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## Validation of Effective Removal of PFAS from Glassware Sampling Train Used for Evaluation of Thermal Treatment of PFAS

Elizabeth Riedel  
*University of Dayton*

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# **Validation of effective removal of PFAS from glassware sampling train used for evaluation of thermal treatment of PFAS**



Honors Thesis

Elizabeth Riedel

Department: Civil & Environmental Engineering & Engineering Mechanics

Advisors: Moshan Kahandawala, Ph.D., Denise Taylor, Ph.D., P.E.

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## Abstract

Per- and polyfluoroalkyl substances (PFAS) are anthropogenic chemicals that are extremely stable and highly toxic to the environment and human health. PFAS are used in a myriad of common products including nonstick cookware, water-resistant fabrics, personal care products, cosmetics, and aqueous film forming foam (AFFF). Because of their widespread use and resistance to degradation, PFAS have infiltrated the environment, including drinking water sources. To combat the spread of PFAS, various methods for treatment and removal of PFAS are being researched. A promising solution that has been identified for PFAS removal is thermal treatment, where degradation of PFAS occurs after exposure to exceedingly high temperatures. However, chemical characteristics of certain PFAS create the potential for them to adhere to equipment used during experimentation. For proper assessment of PFAS removal, it is essential to confirm that there is no carryover from sampling or contamination on the experimental equipment. This paper evaluates two techniques for their potential to effectively eliminate PFAS carry over on glassware used in a sampling train for thermal treatment. The first is a procedure used to clean glassware in the field when sampling for PFAS. The second is a modified version of the glassware cleaning procedure recommended in the OTM 45 protocol from EPA for PFAS sampling. In this experiment, glass impingers contaminated with solutions containing two of the most prevalent PFAS, perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS), were cleaned following the two separate procedures. Results from an analytical laboratory following EPA Method 537 showed that the second cleaning procedure was approximately five times more effective at removing PFOA and about two times more effective than the first cleaning procedure at the removal of PFOS from the impingers. Therefore, the second cleaning procedure is recommended for glassware used in PFAS experimentation.

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## List of Abbreviations

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AFFF – aqueous film-forming foams
CP1 – Cleaning Procedure #1
CP2 – Cleaning Procedure #2
DRE – destruction and removal efficiency
EPA – Environmental Protection Agency
PFAA – perfluoroalkyl substances
PFAS – per- and polyfluoroalkyl substances
PFCA – perfluoroalkylcarboxylic acids
PFOA – perfluorooctanoic acid
PFOS – perfluorooctanesulfonic acid
ppt – parts per trillion (ng/L)
HDPE – high-density polyethylene
IMP1 – experimental glass impinger #1
IMP2 – experimental glass impinger #2
MCL – maximum contaminant level
NPDWR – National Primary Drinking Water Regulation
OTM-45 – Other Test Method 45

## 1. Executive Summary

---

Perfluoroalkyl and polyfluoroalkyl substances (PFAS) are toxic, synthetic chemicals of emerging concern that have infiltrated the environment. These contaminants have been proven to cause adverse health effects, and humans are continuously exposed to them through consumer products, contaminated drinking water, and polluted air. Research is still being conducted to determine the best methods to remove PFAS from the environment as the Environmental Protection Agency (EPA) works to propose regulations for PFAS. An important factor to address when evaluating PFAS removal methods is the effective elimination of PFAS from experimental equipment. Proper cleaning of equipment assures that there are no contaminants remaining on the equipment that would otherwise create errors in the next measurements. Several methods are commonly used to clean sampling equipment used for PFAS experiments, but none of these have been tested and validated at the contamination levels used in this study.

In this thesis, two cleaning procedures are proposed as a solution for PFAS carry over on glassware used in a sampling train from incineration of biosolids. The first cleaning procedure (CP1) is based on the EPA's recommended procedure for cleaning PFAS-contaminated field equipment. The second cleaning procedure (CP2) was a variation of the glassware cleaning method described in the EPA's Other Test Method 45 (OTM-45) for PFAS sampling. These procedures are explained in detail in section 4.2 and section 8.1.

Two glassware cleaning methods were evaluated in this experiment by contaminating two glass impingers with high concentrations of perfluorooctanoic acid (PFOA) and Perfluorooctanesulfonic acid (PFOS) and following each cleaning procedure separately. These particular PFAS were selected because they are the most common and well-documented. Details for the experiments and analytical methods can be found in section 4 and section 8.

This study demonstrated that both cleaning procedures resulted in effective removal of PFAS from glassware at contamination levels of 15.8 mg/L for PFOA and 13.05 mg/L for PFOS. The second cleaning procedure (CP2) clearly demonstrated more efficient removal of the total amount of PFAS from the glassware and is the recommended procedure for cleaning PFAS-contaminated glassware. Analysis of the two cleaning procedures and further recommendations are outlined in section 5.



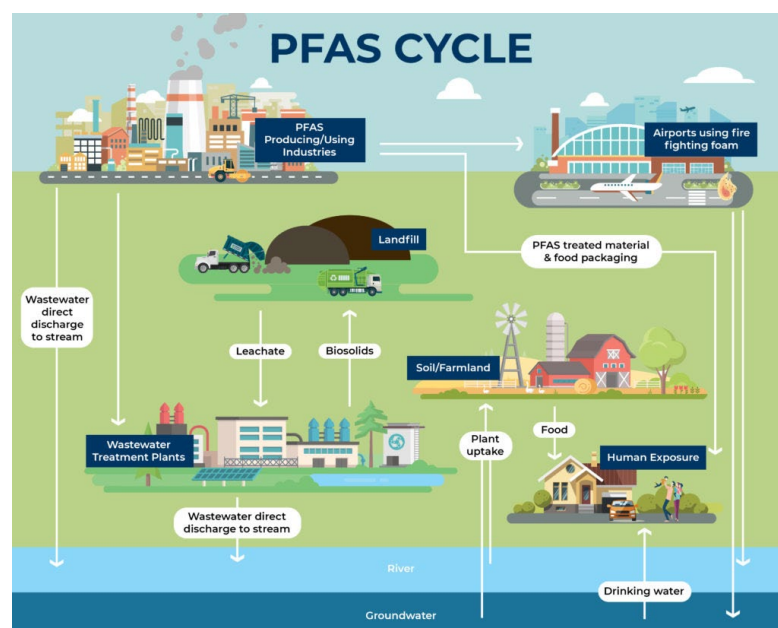
## 2. Literature review

### 2.1 Perfluoroalkyl and polyfluoroalkyl substances (PFAS)

Perfluoroalkyl and polyfluoroalkyl substances (PFAS) consist of a variety of synthetic, long-lasting chemicals that are contaminating the environment. The chemical structure of PFAS consists of strong carbon-fluorine bonds, making them highly stable and resistant to degradation (Kim et al., 2015). The distinctive fluorinated alkyl chains of PFAS contribute to their hydrophobic and lipophobic characteristics, which have made PFAS popular in commercial and industrial products (Wang et al., 2022). Some common consumer products that contain PFAS are cosmetics, hair and skin care products (Pütz et al., 2022), food packaging such as food contact papers (Schaidler et al., 2017), ready-to-eat vegetables (Piva et al., 2023), nonstick cookware (Sinclair et al., 2007), textiles, such as carpets, curtains, and car upholstery (Drage et al., 2023), insulations, construction materials, electronics, and electrical equipment (Bečanová et al., 2016). High concentrations of PFAS are also found in products such as suppressors used in the chromium industry and aqueous film-forming foams (AFFF) used to fight fuel fires at airports and military bases (Favreau et al., 2017).

### 2.2 Fate and transport of PFAS in the environment

PFAS is transported through wildlife and the environment through various pathways. Industries that manufacture PFAS and goods containing PFAS are a major source of environmental contamination. Facilities utilizing AFFF to fight fires are also sources of PFAS point-contamination due to polluted runoff. Products containing PFAS are often discarded into landfills, sent to wastewater treatment facilities and incinerators, or discharged directly into the environment (Houtz et al., 2013). The figure below displays different routes that PFAS travels through the environment and how PFAS can contaminate drinking water sources (Figure 2.1).



**Figure 2.1: PFAS pathways in the environment and drinking water contamination routes (Walnut Valley Water District, Walnut, CA)**

### **2.2.1 PFAS in landfills**

In landfills, PFAS break free from solid wastes as the solids undergo decomposition, leaving them to sink into landfill leachate. According to Coffin et al., (2023) one of the most abundant PFAS in municipal landfill leachate are short-chain perfluoroalkylcarboxylic acids (PFCAs). Short-chain PFAS compounds are more problematic when released into the environment because they are more stable and difficult to degrade compared to longer-chain PFAS (Horst et al., 2020). Polluted landfill leachate creates a high risk for contamination of groundwater – an important source of drinking water for many communities – if leachate collection systems should fail (Hepburn et al., 2019). Most landfills send collected leachate to wastewater treatment facilities, where the liquid waste is treated and released into the environment.

### **2.2.2 PFAS in wastewater treatment facilities**

Current technologies within existing wastewater treatment facilities do not have the capacity to effectively remove PFAS compounds. In fact, studies have shown that effluent from wastewater treatment plants contains higher levels of short-chain PFAS compared to the influent (Lenka et al., 2021; Semerád et al., 2020; Zhang et al., 2013). Although this does suggest that some long-chain PFAS are broken down in treatment processes, their degradation led to the creation of shorter chain PFAS, which are more stable and pose a greater threat to the environment. Effluent from wastewater treatment plants is typically discharged directly into the nearest river or stream, meaning that PFAS-contaminated water is constantly being released into aquatic environments. Effective methods for PFAS removal during the wastewater treatment process are desperately needed to mitigate this detrimental environmental impact.

Biosolids are another significant channel for PFAS contamination in the environment. Within the wastewater treatment process, sewage sludge is separated into solids and liquids. The liquids are treated to remove suspended solids, organic matter, excess nutrients, pathogens, and heavy metals and are released as effluent, and the solids are treated for removal of pathogens and heavy metals and are transformed into biosolids. Because of nutrients contained in organic matter, biosolids are commonly applied to land and used as fertilizer for agricultural purposes or disposed in landfills (Garg et al., 2023). Research has shown that PFAS have the potential to accumulate in biosolids, possibly due to adsorption to organic matter (Lenka et al., 2021; Semerád et al., 2020). This means that applying biosolids to land causes PFAS contamination of soil, rivers, streams, and groundwater. Soil and water contamination is especially problematic because PFAS has strong bioaccumulation potential in plants, invertebrates, humans, and other wildlife (Burkhard, 2021; Cheng et al., 2021; Liu et al., 2019). When humans are exposed to PFAS through consumption of polluted crops or water, PFAS begins to bioaccumulate in the body, leading to excretion of PFAS contaminated feces and urine that ultimately cycle back through the wastewater treatment process (Semerád et al., 2020; Stoiber et al., 2020).

## 2.3 Effects of PFAS on human health and proposed regulations

Human exposure to PFAS has been demonstrated to cause a variety of negative health effects. A study by Vuong et al. (2016) showed connections between PFAS in pregnant mothers and below average executive functioning, metacognition, and behavior regulation in their children. Exposure to PFAS has also been linked to reduced fertility, endocrine disruption, hormone interruptions, high cholesterol, and a compromised immune system (Anderko & Pennea, 2020; Rickard et al., 2022). Research on exposure to PFAS through drinking water also displayed higher risks of diabetes, heart attacks, Alzheimer's disease, kidney cancer, breast cancer, and Parkinson's disease (Mastrantonio et al., 2018). The wide variety of health effects from PFAS points to the need for regulations that support removal of PFAS from the environment.

As of April 2023, the EPA has proposed and passed several guidelines and regulations to combat the spread of PFAS in the environment and in our drinking water. In December 2022, the EPA eliminated an exemption that permitted facilities to bypass reporting the use of low concentrations of PFAS to the Toxics Release Inventory (Changes to Reporting Requirements for PFAS, 2022). Given that PFAS have exhibited harmful effects at concentrations as low as parts per trillion, this action carries considerable weight. A proposal to enforce specific concentrations of six PFAS compounds through the National Primary Drinking Water Regulation (NPDWR) was released by the EPA in March 2023. This includes enforcement of Maximum Contaminant Levels (MCLs) for PFOA and PFOS at 4.0 ng/L (ppt), and a mixture of PFHxS, PFNA, PFBS, and HFPO-DA (GenX) at a 1.0 Hazard Index, which is a sum of the individual PFAS hazard quotient ratios (the level of that particular PFAS in drinking water to its health-based water concentration value) (PFAS NPDWR Rulemaking, 2023). Adding PFAS to the NPDWR is the most progressive action that the EPA has taken so far because it sets enforceable limits on the levels of PFAS that are permitted in public water supplies, therefore reducing the exposure of millions of Americans to these toxic chemicals.

## 2.4 Methods for removal and treatment of PFAS

Research to determine the best technologies for removal and treatment of PFAS is ongoing. Some of the methods that are currently being investigated for removal of PFAS in water include sorption with activated carbon or ion exchange, advanced oxidation and reduction processes, thermal treatment, and microbial treatment (Merino et al., 2016). For most of these methods, however, further research is needed to determine their ability to effectively remove quantities of PFAS at smaller scales (ppt). For removal of PFAS from biosolids, researchers have been testing methods such as supercritical water oxidation, thermal hydrolysis, pyrolysis, hydrothermal liquefaction, and incineration (Garg et al., 2023). Techniques that promote biodegradation of PFAS are also important methods for natural removal of PFAS from the environment (Zhang et al., 2013).

### 2.4.1 Incineration of PFAS

As concerns grow over the application of PFAS-contaminated biosolids to croplands, the options for the fate of biosolids are limited to landfilling and incineration. Due to the

cyclical effect of landfilling biosolids, incineration is a preferable alternative. Thermal treatment of PFAS via incineration of biosolids has proven to be a promising solution for PFAS removal because extremely high temperatures are required to break the stable C-F bond in these compounds and achieve complete mineralization (Altarawneh et al., 2022). Mineralization is the complete defluorination of the perfluoroalkyl chain and the PFAS molecule. This results in fluorine atoms as fluoride or hydrogen fluoride and carbon dioxide (Horst et al., 2020). If complete mineralization does not occur, the result is products of incomplete combustion, which can include harmful pollutants released into the air (Merino et al., 2016). Pollutant emissions, specialized equipment, and high costs are some of the main challenges for incineration as a PFAS-removal method. Nonetheless, incineration of biosolids is a promising solution for destruction of PFAS in the environment.

## **2.5 PFAS interactions with sampling equipment**

An important factor to consider when performing experiments with PFAS is the interactions between PFAS and experimental equipment. Because PFAS is widely used in the manufacture of plastics, packaging, and other types of containers, there is potential for cross-contamination between experimental equipment and samples containing PFAS. Studies have also indicated that certain materials have a tendency to adsorb PFAS, creating the possibility of contaminant carryover between experimental replicates if the materials are not cleaned properly (Lath et al., 2019; Zenobio et al., 2022). In most cases, evidence revealed that PFAS was least likely to adsorb to glassware and most likely to adsorb to polypropylene (PP) and high-density polyethylene (HDPE) containers (Lath et al., 2019; Zenobio et al., 2022). Even though glassware had lower adsorption energies with PFAS, the small amounts of PFAS remaining are significant when analyzing removal of PFAS at a scale of parts per trillion (ppt or ng/L).

### 3. Experimental purpose and context

This study is based on a parent experiment where biosolids from a wastewater reclamation facility were injected with PFAS and incinerated at various temperatures and air flow residence times. The specific PFAS compounds tested were based off the National Institute of Standards and Technology (NIST) domestic sludge Standard Reference Mater (SRM) 2781, along with additional PFAS of interest. An impinger sampling train, consisting of four glass impingers filled with deionized (DI) water in an ice bath, was included in this experiment to capture soluble PFAS and incineration by-products (Figure 3.1). Before experiments were conducted, a blank test was performed without introducing any PFAS into the system. This was completed to check for ambient PFAS levels in the system. After a significant amount of PFAS was found in a blank sample run in this experiment, the sources of these contaminants were investigated. In this study, residual PFAS on impinger glassware due to ineffective cleaning procedures was analyzed as a potential source of contamination from experimental carry over.

The aim of this study is to validate and confirm a glassware cleaning procedure that effectively decontaminates glassware used in PFAS removal experiments. Eliminating residuals and contamination from carry over on sampling equipment is critical in experiments for proper analysis of PFAS removal.

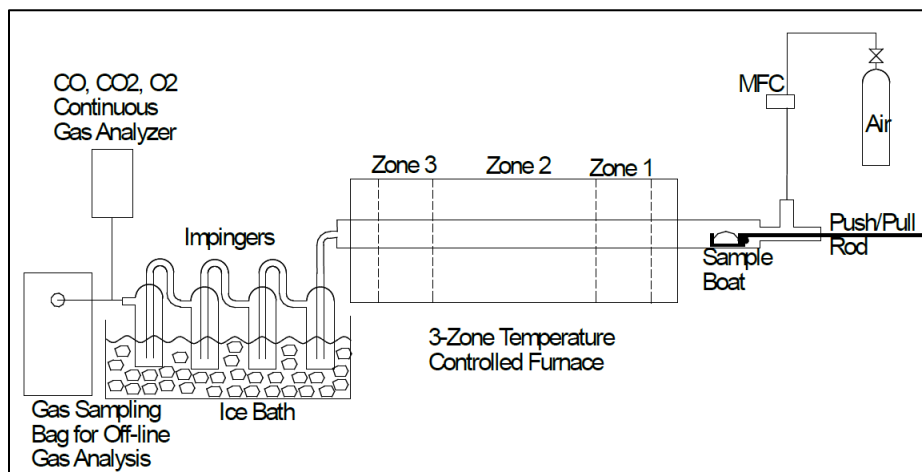


Figure 3.1: Parent incineration experimental set-up

## 4. Materials and methods

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Two glassware cleaning methods for PFAS removal were tested in this experiment. The first cleaning procedure (CP1) follows the “Decontamination Procedure for Equipment used for Sample Collection for Per- and Poly-Fluoroalkyl Substances (PFAS)” as outlined by the Laboratory Services and Applied Science Division of the EPA (ASBPROC-206-R4) (U.S. EPA, 2021). The second procedure (CP2) was a modified version of the recommended procedure for glassware cleaning before PFAS sampling in the Other Test Methods 45 (OTM-45) protocol from the Emission Measurement Center of the EPA (U.S. EPA, 2019).

### 4.1 Glassware contamination simulation

In this study, PFOA (95% purity level) and PFOS (95% purity level) were purchased to contaminate glass impingers. A high-concentration contamination mixture of PFOA and PFOS was formed to achieve an optimal removal efficiency and simulate the maximum destruction and removal efficiency goal for the parent incineration experiment. The 1L mixture contained approximately 15.8 mg/L of PFOA and 13.05 mg/L of PFOS, which was calculated based on the concentration and weight of PFAS in the biosolids mixture for incineration in the parent experiment (30g). The volume of the glass impingers in the sampling train was taken into account during analysis to establish the amount of carry over. Detailed calculations can be found in section 8.4.

The PFOS and PFOA solution was mixed for 24 hours before it was poured into each impinger. The impingers were contaminated by shaking and swirling the solution in the impingers for 15 minutes and then leaving the impingers to rest at room temperature for one hour. Then, one impinger was cleaned with the first cleaning procedure and another impinger was cleaned with the second cleaning procedure, as outlined in section 4.2.

### 4.2 Experimental cleaning methods

The first cleaning procedure (CP1) aligned closely with the EPA’s standard procedure for cleaning field equipment used for PFAS sampling. The first step involved sonicating the first glass impinger (IMP1) in a ultrasonic bath consisting of a 4% concentration of Liquinox detergent and deionized water at 55°C for 15 minutes. Then, the impinger was rinsed with tap water three times and deionized/distilled water three times. A 10% nitric acid solution was prepared for each decontamination session and used to rinse the impinger. After dumping the nitric acid solution, the impinger was rinsed again with deionized/distilled water and placed under a ventilated hood to air dry.

The second cleaning procedure (CP2) was a modified combination of the EPA’s standard procedure for cleaning field equipment used for PFAS sampling and OTM-45. In this procedure, Liquinox soap at a 4% concentration was dispensed into the second glass impinger (IMP2), and a brush was used to scrub the impinger with hot tap water. After that, the impinger was rinsed three times with PFAS-free deionized water. Next, the impinger was rinsed with a sequence of solvents that included acetone, methylene

chloride, hexane, and methanol. The impinger was then oven-dried at 450°C for approximately 16 hours and sealed in plastic bags or aluminum foil for future use.

### 4.3 Analysis of cleaning procedures

Once the glassware was cleaned, 500 mL of deionized (DI) water was poured into the glassware and let sit for 24 hours. For the first impinger (IMP1), the DI water was obtained from the laboratory facilities, but the second impinger (IMP2) was filled with PFAS-free DI water obtained from an analytical laboratory. After 24 hours, the DI water was collected from each impinger into separate bottles. High-density polyethylene (HDPE) bottles were rinsed with methanol, let sit to air dry, and used to collect the two separate samples from each impinger. To ensure full sample recovery, the empty impingers were then rinsed with more DI water (500 mL). The DI water from this rinse was added to the sample bottle with the original DI water sample for each impinger. The HDPE sample bottles were stored in a refrigerator at approximately 6.0°C until they were shipped to the analytical laboratory.

Collected samples from the glassware were sent to Eurofins Knoxville Laboratory to be analyzed for 36 various PFAS compounds of interest that were part of the targeted analysis in the parent experiment (Table 4.1). Analyzing 36 samples provides insight into any potential sample carry over from the parent incineration experiment or impurities in the parent PFAS compounds. The laboratory followed EPA Method 537.1, which uses solid phase extraction and liquid chromatography/tandem mass spectrometry (LC/MS/MS) to determine concentrations of PFAS in water samples (Shoemaker & Tettenhorst, 2020).

**Table 4.1: Target PFAS Analytes**

Common Name	Abbreviation	CAS Registry Number
<b>Perfluoroalkylcarboxylic acids (PFCAs)</b>		
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-2
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluorotridecanoic acid	PFTTrDA	72629-94-8
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorohexadecanoic acid	PFHxDA	67905-19-5
Perfluorooctadecanoic acid	PFODA	16517-11-6
<b>Perfluorinated sulfonic acids (PFSAs)</b>		

Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoS	79780-39-5
<b>Perfluorinated sulfonamides (FOSAs)</b>		
Perfluorooctanesulfonamide	PFOSA	754-91-6
N-ethylperfluoro-1-octanesulfonamide	EtFOSA	4151-50-2
<b>Perfluorinated sulfonamidoacetic acids (FOSAAs)</b>		
N-methyl perfluorooctanesulfonamidoacetic acid	MeFOSAA	2355-31-9
N-ethyl perfluorooctanesulfonamidoacetic acid	EtFOSAA	2991-50-6
<b>Perfluorinated sulfonamide ethanols (FOSEs)</b>		
2-(N-methylperfluoro-1-octanesulfonamido) ethanol	NMeFOSE	24448-09-7
2-(N-ethylperfluoro-1-octanesulfonamido) ethanol	NEtFOSE	1691-99-2
<b>Fluorotelomer sulfonates (FTS)</b>		
4:2 Fluorotelomer sulfonic acid	4:2 FTS	757124-72-4
6:2 Fluorotelomer sulfonic acid	6:2 FTS	27619-97-2
8:2 Fluorotelomer sulfonic acid	8:2 FTS	39108-34-4
10:2 Fluorotelomer sulfonic acid	10:2 FTS	120226-60-0
<b>Fluorinated Replacement Chemicals</b>		
4,8-Dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4
Hexafluoropropyleneoxide dimer acid	HFPO-DA (GenX)	13252-13-6
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	756426-58-1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9



## 5. Results and discussion

During thermal treatment experimentation, effective destruction of PFAS is quantified by the destruction and removal efficiency (DRE) of the parent PFAS compounds. Performing a system blank test allows for the determination of any residual or leftover PFAS in the sampling train that could affect the DRE results. In this study, the initial concentration of PFAS was substantially higher than the background levels found in the sampling train glassware blank test. A higher concentration allowed for analysis of the residual parent PFAS at the targeted DRE during experiments. Therefore, the effectiveness of CP1 and CP2 was evaluated by analyzing the concentrations of the remaining PFAS and their potential impacts on the DRE.

### 5.1 PFAS destruction and removal efficiency

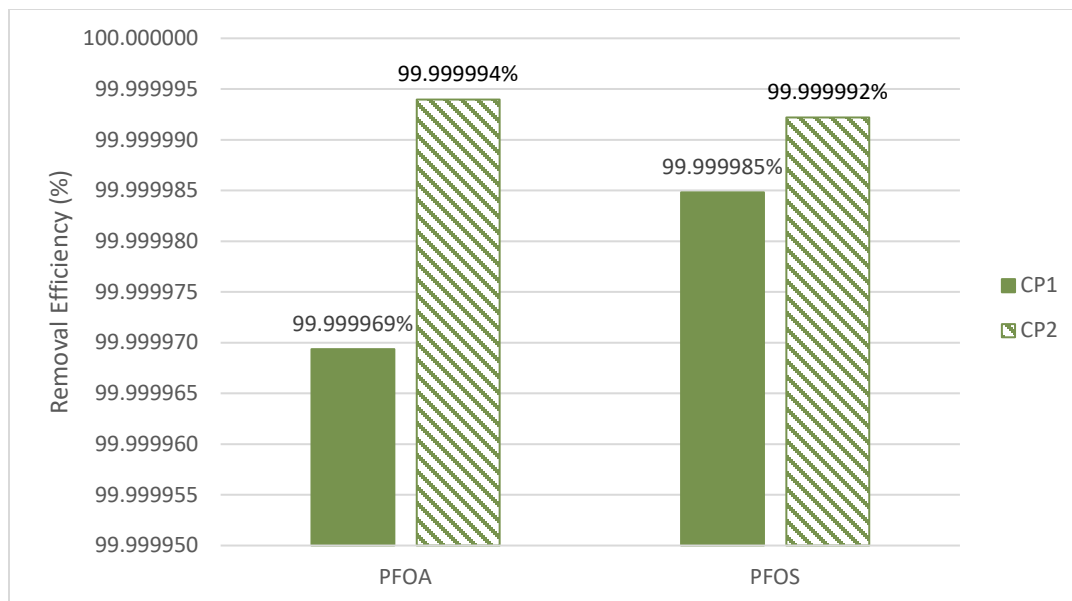
As mentioned previously, the PFOA and PFOS solution was highly concentrated to target the destruction and removal efficiency (DRE) of the thermal treatment experiment. The DRE is a measurement of the performance of an incinerator based on its ability to release molecules from organic compounds into the air (Eq 5-1). For principle organic hazardous constituents (POHCs), the required DRE set by the Resource Conservation and Recovery Act (RCRA) is 99.99%. Therefore, the targeted DRE for the thermal treatment of PFAS study was 99.99%. The goal for this experiment was to acquire the DRE necessary to mimic the conditions of the thermal treatment study.

$$(Eq\ 5-1) \quad DRE = \frac{(W_{in} - W_{out})}{W_{in}} \times 100$$

Where  $W_{in}$  = mass flow rate of POHC input

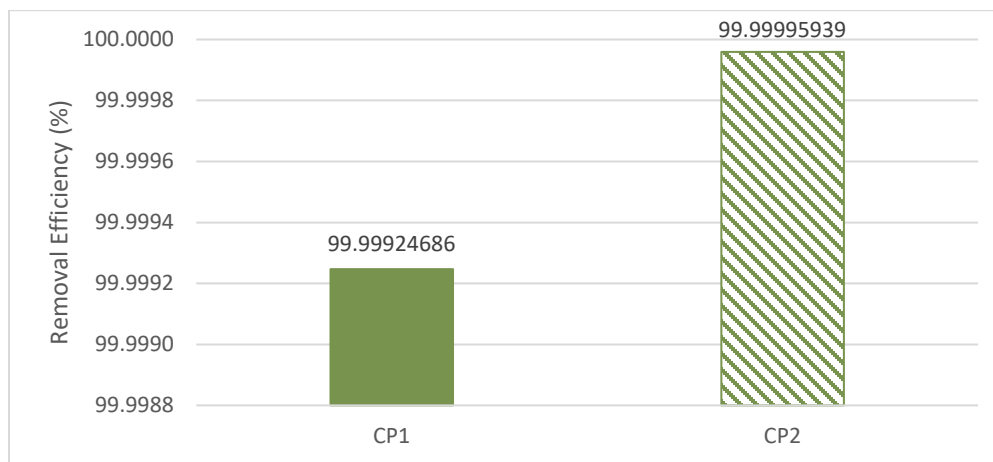
$W_{out}$  = mass flow rate of POHC output

Both glassware cleaning procedures demonstrated the ability to effectively remove PFOA and PFOS carry over on glassware (Figure 5.1). Compared to the first procedure (CP1), the second procedure (CP2) featured a significant increase in the removal efficiency for PFOA and a slight increase in the removal efficiency for PFOS. The overall removal efficiency for each cleaning procedure must be much greater than the 99.99% DRE goal for PFAS to reach this DRE goal. Both procedures have removal efficiencies that were significantly greater than 99.99%. Based on Figure 5.2, CP2 removed slightly more PFAS than CP1, with a difference of approximately 0.00071% between the two procedures. The probable cause for this variation between glassware cleaning procedures is the difference in solvents used in each procedure. In CP1, nitric acid was the only solvent used to decontaminate the glassware. Although nitric acid is an effective polar solvent, it was most likely ineffective at removing all the PFAS adhered to the glassware. CP2 included acetone, methylene chloride, hexane, and methanol, which formed a sequence of polar and nonpolar solvents containing a multitude of carbon-hydrogen bonds. These solvents were most likely more effective at removing the PFAS adhered to the glassware.



**Figure 5.1: Removal efficiencies for PFOA and PFOS**

Note: Figure 5.1 displays the removal efficiencies of PFOA and PFOS for the DI water samples collected from IMP1 after CP1 and IMP2 after CP2.



**Figure 5.2: Total PFAS removal efficiency for each cleaning procedure**

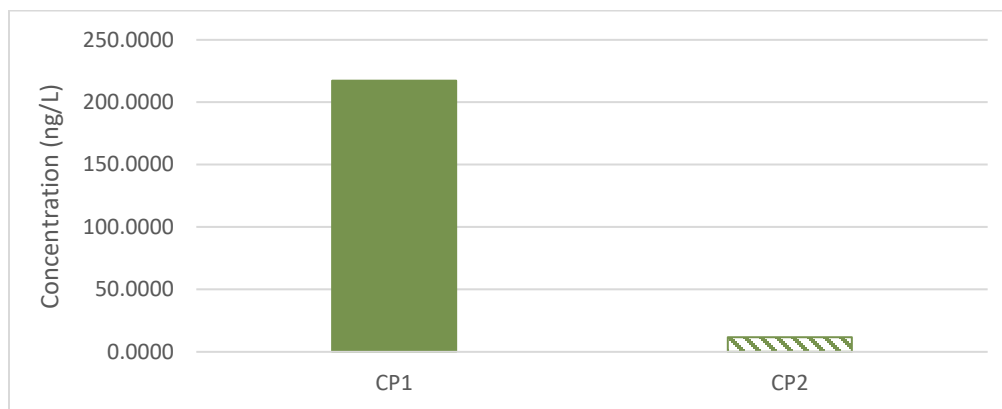
Note: Figure 5.2 displays the removal efficiencies of all targeted PFAS compounds (Table 4.1) analyzed with EPA Method 537.1 for the DI water samples collected from IMP1 after CP1 and IMP2 after CP2.

## 5.2 PFAS remaining in glassware samples

Although both cleaning procedures resulted in acceptable removal efficiency values, the second cleaning procedure demonstrated greater efficiency at total PFAS removal because there were lower concentrations and fewer PFAS compounds remaining (Figure 5.3). Results from CP1 displayed higher concentrations of PFBA and PFHxA relative to

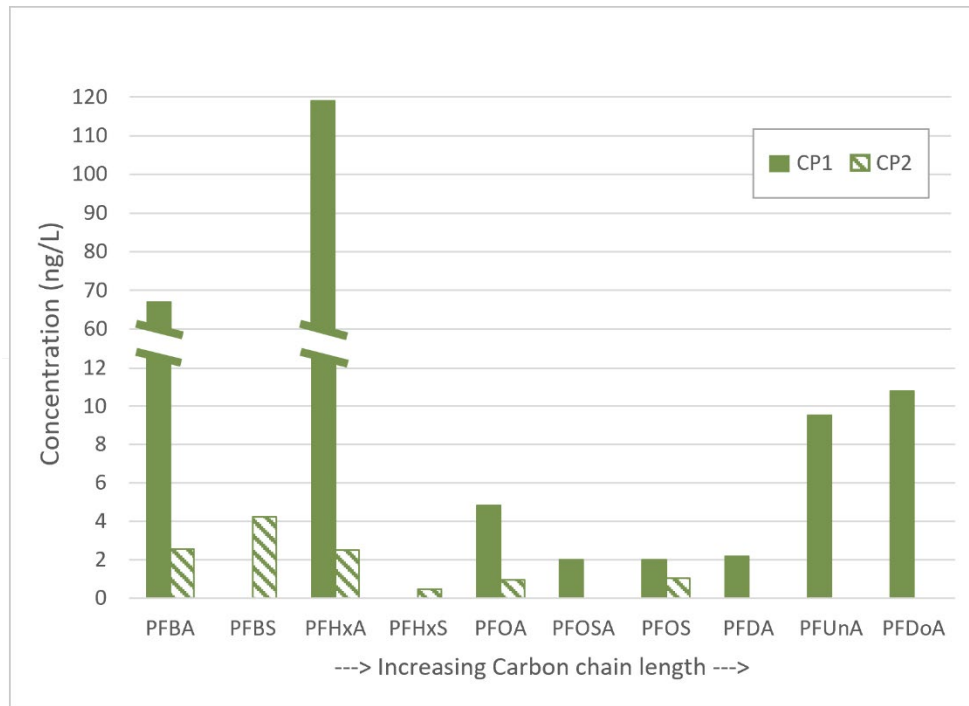
CP2 (Figure 5.4). PFOSA, PFDA, PFUnA, and PFDoA were also recovered from glassware after CP1 but were absent from the sample for CP2. A potential explanation for the source of these compounds is the DI water used throughout CP1. Due to time and cost constraints, the DI water was not analyzed for PFAS, but it could have contained trace amounts of PFAS. Another probable source of these excess PFAS compounds is adsorption to the glassware during incineration experimentation, or contaminant carry over from previous use due to ineffective cleaning. PFAS test compounds for the incineration experiment included PFHxA, PFOSA, PFDA, PFUnA, and PFDoA, which were all recovered solely from CP1. A study on adsorption of PFAS to containers showed that longer-chain PFAS, such as PFOS, PFOA, and PFOSA were more likely to adsorb to containers than shorter-chain PFAS (Zenobio et al., 2022). The carbon chain lengths of PFDA, PFUnA, and PFDoA are longer than PFOSA and PFOS, indicating a strong likelihood of adsorption to the glass impingers from the parent experiment. It is probable that these compounds adsorbed to both IMP1 and IMP2 and their absence from the results for CP2 indicates that CP2 was more effective than CP1 at removal of these longer-chain compounds.

For CP2, PFAS-free DI water was used rather than the DI water in the laboratory. This water was analyzed separately for the same PFAS compounds as the samples (Table 4.1) to assure an absence of PFAS. However, trace amounts of PFBA, PFOA, and PFOS were discovered in the “PFAS-free” DI water, which may have been significant contributing factors to the concentrations PFAS in the results for the second cleaning procedure (Figure 5.5). It is also possible that these compounds leached into the PFAS-free water during transport, such as from shipping or sampling containers, or from handling in the laboratory. The trace amounts of PFAS that were detected in the “PFAS-free” water emphasize the sensitivity of PFAS experimentation and demonstrate the need for good laboratory practices to prevent contamination and implement precise and accurate measurements in PFAS research.



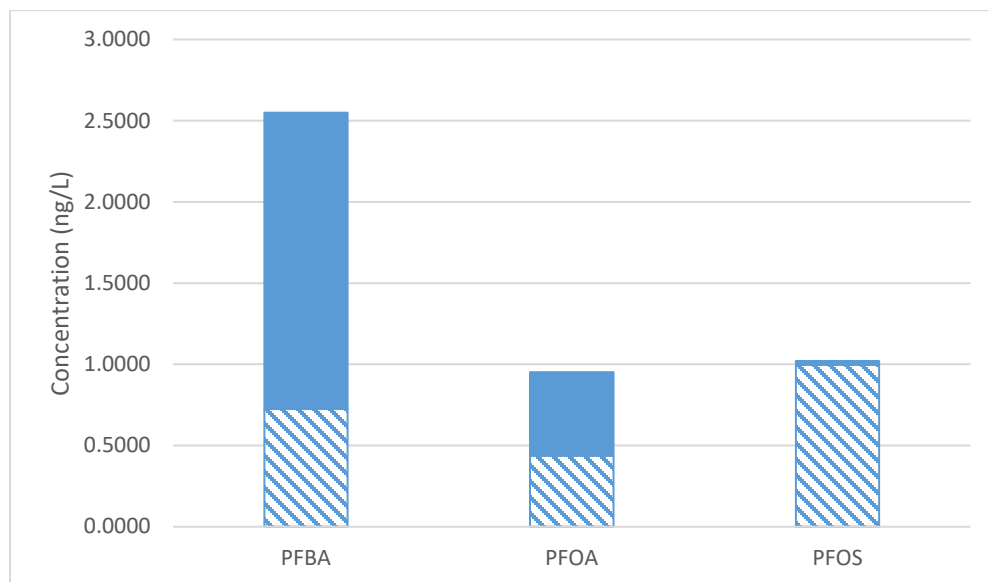
**Figure 5.3: Total PFAS concentration remaining after each cleaning procedure**

Note: Figure 5.3 displays the remaining total concentrations of all targeted PFAS compounds combined (Table 4.1) for the DI water samples collected from IMP1 after CP1 and IMP2 after CP2.



**Figure 5.4: Breakdown of PFAS remaining in samples after cleaning**

Note: Figure 5.4 displays the remaining concentrations of each PFAS compound detected in the DI water samples collected from IMP1 after CP1 and IMP2 after CP2.



**Figure 5.5: Composition of PFAS in CP2 results from "PFAS-free" DI water**

Note: Total column height displays the concentration of the PFAS compound in the results for CP2. Striped column portions indicate the concentration of PFAS detected from the "PFAS-free" DI water for each compound.

### **5.3 Recommended procedure**

Based on the findings and data presented in this study, the second cleaning procedure clearly demonstrated a higher effectiveness than the first cleaning procedure. The biggest differences between the two procedures were the solvents used to decontaminate the glassware and the type of DI water used to rinse the glassware. CP2 included acetone, methylene chloride, hexane, and methanol, which were more effective at breaking the firm carbon-fluorine bonds in the PFAS compounds. PFAS-free DI water was also used in CP2, likely reduced PFAS contamination during the cleaning process. Therefore, the second cleaning procedure is recommended for future glassware cleaning and decontamination of PFAS.

### **5.4 Experimental constraints, improvements, and suggestions**

There were several factors within this study that prevented further analysis of each cleaning procedure. The most impactful experimental constraints were time and cost. The procedure for analyzing PFAS in water samples is very expensive, which limited the number of samples per cleaning procedure outlined in this paper. Reliance on an external laboratory for PFAS analysis contributed to the time limitation.

Due to these constraints, this experiment is considered a preliminary screening, and more samples should be collected and analyzed to obtain statistically meaningful data in future experiments. Future research should include a comparison of multiple samples per glassware cleaning procedure, explore the effectiveness of cleaning procedures to remove PFAS from various types of experimental equipment, and investigate the effects of temperature and residence time of PFAS in glassware on the performance of various solvents.

## 6. Conclusion

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In this study, the effectiveness of two cleaning procedures to remove PFAS from glassware used in incineration experiments was investigated. Eliminating residuals or sample residue from experimental equipment is essential for proper assessment of experimentation. This is especially important for experiments involving PFAS destruction since research is still being conducted to determine specific contamination levels of PFAS for regulatory purposes. Current protocols recommended by the EPA for cleaning PFAS contaminated glassware were validated in this experiment.

Although both cleaning procedures exhibited removal efficiencies that were substantially greater than 99.99%, the CP2 removal efficiency was slightly higher than that of CP1, which demonstrates an improvement in the cleaning efficacy of CP2. The second cleaning procedure was more successful at PFAS removal than CP1 because it included a sequence of polar and nonpolar solvents as well as “PFAS-free” DI water. Even though the “PFAS-free” DI water contained minute levels of PFAS, the resulting concentrations of PFAS were significantly lower than the concentrations from the first cleaning procedure. Due to the efficient performance of the second cleaning procedure, a variation of the OTM-45 glassware cleaning method is recommended for decontamination of glassware used for PFAS experimentation. Application of this recommended glassware cleaning procedure should include water obtained from a laboratory that is PFAS-free for effective removal of PFAS at trace levels.

More research is needed to continue the investigation into effective removal of PFAS residuals from experiment equipment. Further research should investigate the effectiveness of various cleaning methods on different types of material surfaces to ensure the validity of continued research on PFAS elimination methods.

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## 8. Appendix

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### 8.1 Detailed experimental cleaning methods

Cleaning Procedure #1 (CP1):

1. Place two impingers in an ultrasonic bath with 40 mL of Liquinox detergent (4% concentration) and DI water at 55°C for 15 minutes.
2. Rinse glassware with hot tap water three times.
3. Rinse glassware with deionized/distilled water three times.
4. Rinse glassware with 10% nitric acid solution (190 mL DI water with 20 mL of 69% Nitric acid). A fresh nitric acid solution to be prepared for each decontamination session.
5. Dump nitric acid solution and rinse glassware with deionized/distilled water three times.
6. Completely air dry under a ventilated hood.

Cleaning Procedure #2 (CP2):

1. Prepare Liquinox soap (4% concentration) for the glassware cleaning (impingers).
2. Dispense soap (4% conc.) five times from soap dispenser into the impinger bottle.
3. Use hot tap water and scrub the impinger with brush and then clean with hot tap water.
4. Repeat steps 2 and 3 five more times (A total of 6 washes with soap and hot water should be performed).
5. Rinse all glassware three (3) times with deionized (DI) water.
6. Solvent rinse three (3) times all glassware with the following sequence of solvents: acetone, methylene chloride, hexane, and methanol.
7. Bake glassware at 450°C for approximately 16 hours in oven.
8. Clean glassware and tools will be sealed in plastic bags or aluminum foil for future use.
9. Clean glassware with MeOH/ 5% NH<sub>4</sub>OH solvent prior to use.

### 8.2 Contamination details and calculations

Impingers #1 and #2 were contaminated based on the procedure listed below.

- a. Filled a clean (washed with procedures above) glass bottle with 1000 mL deionized water using clean (washed with procedures above) 250 mL graduated cylinder.
- b. Contaminated with perfluorooctanoic acid (PFOA, solid) and perfluorooctanesulfonic acid (PFOS, liquid):
  - i. PFOA:  $(3.16 \text{ mg/g}) \cdot (30\text{g}) / (6\text{L}) = 15.8 \text{ mg/L}$
  - ii. PFOS:  $(2.61 \text{ mg/g}) \cdot (30\text{g}) / (6\text{L}) = 13.05 \text{ mg/L}$
- c. PFOS:

- i. Weighed empty glass vial
- ii. Weighed vial with 1 mL PFOS added with pipette
- iii. Weighed vial with 2 mL PFOS (1 mL added with pipette)

Condition	Mass (grams)
Empty Vial	2.121218
Vial w/ 1 mL PFOS	3.279082
Vial w/ 2 mL PFOS	4.523450

- iv. Calculated average of 1.201116 g/mL PFOS
  - v. Calculated 10.86  $\mu$ L needed for contamination
- d. PFOA:
- i. Rinsed the weigh pan with methanol, let air dry, then weighed the pan.
  - ii. Added PFOA to the pan (after zero-out pan mass) until mass reaches 0.0158 grams.

Condition	Mass (grams)
Weigh Pan	0.672937
PFOA	0.015842

- iii. Determined 0.0158 grams needed for contamination
- e. Added 10.86  $\mu$ L PFOS and 0.015842 grams PFOA to the glass bottle with the 1000 mL deionized water and placed on magnetic plate with magnetic stirrer at level 6 for 24 hours.
  - f. After 24 hours, poured about 400 mL of contaminated solution into each impinger (impingers #1 & #2).

### 8.3 Final results

**Table 8.1: PFAS Results for Cleaning Procedure #1 (CP1)**

Analyte	Acronym	Formula	Result	Unit	% Destruction
Perfluorobutanoic acid	PFBA	C3F7CO2H	67	ng/L	
Perfluorobutanesulfonic acid	PFBS	C4HF9O3S	--		
Perfluorohexanoic acid	PFHxA	C6HF11O2	119	ng/L	
Perfluorohexanesulfonic acid	PFHxS	C6HF13O3S	--		
Perfluorooctanoic acid	PFOA	C8HF15O2	4.84	ng/L	0.999999694
Perfluorooctanesulfonamide	PFOSA	C8H2F17NO2S	1.98	ng/L	
Perfluorooctanesulfonic acid	PFOS	C8HF17O3S	1.98	ng/L	0.999999848
Perfluorodecanoic acid	PFDA	C10HF19O2	2.16	ng/L	
Perfluoroundecanoic acid	PFUnA	C11HF21O2	9.52	ng/L	
Perfluorododecanoic acid	PFDoA	C12HF23O2	10.8	ng/L	

<b>TOTAL PFAS CONCENTRATION</b>	<b>217.28</b>	<b>ng/L</b>	0.999992469
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**Table 8.2: PFAS Results for Cleaning Procedure #2 (CP2)**

Analyte	Acronym	Formula	Result	Unit	% Destruction
Perfluorobutanoic acid	PFBA	C3F7CO2H	2.55	ng/L	
Perfluorobutanesulfonic acid	PFBS	C4HF9O3S	4.22	ng/L	
Perfluorohexanoic acid	PFHxA	C6HF11O2	2.5	ng/L	
Perfluorohexanesulfonic acid	PFHxS	C6HF13O3S	0.475	ng/L	
Perfluorooctanoic acid	PFOA	C8HF15O2	0.952	ng/L	0.99999994
Perfluorooctanesulfonamide	PFOSA	C8H2F17NO2S	--	ng/L	
Perfluorooctanesulfonic acid	PFOS	C8HF17O3S	1.02	ng/L	0.999999922
Perfluorodecanoic acid	PFDA	C10HF19O2	--	ng/L	
Perfluoroundecanoic acid	PFUnA	C11HF21O2	--	ng/L	
Perfluorododecanoic acid	PFDoA	C12HF23O2	--	ng/L	
<b>TOTAL PFAS CONCENTRATION</b>			<b>11.717</b>	<b>ng/L</b>	0.999999594

**Table 8.3: PFAS Results for PFAS-free DI water**

Analyte	Acronym	Result	Unit
Perfluorobutanoic acid	PFBA	0.725	ng/L
Perfluorobutanesulfonic acid	PFBS	--	ng/L
Perfluorohexanoic acid	PFHxA	--	ng/L
Perfluorohexanesulfonic acid	PFHxS	--	ng/L
Perfluorooctanoic acid	PFOA	0.438	ng/L
Perfluorooctanesulfonamide	PFOSA	--	ng/L
Perfluorooctanesulfonic acid	PFOS	0.996	ng/L
Perfluorodecanoic acid	PFDA	--	ng/L
Perfluoroundecanoic acid	PFUnA	--	ng/L
Perfluorododecanoic acid	PFDoA	--	ng/L
<b>TOTAL PFAS CONCENTRATION</b>		<b>2.159</b>	<b>ng/L</b>

## 8.4 Sample removal efficiency calculations

PFOA removal efficiency for CP1:

$$RE = \frac{\left(15.8 \frac{mg}{L} * 10^6 \frac{ng}{mg}\right) - \left(4.84 \frac{ng}{L}\right)}{\left(15.8 \frac{mg}{L} * 10^6 \frac{ng}{mg}\right)} * 100 = 99.9999694\%$$

PFOS removal efficiency for CP1:

$$RE = \frac{\left(13.05 \frac{mg}{L} * 10^6 \frac{ng}{mg}\right) - \left(1.98 \frac{ng}{L}\right)}{\left(13.05 \frac{mg}{L} * 10^6 \frac{ng}{mg}\right)} * 100 = 99.9999848\%$$

Total PFAS removal efficiency for CP1:

$$RE = \frac{\left[\left(15.8 \frac{mg}{L} * 10^6 \frac{ng}{mg}\right) + \left(13.05 \frac{mg}{L} * 10^6 \frac{ng}{mg}\right)\right] - \left(217.28 \frac{ng}{L}\right)}{\left(15.8 \frac{mg}{L} * 10^6 \frac{ng}{mg}\right) + \left(13.05 \frac{mg}{L} * 10^6 \frac{ng}{mg}\right)} * 100$$

$$RE = 99.99924686\%$$