for drinking water, making them available to support future drinking water monitoring programs.

Publish final recommended ambient water quality criteria for PFAS

Expected Winter 2022 and Fall 2024

EPA will develop national recommended ambient water quality criteria for PFAS to protect aquatic life and human health. Tribes and states use EPA-recommended water quality criteria to develop water quality standards to protect and restore waters, issue permits to control PFAS discharges, and assess the cumulative impact of PFAS pollution on local communities. EPA will publish recommended aquatic life criteria for PFOA and PFOS and benchmarks for other PFAS that do not have sufficient data to define a recommended aquatic life criteria value. EPA will first develop human health criteria for PFOA and PFOS, taking into account drinking water and fish consumption. This initiative will consider the latest scientific information and will develop human health criteria for additional PFAS when final toxicity assessments are available. Additionally, EPA will support Tribes in developing water quality standards that will protect waters under Tribal jurisdiction under the same framework as waters in adjacent states. Aquatic life criteria are expected in Winter 2022, and human health criteria are expected Fall 2024.

Monitor fish tissue for PFAS from the nation’s lakes and evaluate human biomarkers for PFAS

Expected Summer 2022

States and Tribes have highlighted fish tissue data in lakes as a critical information need. Food and water consumption are important pathways of PFAS exposure, and PFAS can accumulate in fish tissue. In fact, EPA monitoring to date shows the presence of PFAS, at varying levels, in approximately 100 percent of fish tested in the Great Lakes and large rivers. In Summer 2022, EPA will collect fish tissue in the National Lakes Assessment for the first national study of PFAS in fish tissue in U.S. lakes. This will provide a better understanding of where PFAS fish tissue contamination is occurring, which

PFAS are involved, and the severity of the problem. The new data will complement EPA's analyses of PFAS in fish tissue and allow EPA to better understand unique impacts on subsistence fishers, who may eat fish from contaminated waterbodies in higher quantities. EPA's preliminary analysis on whether concentrations of certain PFAS compounds in human blood could be associated with eating fish using the Centers for Disease Control and Prevention's National Health and Nutrition Examination Survey (NHANES) data found a positive correlation. Completing this analysis will help make clear the importance of the fish consumption pathway for protecting communities. EPA will continue to pursue collaboration with Tribal and federal partners to investigate this issue of mutual interest.

Finalize list of PFAS for use in fish advisory programs

Expected Spring 2023

EPA will publish a list of PFAS for state and Tribal fish advisory programs that are either known or thought to be in samples of edible freshwater fish in high occurrence nationwide. This list will serve as guidance to state and Tribal fish tissue monitoring and advisory programs so that they know which PFAS to monitor and how to set fish advisories for PFAS that have human health impacts via fish consumption. This information will encourage more robust data collection from fish advisory programs and promote consistency of fish tissue PFAS monitoring results in EPA's publicly accessible Water Quality Portal. By issuing advisories for PFAS, state and Tribal programs can provide high-risk populations, including communities and individuals who depend on subsistence fishing, with more information about how to protect their health.

Finalize risk assessment for PFOA and PFOS in biosolids

Expected Winter 2024

Biosolids, or sewage sludge, from wastewater treatment facilities can sometimes contain PFAS. When spread on agricultural fields, the PFAS can contaminate crops and livestock. The CWA authorizes EPA to set pollutant limits and monitoring and reporting requirements for contaminants in biosolids if sufficient scientific evidence shows that there is potential harm to human health or the environment. A risk assessment is key to determining the potential harm associated with human exposure to chemicals. EPA will complete the risk assessment for PFOA and PFOS in biosolids by Winter 2024. The risk assessment will serve as the basis for determining whether regulation of PFOA and PFOS in biosolids is appropriate. If EPA determines that a regulation is appropriate, biosolids standards would improve the protection of public health and wildlife health from health effects resulting from exposure to biosolids containing PFOA and PFOS.

Office of Land and Emergency Management

Propose to designate certain PFAS as CERCLA hazardous substances

Proposed rule expected Spring 2022; Final rule expected Summer 2023

EPA is developing a Notice of Proposed Rulemaking to designate PFOA and PFOS as Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) hazardous substances. Such designations would require facilities across the country to report on PFOA and PFOS releases that meet or exceed the reportable quantity assigned to these substances. The hazardous substance designations would also enhance the ability of federal, Tribal, state, and local authorities to obtain information regarding the location and extent of releases. EPA or other agencies could also seek cost recovery or contributions for costs incurred for the cleanup. The proposed rulemaking will be available for public comment in Spring 2022. The Agency commits to conducting robust stakeholder engagement with communities near PFAS-contaminated sites.

Issue advance notice of proposed rulemaking on various PFAS under CERCLA

Expected Spring 2022

In addition to developing a Notice of Proposed Rulemaking designating PFOA and PFOS as hazardous substances under CERCLA, EPA is developing an Advance Notice of Proposed Rulemaking to seek public input on whether to similarly designate other PFAS. The Agency may request input regarding the potential hazardous substance designation for precursors to PFAS, additional PFAS, and groups or subgroups of PFAS. The Agency will engage robustly with communities near PFAS-contaminated sites to seek their input and learn about their lived experiences. Going forward, EPA will consider designating additional PFAS as hazardous substances under CERCLA as more specific information related to the health effects of those PFAS and methods to measure them in groundwater are developed.

Issue updated guidance on destroying and disposing of certain PFAS and PFAS-containing materials

Expected by Fall 2023

The 2020 NDAA requires that EPA publish interim guidance on destroying and disposing of PFAS and certain identified non-consumer PFAS-containing materials. It also requires that EPA revise that guidance at least every three years, as appropriate. EPA published the first interim guidance in December 2020 for public comment. It identifies three technologies that are commercially available to either destroy or dispose of PFAS and PFAS-containing materials and outlines the significant uncertainties and information gaps that exist concerning the technologies' ability to destroy or dispose of PFAS while minimizing the migration of PFAS to the environment. The guidance also highlights research that is underway and planned to address some of these information gaps. Furthermore, the interim guidance identifies existing EPA tools, methods, and approaches to characterize and assess the risks to disproportionately impacted people of color and low-income communities living near likely PFAS destruction or disposal sites. EPA's updated guidance will address the public comments and reflect newly published research results. Since the publication of the interim guidance, EPA and other agencies have been conducting relevant research on destruction and disposal technologies. EPA anticipates that additional research data will become available starting in 2022. EPA will update the guidance when sufficient useful information is available and no later than the statutory deadline of December 2023.

Office of Air and Radiation

Build the technical foundation to address PFAS air emissions

Expected Fall 2022 and Ongoing

The Clean Air Act requires EPA to regulate emissions of hazardous air pollutants (HAPs), which are pollutants that are known or suspected to cause cancer or other serious health effects. At present, EPA actively works with Tribal, state, and local governments to reduce air emissions of 187 HAPs to the environment. While PFAS are not currently listed as HAPs under the Clean Air Act, EPA is building the technical foundation on PFAS air emissions to inform future decisions. EPA is conducting ongoing work to:

- Identify sources of PFAS air emissions;
- Develop and finalize monitoring approaches for measuring stack emissions and ambient concentrations of PFAS;
- Develop information on cost-effective mitigation technologies; and
- Increase understanding of the fate and transport of PFAS air emissions to assess their potential for impacting human health via contaminated groundwater and other media pathways.

EPA will use a range of tools, such as EJSCREEN, to determine if PFAS air pollution disproportionately affects communities with environmental justice concerns. Data from other ongoing EPA activities, such as field tests, TRI submissions, and new TSCA reporting and recordkeeping requirements, will help EPA collect additional information on sources and releases. By Fall 2022, EPA will evaluate mitigation options, including listing certain PFAS as hazardous air pollutants and/or pursuing other regulatory and non-regulatory approaches. The Agency will continue to collect necessary supporting technical information on an ongoing basis.

Office of Research and Development

Develop and validate methods to detect and measure PFAS in the environment

Ongoing Actions

Robust, accurate methods for detecting and measuring PFAS in air, land, and water are essential for understanding which PFAS are in the environment and how much are present. These methods are also essential for evaluating the effectiveness of different technologies for removing PFAS from air, land, and water and for implementing future regulations. To date, EPA has developed validated methods to measure 29 PFAS in drinking water and 24 PFAS in groundwater, surface water, and wastewater. EPA has also developed a method for measuring selected PFAS in air emissions. EPA will build on this work by developing additional targeted methods for detecting and measuring specific PFAS and non-targeted methods for identifying unknown PFAS in the environment. EPA also recognizes the need for “total PFAS” methods that can measure the amount of PFAS in environmental samples without identifying specific PFAS. EPA will increase its efforts to develop and, if appropriate, validate “total PFAS” methods, focusing on air emissions, wastewater, and drinking water. Near-term deliverables include:

- Draft total adsorbable fluorine method for wastewater for potential laboratory validation (Fall 2021);
- Draft method for measuring additional PFAS in air emissions (Fall 2022); and
- Draft methods and approaches for evaluating PFAS leaching from solid materials (Fall 2022).

Advance the science to assess human health and environmental risks from PFAS

Ongoing Actions

EPA will expand understanding of the toxicity of PFAS through several ongoing research activities. First, EPA will continue to develop human health toxicity assessments for individual PFAS under EPA’s Integrated Risk Information System (IRIS) Program, and if needed, other fit-for-purpose toxicity values. When combined with exposure information and other important considerations, EPA can use these toxicity assessments to assess potential human health risks to determine if, and when, it is appropriate to address these chemicals. Most PFAS, however, have limited or no toxicity data to inform human health or ecological toxicity assessments. To better understand human health and ecological toxicity across a wider variety of PFAS, EPA will continue to compile and summarize available and relevant scientific information on PFAS and conduct toxicity testing on individual PFAS and PFAS mixtures. This will inform the development and refinement of PFAS categories for hazard assessment. EPA will also conduct research to identify PFAS sources in the outdoor and indoor environment, to characterize PFAS movement through the environment, and to identify the relative importance of different human exposure pathways to PFAS (e.g., ingestion of contaminated food or water, interaction with household articles or consumer products, and inhalation of indoor or outdoor air containing PFAS). EPA also will work to characterize how exposure to PFAS may contribute to cumulative impacts on communities, particularly communities with environmental justice concerns. Near-term deliverables include:

- Identify initial PFAS categories to inform TSCA test orders as part of the PFAS National Testing Strategy (Fall 2021)
- Consolidate and update data on chemical/physical properties, human health toxicity and toxicokinetics, and ecotoxicity (Spring 2022 – Fall 2024)
- Complete draft PFHxS, PFHxA, PFNA, and PFDA IRIS assessments for public comment and peer review (Spring – Fall 2022)
- Complete and publish the final PFBA IRIS assessment (Fall 2022)

Evaluate and develop technologies for reducing PFAS in the environment

Ongoing Actions

EPA needs new data and information on the effectiveness of different technologies and approaches for removing PFAS from the environment and managing PFAS and PFAS-containing materials to inform decisions on drinking water and wastewater treatment, contaminated site cleanup and remediation, air emission controls, and end-of-life materials management. This information is also needed to better ensure that particular treatment and waste management technologies and approaches do not themselves lead to additional PFAS exposures, particularly in overburdened communities where treatment and waste management facilities are often located. Toward that end, EPA will continue efforts to develop approaches for characterizing PFAS in source waters, at contaminated sites, and near PFAS production and treatment/disposal facilities. EPA will also continue to evaluate and develop technologies for drinking water and wastewater treatment, contaminated site remediation, air emission controls, and destruction and disposal of PFAS-containing materials and waste streams. These efforts include conducting laboratory- and pilot-scale studies, which will inform the design of full-scale field studies done in partnership with facilities and states to evaluate real-world applications of different PFAS removal technologies and management approaches.

EPA will prioritize efforts to evaluate conventional thermal treatment of PFAS-containing wastes and air emissions and assess the effectiveness of conventional drinking water and wastewater treatment processes. EPA will also continue to evaluate and advance the application of innovative, non-thermal technologies to treat PFAS waste and PFAS-contaminated materials. Building upon these evaluations, EPA will document the performance of PFAS removal technologies and establish technology-based PFAS categories that identify the list of PFAS that are effectively removed through the application of the associated technology. Near-term deliverables include:

- Collect data to inform the 2023 guidance on destroying and disposing of certain PFAS and PFAS-containing materials (Spring 2022 – Fall 2023);
- Identify initial PFAS categories for removal technologies (Summer 2022); and
- Develop effective PFAS treatment technologies for drinking water systems (Fall 2022).

Cross-Program

Engage directly with affected communities in every EPA Region

Expected Fall 2021 and Ongoing

EPA must fully understand the challenges facing individuals and communities grappling with PFAS contamination to understand their lived experiences and determine the most effective interventions. As recommended by the National Environmental Justice Advisory Council (NEJAC), EPA will meet with affected communities in each EPA Region to hear how PFAS contamination impacts their lives and livelihoods. EPA will use the knowledge from these engagements to inform the implementation of the actions described in this roadmap. EPA will also use the input to develop and share information to reduce potential health risks in the near term and help communities on the path to remediation and recovery from PFAS contamination.

Use enforcement tools to better identify and address PFAS releases at facilities

Ongoing Actions

EPA is initiating actions under multiple environmental authorities—RCRA, TSCA, CWA, SDWA and CERCLA—to identify past and ongoing releases of PFAS into the environment at facilities where PFAS has been used, manufactured, discharged, disposed of, released, and/or spilled. EPA is conducting inspections, issuing information requests, and collecting data to understand the level of contamination and current risks posed by PFAS to surrounding communities and will seek to address threats to human health with all its available tools. For example, EPA's enforcement authorities allow the Agency, under certain circumstances, to require parties responsible for PFAS contamination to characterize the nature and extent of PFAS contamination, to put controls in place to expeditiously limit future releases, and to address contaminated drinking water, soils, and other contaminated media. When EPA becomes aware of a potential imminent and substantial endangerment situation where PFAS poses a threat to human health, the Agency will swiftly employ its expertise to assess the situation and take appropriate action, including using statutorily authorized powers.

Accelerate public health protections by identifying PFAS categories

Expected Winter 2021 and Ongoing

To accelerate EPA's ability to address PFAS and deliver public health protections sooner, EPA is working to break the large, diverse class of PFAS into smaller categories based on similarities across defined parameters (such as chemical structure, physical and chemical properties, and toxicological properties). EPA plans to initially categorize PFAS using two approaches. In the first approach, EPA plans to use toxicity and toxicokinetic data to develop PFAS categories for further hazard assessment and to inform hazard- or risk-based decisions. In the second approach, EPA plans to develop PFAS categories based on removal technologies using existing understanding of treatment, remediation, destruction, disposal, control, and mitigation principles.

EPA plans to use the PFAS categories developed from these two approaches to identify gaps in coverage from either a hazard assessment or removal technology perspective, which will help EPA prioritize future actions to research, restrict, and remediate PFAS. For example, EPA may choose to prioritize research to characterize the toxicity of PFAS that are not being addressed by regulations that require the implementation of removal technologies. Conversely, EPA may prioritize research to evaluate the efficacy of technologies designed to remove PFAS that are included in a hazard-based category with relatively higher toxicities. To support coordination and integration of information across PFAS categories, EPA plans to develop a PFAS categorization database that will capture key characteristics of individual PFAS, including category assignments.

Establish a PFAS Voluntary Stewardship Program

Expected Spring 2022

Reduction of PFAS exposure through regulatory means can take time to develop, finalize, and implement. Moreover, current PFAS regulatory efforts do not extend to all of the approximately 600 PFAS currently in commerce. As a companion to other efforts described in this roadmap, EPA will establish a voluntary stewardship program challenging industry to reduce overall releases of PFAS into the environment. The program, which will not supplant industry's regulatory or compliance requirements, will call on industry to go beyond those requirements by reporting all PFAS releases in order to establish a baseline and then continuing to report to measure progress in reducing releases over time. EPA will validate industry efforts to meet reduction targets and timelines.

Educate the public about the risks of PFAS

Expected Fall 2021 and Ongoing

Addressing PFAS contamination is a critical part of EPA's mission to protect human health and the environment. This important mission cannot be achieved without effectively communicating with communities, individuals, businesses, the media, and Tribal, state, and local partners about the known and potential health risks associated with these chemicals. When EPA communicates risk, it is the Agency's goal to provide meaningful, understandable, and actionable information to many audiences. To accomplish this goal, EPA will make available key explainers that help the public understand what PFAS are, how they are used, and how PFAS can impact their health and their lives. These explainers and other educational materials will be published in multiple languages, and the Agency will work to ensure information reaches targeted communities (including those with limited access to technology and resources).

Issue an annual public report on progress towards PFAS commitments

Winter 2022 and Ongoing

EPA is committed to acting on PFAS with transparency and accountability. On an annual basis, EPA will report to the public on the status of the actions outlined in this roadmap, as well as future actions the Agency may take. EPA will also engage regularly with communities experiencing PFAS contamination, co-regulators, industry, environmental groups, community leaders, and other stakeholders to clearly communicate its actions and to stay abreast of evolving needs.

Conclusion

Every level of government—federal, Tribal, state, and local—needs to exercise increased and sustained leadership to accelerate progress to clean up PFAS contamination, prevent new contamination, and make game-changing breakthroughs in the scientific understanding of PFAS. This strategic roadmap represents the Agency's commitment to the American people on what EPA seeks to deliver from 2021 to 2024.

The risks posed by PFAS demand that the Agency take a whole-of-agency approach to attack the problem from multiple directions. Focusing only on remediating legacy contamination, for example, does nothing to prevent new contamination from occurring. Focusing only on preventing future contamination fails to minimize risks to human health that exist today. To build more enduring, comprehensive, and protective solutions, EPA seeks to leverage its full range of statutory authorities and work with its partners—including other federal agencies, state and Tribal regulators, scientists, industry, public health officials, and communities living with PFAS contamination—to implement this multi-media approach and achieve tangible benefits for human health and the environment.[vi]

Endnotes

[i] Approximately 650 PFAS are currently in commerce under TSCA, roughly half of which were grandfathered into the TSCA inventory.

[ii] EPA has identified 6-8 facilities that produce PFAS feedstock.

[iii] Key industries with significant documented discharges include PFAS production and processing, metal finishing, airports, pulp and paper, landfills, and textile and carpet manufacturing.

[iv] Examples include de minimis exemption, supplier notification requirements, and applicability of those requirements to wastes.

[v] Federally-issued permits are those that EPA issues in MA, NH, NM, DC, territories, federal waters, and Indian Country (and federal facilities in DE, CO, VT, WA).

[vi] This document provides information to the public on how EPA intends to exercise its discretion in implementing statutory and regulatory provisions that apply to PFAS. Those provisions contain legally binding requirements, and this document does not substitute for those statutory and regulatory provisions or regulations, nor is it a regulation itself.

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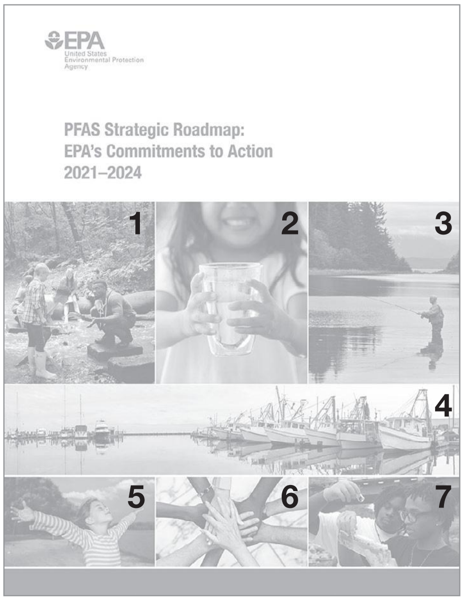

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United States
Environmental Protection
Agency

EPA-100-K-21-002
October 2021

Proposed PFAS National Primary Drinking Water Regulation
FAQs for Drinking Water Primacy Agencies

Overview: What action is EPA taking to address PFAS in drinking water?

The U.S. Environmental Protection Agency (EPA) is taking a key step to protect public health by proposing to establish legally enforceable levels for six per- and polyfluoroalkyl substances (PFAS) known to occur in drinking water, fulfilling a foundational commitment in the Agency's PFAS Strategic Roadmap. Through this proposed rule, EPA is leveraging the most recent science and building on existing state efforts to limit PFAS and provide a nationwide, health-protective standard for these specific PFAS in drinking water.

Some states have established drinking water regulations or guidance values for some PFAS, leading the way in monitoring for and limiting PFAS. The National Primary Drinking Water Regulation (NPDWR) proposed by EPA, if finalized, will provide a nationwide, health-protective level for six PFAS in drinking water: PFOA, PFOS, PFHxS, GenX Chemicals, PFNA, and PFBS. EPA's proposed rule is informed by regulatory development requirements under the Safe Drinking Water Act (SDWA), including EPA's analysis of the best available and most recent peer-reviewed science. The proposal also takes into account the feasibility of analysis and treatment, as well as consideration of costs and benefits.

At this time, communities and water systems should follow applicable state requirements, recognizing that EPA's proposed rule does not currently require water systems to take any action. When the final NPDWR goes into effect, states will be required to have a standard that is no less strict than the NPDWR – as SDWA requires.

Question 1: What is the difference between this proposed drinking water regulation for PFOA, PFOS, PFHxS, GenX Chemicals, PFNA, and PFBS and the 2022 EPA Health Advisories for PFOA, PFOS, PFBS, and GenX Chemicals?

This is a proposed rule for public comment. It does not require any actions for drinking water systems until the rule is finalized. Once the rule is finalized, water systems would have three years to be in compliance with the MCLs.

The proposed regulation includes Maximum Contaminant Levels (MCLs) which, if finalized, are legally enforceable regulatory drinking water standards. EPA establishes MCLs as close as feasible to the health based, non-enforceable, Maximum Contaminant Level Goal (MCLG), taking into consideration the ability to measure and treat to remove a contaminant, as well as the costs and benefits.

Drinking water health advisories are different from MCLs and MCLGs. Each serves a different purpose. Health advisories are not regulatory and are not legally enforceable. Health advisories reflect EPA's assessment of health risks of a contaminant based on the best available science and provide advice and information on actions that water systems may take to address contamination for these and other PFAS. After EPA has considered public comments and issues a final NPDWR, EPA will decide whether to update or remove the interim health advisories for PFOA and PFOS and the final health advisories for PFBS and GenX Chemicals. For more information on the health advisories, please visit https://www.epa.gov/sdwa/drinking-water-health-advisories-pfoa-and-pfos.

Question 2: Why did EPA propose a Hazard Index for PFHxS, GenX Chemicals, PFNA, and PFBS?

EPA is following recent peer-reviewed science that indicates that mixtures of PFAS can pose a health risk greater than each chemical on its own. A Hazard Index helps to account for the increased risk from mixtures of PFAS that may be found in contaminated drinking water. The Hazard Index is a long-established tool that EPA regularly uses, for example, to inform risks of chemical mixtures. It is, for example used at contaminated Superfund sites (under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and Superfund Amendments and Reauthorization Act (SARA)). A Hazard Index considers how toxic each of the four PFAS are and allows a site-specific determination based on the specific drinking water concentrations.

Question 3: How is the Hazard Index for PFHxS, GenX Chemicals, PFNA, and PFBS calculated?

To determine the Hazard Index for these four PFAS, water systems would monitor and use those sampling results as inputs into a formula with their Health-Based Water Concentration (HBWC) (i.e., the level at which no health effects are expected for that PFAS). The proposed HBWCs for each of the four PFAS are below.

Compound	Health-Based Water Concentration (ppt)
PFHxS	**9.0**
GenX Chemicals	**10**
PFNA	**10**
PFBS	**2000**

Water systems would use a calculator tool provided by EPA to easily determine their Hazard Index result. The tool performs the calculation explained below.

For each of the four PFAS, the calculation first divides the results of the drinking water sample by the HBWC and then adds all the values for each PFAS. If the total value is greater than 1.0, it would be an exceedance of the proposed Hazard Index MCL as follows:

$$\text{Hazard Index} = \left(\frac{[\text{GenX}_{\text{water}}]}{[10\text{ ppt}]}\right) + \left(\frac{[\text{PFBS}_{\text{water}}]}{[2000\text{ ppt}]}\right) + \left(\frac{[\text{PFNA}_{\text{water}}]}{[10\text{ ppt}]}\right) + \left(\frac{[\text{PFHxS}_{\text{water}}]}{[9.0\text{ ppt}]}\right)$$

Where $\text{GenX}_{\text{water}}$ = monitored concentration of GenX
$\text{PFBS}_{\text{water}}$ = monitored concentration of PFBS
$\text{PFNA}_{\text{water}}$= monitored concentration of PFNA
$\text{PFHxS}_{\text{water}}$ = monitored concentration of PFHxS

For example, if the mixture contains the following levels of these four PFAS, the Hazard Index for that mixture would exceed the proposed MCL.

$$2.1 = \left(\frac{[5\text{ ppt}]}{[10\text{ ppt}]}\right) + \left(\frac{[200\text{ ppt}]}{[2000\text{ ppt}]}\right) + \left(\frac{[5\text{ ppt}]}{[10\text{ ppt}]}\right) + \left(\frac{[9\text{ ppt}]}{[9.0\text{ ppt}]}\right)$$

Question 4: Under the proposed rule, do all four PFAS under the Hazard Index need to be present for a water system to exceed the proposed PFAS NPDWR?

No. The Hazard Index works at the local level and applies to any combination of the four PFAS. In some cases, a water system could exceed the proposed Hazard Index MCL when only one, two, or three PFAS are present.

Moreover, a high concentration of one Hazard Index PFAS could drive an MCL exceedance.

Question 5: Why didn't EPA include PFOA and PFOS in the proposed Hazard Index MCL?

EPA determined that PFOA and PFOS are likely carcinogens (i.e., cancer causing) and that there is no level of these contaminants that is without a risk of adverse health effects. Therefore, EPA is proposing to set the MCL for these two contaminants at 4 parts per trillion, the lowest feasible level based on the ability to reliably measure and remove these contaminants from drinking water.

Question 6: What is the Practical Quantitation Level (PQL)?

The PQL is defined as the lowest concentration of a contaminant that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. This level provides the precision and accuracy that EPA estimates can be achieved across laboratories nationwide. EPA has used the PQLs for the six PFAS proposed for regulation in determining the proposed MCLs. EPA has identified the following PQLs for the six PFAS proposed for regulation.

Compound	Practical Quantitation Level (ppt)
PFOS	**4.0**
PFOA	**4.0**
PFHxS	**3.0**
GenX Chemicals	**5.0**
PFNA	**4.0**
PFBS	**3.0**

Question 7: What are the proposed rule's monitoring requirements?

The proposed rule would require that all community water systems and non-transient, non-community water systems conduct initial monitoring within three years after the rule's promulgation. The monitoring must be conducted at the entry point to the distribution system. Based on their size and source water, systems must conduct initial monitoring either twice or quarterly during a 12-month period as follows:

- **Groundwater systems serving greater than 10,000 customers.** Initially, these systems would be required to monitor quarterly within a 12-month period.
- **Groundwater systems serving under 10,000 customers.** EPA is proposing that these systems would initially be required to only monitor twice within a 12-month period, with each sample 90 days apart.
- **Surface water systems.** All surface water systems would initially be required to monitor quarterly within a 12-month period.

In order to reduce costs for systems, systems would be allowed to use previously collected monitoring data to satisfy the initial monitoring requirements, if the sampling was conducted using EPA Methods 533 or 537.1 as part of UCMR 5 or other state-level or other appropriate monitoring campaigns. EPA is aware of many state and federal monitoring programs whose data would potentially satisfy the initial monitoring requirements.
If finalized, after rule promulgation, community water systems and non-transient, non-community water systems would conduct quarterly compliance monitoring. Based on initial monitoring or later compliance results, primacy agencies would have the authority to reduce compliance monitoring frequency for a system to once (for systems serving fewer than 3,300 persons) or twice (for systems serving 3,300 or more persons) every

three years if monitoring results are below the trigger level. The trigger level is set at one-third of the MCLs for PFOA and PFOS (1.3 ppt) and one-third of the Hazard Index MCL (0.33) for mixtures of PFHxS, GenX Chemicals, PFNA, and PFBS. Any system that monitors less frequently and finds sample results at or above the rule trigger level would need to revert to quarterly monitoring.

Reduced monitoring would reduce burden on water systems that demonstrate through sampling that they are at lower risk of PFAS contamination.

Question 8: Why is EPA setting a reduced-monitoring trigger level below the PQL for certain PFAS?

The proposed reduced-monitoring trigger level is set at a level that is useful in determining whether the contaminant is present in a sample rather than to determine its specific concentration. While measurements below the PQLs may be less definitive, they are appropriate for determining if PFAS are present and establishing monitoring frequency.

Question 9: Can systems utilize composite samples?

EPA is proposing not to allow composite samples. Composite sampling is an approach in which equal volumes of water from multiple entry points are combined into a single container and analyzed as a mixture. The reported concentration from the analysis of the composite samples therefore reflects the average of the concentrations from the entry points. This can potentially reduce analytical costs because the required analysis is reduced by combining samples into one. However, because PFAS are in the environment at low concentrations and precision is critical, incidental contamination could result in false positives.

Question 10: Will EPA consider granting monitoring waivers?

Based on consultation with state regulators and small public water systems, EPA believes that the ubiquity and environmental persistence of PFAS would make granting waivers challenging and is therefore not proposing to grant them. EPA is taking comment on whether water systems should be allowed to apply for a monitoring waiver of up to 9 years (one full compliance cycle) for proposed PFAS if after one year of quarterly sampling the results are below the trigger level of 1/3 of the MCL (1.3 ppt).

Question 11: How can a system comply with an MCL when it is set at the Practical Quantitation Level? Would any monitoring result above the PQL result in non-compliance?

Not necessarily. Compliance will be determined based on analytical results at each sampling point. For systems monitoring quarterly, compliance will be determined by running annual averages at the sampling point. If a system takes more than one compliance sample during each quarter at a particular location, the system must average all samples taken at that location during that quarter. A system would not be considered in violation of an MCL unless or until it has completed one year of quarterly sampling (except where a sample would be high enough to cause the annual average to exceed an MCL).

For example, if the results of sampling for PFOA at a compliance location for the most recent four quarters are 2.0, 1.5, 5.0, and 1.5 ppt, the values used to calculate the running annual average would be 0.0, 0.0, 5.0, and 0.0. In this case the PFOA running annual average would be 1.3 ppt and in compliance.

Question 12: Does EPA have PFAS treatment disposal guidance, especially regarding higher volumes of PFAS laden materials such as used carbon and anion exchange media?

A facility that has spent carbon or other media from treating PFAS and/or other contaminants must determine whether the material is a regulated waste. If the material was only used to treat PFAS, it is likely not considered hazardous waste (under federal statutes). EPA published "*Interim Guidance on the Destruction and Disposal of Perfluoroalkyl and Polyfluoroalkyl Substances and Materials Containing Perfluoroalkyl and Polyfluoroalkyl Substances*" that describes the options of landfilling, injection and thermal treatment for disposing PFAS laden materials. The guidance notes that thermal treatment techniques, including carbon reactivation, may allow PFAS to migrate to the environment. EPA and partners are undertaking research to further address the subject. EPA is also working to update this guidance in 2023. Materials used to treat PFAS may become hazardous if there are additional contaminants that are hazardous removed along with PFAS.

Question 13: What are Consumer Confidence Reports (CCR) requirements of the proposed rule?

A community water system (CWS) must prepare and deliver to its customers a CCR, also known as an Annual Water Quality Report, which provides information about their local drinking water quality as well as information regarding the water system compliance with drinking water regulations. If this rule is finalized as proposed, CWSs would be required to report measured levels of PFOA, PFOS, PFHxS, GenX Chemicals, PFNA, and PFBS, and the Hazard Index for the mixtures of PFHxS, GenX Chemicals, PFNA, and PFBS.

Question 14: What are the public notification requirements for PFAS under this proposed rule?

The proposed rule would require water systems to provide notification of an MCL violation as soon as practicable but no later than 30 days after the system learns of the violation. The notices would alert consumers of the violation and if there is a risk to public health.

Question 15: What is the timeline and process for state primacy?

Primacy agencies must have regulations for contaminants regulated under National Primary Drinking Water Regulations (NPDWRs) that are no less stringent than the regulations promulgated by EPA. States will have up to two years to develop regulations after the rule is final. EPA will provide guidance to support states, territories, and Tribes in obtaining primacy for the PFAS NPDWR. More information on primacy responsibilities under the Safe Drinking Water Act can be found at: https://www.epa.gov/dwreginfo/primacy-enforcement-responsibility-public-water-systems

Appendix D: Sensitive Environment Contacts

AGENCY	NAME & CONTACT INFORMATION	SENSITIVE ENVIRONMENT
NC Division of Conservation, Planning, and Community Affairs Natural Heritage Program	Visit the Natural Heritage Program's interactive maps of Natural Heritage resources to search for records within 2 miles of your project area or the database search tool for record summaries by county and USGS 7.5-minute topo map. You can also download GIS shapefiles of our data; see the "GIS Download" page for details. Email inquiries to: natural.heritage@ncdcr.gov	State Parks Areas Important to Maintenance of Unique Natural Communities Sensitive Areas Identified Under the National Estuary Program Designated State Natural Areas State Seashore, Lakeshore and River Recreational Areas Rare species (state and federal Threatened and Endangered) Sensitive Aquatic Habitat State Wild & Scenic Rivers
National Park Service Public Affairs Office	Anita Barnett, EEO Counselor & Environment Protection Specialist: Planning and Compliance Division Anita_Barnett@nps.gov (404) 507-5706 http://www.nps.gov/rivers	National Seashore, Lakeshore and River Recreational Areas National Parks or Monuments Federal Designated Wild & Scenic Rivers
US Forest Service	Heather Luczak, Forest NEPA Coordinator heather.luczak@usda.gov (828) 257- 4817	Designated and Proposed Federal Wilderness and Natural Areas National Preserves and Forests Federal Land Designated for the Protection of Natural Ecosystems
NC Division of Water Resources	Nora Deamer, Basin Planner Nora.Deamer@deq.nc.gov (919) 707-9116 General Basin Planning e-mail: DEQ.DWR.BasinPlanning@deq.nc.gov	State-Designated Areas for Protection or Maintenance of Aquatic Life

NC Division of Water Resources, cont'd	Michelle Raquet, Branch Supervisor michelle.raquet@deq.nc.gov (919) 707-9026 Ask for Clean Water Act 305b report	
NC Forest Service	Michael Foushee, Director, Safety, Planning & Analysis michael.foushee@ncagr.gov (919) 857-4820	State Preserves and Forests
US Fish & Wildlife Service	Pete Benjamin, Field Supervisor pete_benjamin@fws.gov (919) 856-4520 x 11	Endangered Species
NC Department of Natural and Cultural Resources	Renee Gledhill-Earley, Environmental Review Coordinator renee.gledhill-earley@dncr.nc.gov (919) 814–6579	National and State Historical Sites
NC Division of Coastal Management	Mike Lopazanski, Deputy Director Mike.lopazanski@deq.nc.gov (252) 515-5431 http://dcm2.enr.state.nc.us	Areas Identified Under Coastal Protection Legislation Coastal Barriers or Units of a Coastal Barrier Resources System
NC Wildlife Resources Commission	David Cox, Technical Guidance Supervisor David.Cox@ncwildlife.org (919) 707-4055	National or State Wildlife Refuges State lands designated for wildlife or game management Migratory pathways and feeding areas critical for maintenance of anadromous fish species within river reaches or lakes or coastal tidal waters Spawning areas critical for the maintenance of fish/shellfish species within river, lake or coastal tidal waters

US Army Corps of Engineers	Asheville Regulatory Field Office Dina Supple: (828) 271-7980 AshevilleNCREG@usace.army.mil Charlotte Regulatory Field Office Lisa Hreha: (704) 510-1441 CharlotteNCREG1@usace.army.mil Raleigh Regulatory Field Office Josephine Schaffer: (919) 554-4884 RaleighNCREG@usace.army.mil Washington Regulatory Field Office Nikki Dameron: (252) 975-1399 WashingtonNCREG@usace.army.mil Wilmington Regulatory Field Office Kasey Jones: (910) 251-4811 WilmingtonNCREG@usace.army.mil	Wetlands