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Industrial SCWO for the Treatment of PFAS/AFFF Within a Water Matrix

Office of Research and Development
Center for Environmental Solutions and Emergency Response
Land Remediation and Technology Division

Industrial Supercritical Water Oxidation for the Treatment of Polyfluorinated Alkyl Substances/Aqueous Film Forming Foam Within a Water Matrix

Full-Scale Technology Test Report

by

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NOTICE/DISCLAIMER

The U.S. Environmental Protection Agency, through its Office of Research and Development, funded and conducted the research described herein under an approved Quality Assurance Project Plan (Quality Assurance Identification Number K-LRTD-003047-QP-1-0. It has been subjected to the Agency's peer and administrative review and has been approved for publication as an EPA document.

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Gregory Sayles, Director

Center for Environmental Solutions and Emergency Response

ABSTRACT

Supercritical water oxidation (SCWO) technology presents significant environmental advantages for treating hazardous industrial wastewater and sludges. SCWO is a rapidly maturing technology as many pilot- and full-scale plants are being tested for the destruction of recalcitrant pollutants.

The U.S. EPA and General Atomics (GA) have conducted joint studies on performance and engineering assessment of industrial-scale SCWO for the removal efficacy and destruction of per- and polyfluoroalkyl substances (PFAS) within aqueous film-forming foam (AFFF). The tests were intended to treat low-to-medium dilutions of AFFF to identify the destruction efficiency of PFAS and measure possible emissions. The study aimed to evaluate SCWO as an alternative to incineration destruction and disposal approaches for stockpiled legacy aqueous film-forming foams (AFFF) products used for firefighting applications. Although the U.S. EPA has tested SCWO and other non-incineration technologies at pilot- and industrial demostration-scale, this study was performed at full-scale.

The tests achieved 99.99% destruction and removal efficacies of targeted PFAS and total organic carbon. The tests show that hydrothermal flame as an internal heat source reduces residence time, with minimum corrosion, by controlling the wall temperature and construction materials. SCWO process shows limited partial and incomplete oxidation products that are entrained in the solution, and no fluorinated compounds were detected in the stack gas emission. The effluent from SCWO is easily collected, analyzed, and can be recycled. Gaseous effluents from SCWO were carbon dioxide and oxygen with traces of carbon monoxide and trace quantities of hydrothermal heat source oxidized products. The hydrogen fluoride formed within the reactor was neutralized, precipitated from the SCWO reactor water solution, and removed from the SCWO reaction vessel. The study provided additional data on the effectiveness of SCWO as an alternative technology for treating high PFAS-concentrated aqueous waste.

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EXECUTIVE SUMMARY

General Atomics (GA) (San Diego, CA) and U.S. EPA – Office of Research and Development (EPA-ORD) entered into an agreement in November 2020 to allow for testing of the removal efficacy and destruction of per- and polyfluoroalkyl substances (PFAS) within aqueous film-forming foam (AFFF) using supercritical water oxidation (SCWO) technology currently available on the market. The intent was to conduct treatability studies of low-to-medium dilutions of AFFF and to identify the destruction efficiency of PFAS per the agreed-upon quality assurance project plan (QAPP).

This document is intended to provide an overview of the GA SCWO technology tests, a summary of the results, and discuss various opportunities for use. Available information regarding the project is briefly summarized within this document. This document aims to examine the destruction of PFAS in diluted AFFF using this specific technology; this document does not represent an exhaustive review of all SCWO technologies or apply to all PFAS sources. Liquid influent feed stream and both liquid and gas effluent stream samples were collected and tested using various methods including, but not limited to EPA Method 537.1 for quantitative analysis of targeted PFAS in liquids, total organic carbon, chemical oxygen demand, total organic fluoride, non-targeted PFAS analysis, elemental analysis, and real-time measurement of stack emission flow to confirm the absence of various gases. Sorbent tubes and canisters were used to sample gas for volatile organic compounds, PFAS, and other reaction by-products.

In all tests, PFAS and total organic carbon (TOC) destruction efficiencies were > 99.99%. PFAS target compounds were not detected above the background level in the gas analyses. The sorbent tubes detected many trace organic compounds but no fluorinated compounds. After appropriate caustic neutralization of the reaction products, there was no HF detected in effluent streams. After 50 hours of operation, limited corrosion was observed since there was a slight increase in chromium concentrations in the effluent stream. Fluorine appeared mainly as a fluoride ion in the liquid effluent. Trace amounts of fluorinated hydrocarbon (FH) were also found in the liquid effluent; however, it is unclear if it came from the contaminated quench water (softened tap water) that was shown to have higher PFAS levels than the liquid effluent.

Note that some qualitative interpretation is provided regarding Non-Targeted Analysis (NTA), and only some NTA work is comparable. Limited data on the SCWO degradation pathways for the different PFAS substances are available.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

ACRONYMS AND ABBREVIATIONS

Acronyms	Definition
ADQ	audit of data quality
AFFF	Aqueous film forming foam
ASTM	American Society for Testing and Materials
CALC	Calculated
CCC	continuing calibration check
CEMM	Center for Environmental Measurement and Modeling
COC	Chain of custody
COD	Chemical oxygen demand
CTS	Calibration transfer standard
DAS	Data Acquisition System
DE	Destruction Efficiency
DQI	Data quality indicator
DRE	Destruction and removal efficiency
EDD	Electronic data deliverable
ELAP	Environmental Laboratory Accreditation Program
EPA	Environmental Protection Agency
Est	Estimated
FB	Field blank
FRB	field reagent blank
GA	General Atomics
GA-EMS	General Atomics-Electromagnetic Systems
GLS	Gas-Liquid Separator
GPM	Gallons Per Minute
HF	hydrogen fluoride
HMI	Human Machine Interface
HP	High Pressure
IDC	initial demonstration of capability
iSCWO	Industrial Supercritical Water Oxidation
L	Liter
LC/MS/MS	liquid chromatography/tandem mass spectrometry
LE	Liquid effluent
LFB	laboratory fortified blank
LFSM	laboratory fortified sample matrix
LP	Low Pressure
lpm	Liters per minute
LRB	laboratory reagent blank
MCL	maximum contaminant level
MDC	minimum detectable concentration
mL	Milliliter
MRL	Method Reporting Limit
ng	Nanogram
ORD	Office of Research and Development
PFAA	perfluoroalkyl acid

PFAS	Per- and polyfluoroalkyl substances
PFBA	perfluorobenzoic acid
PFC	perfluoro chemical
PFCA	polyfluorocarboxylic acid
PFDA	perfluorodecanoic acid
PFHxA	perfluorohexanoic acid
PFNA	perfluorononanoic acid
PFOA	perfluorooctanoic acid
PFOS	perfluorooctane sulfonate
PFOSA	perfluorooctane sulfonamide
PFPeA	perfluoropentanoic acid
PFSA	perfluorosulfonic acid
PI	Principal Investigator
PITT	PFAS Innovation Technology Team
PO	purchase order
POC	Point of contact for EPA procurement
ppb	parts per billion
PPE	personal protective equipment
ppm	parts per million
PTFE	polytetrafluoroethylene
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RSD	relative standard deviation
sccm	Standard cubic centimeter per minute
scfm	Standard cubic feet per minute
SCWO	Supercritical Water Oxidation
SD	San Diego
SDM	scientific data management
SDS	safety data sheet
SOW	statement of work
SPE	solid phase extraction
TOC	Total Organic Carbon
TOCOR	Task Order Contracting Officer’s Representative
TSA	technical systems audit

1. INTRODUCTION

1.1 PROJECT SUMMARY

An agreement between General Atomics-Electromagnetic Systems (GA-EMS) and Environmental Protection Agency – Office of Research and Development (EPA-ORD) was made in November 2020 to initiate a test program to process a specified source of an aqueous film-forming foam (AFFF) that contained per- and polyfluoroalkyl substances (PFAS). The intent was to conduct treatability studies of low to medium dilutions of AFFF and determine the destruction efficiency of PFAS per the agreed upon quality assurance project plan (QAPP). The test facility utilized was a commercial-scale industrial demonstration supercritical water oxidation (iSCWO) unit located at GA-EMS' facility in San Diego, CA, used for demonstrations. This is the same size system that GA-EMS has been manufacturing for commercial waste destruction since 2013.

A full-day preliminary workup test and system preparation were followed by three performance tests conducted at reactor temperatures about 650 °C and pressures near 3,200 psi. The water-to-AFFF ratio ranged from 217 to 34, and the AFFF concentration was increased throughout the four-test sequence. This document provides the basic process flows, operating conditions, a summary of the data collected, and a summary of the iSCWO demonstration facility. The report also contains:

- The liquid and gas phase sampling protocols.
- The sample collection procedure.
- Analytical methods for assessing SCWO treatment performance and destruction efficiency of targeted compounds.

1.2 PROBLEM DEFINITION

In 2019, the EPA published a comprehensive PFAS action plan (EPA, 2019a), indicating it intends to develop maximum contaminant levels (MCLs) for perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) in drinking water and to take steps to classify PFAS as hazardous substances. Because of these potential regulatory drivers and the lack of demonstrated treatment approaches for a wide range of PFAS waste streams, there is a need to evaluate innovative technologies. EPA formed the PFAS Innovative Treatment Team (PITT) to assess existing and emerging destruction methods for PFAS that are being examined by EPA, academia, industry and others. The PITT investigated the efficacy of several treatment approaches while improving the understanding of by-products to avoid creating new environmental issues. These efforts examined the feasibility, performance, and cost of treatment approaches to validate possible solutions applicable to various PFAS waste streams. (Inside EPA, 2020b).

Determination of optimal strategies for reducing the environmental impacts of PFAS is a critical challenge for EPA and the nation. PFAS have strong carbon-fluorine chemical bonds that lead to persistence in the environment and make complete destruction difficult. One important source of PFAS in the environment originates from the use of aqueous film-forming foams

(AFFF) in firefighting applications. Due to their unique characteristics, PFAS have been used as suppressants in AFFF firefighting products for decades, and some concentrated formulations (concentrates) contain significant amounts of PFAS (e.g., 3% PFAS). AFFF formulations have been improved over time to reduce environmental impact, but large stockpiles of legacy and unused PFAS-AFFF concentrates require treatment and disposal. In this report, AFFF refers to Class B foams that contain some long-chain PFAS and fluorotelomers, as opposed to the modern fluorotelomer AFFF, which contain mainly short-chain PFAS (ITRC, 2018).

The PITT investigated several technical approaches for the destruction and disposal of unused AFFF concentrates. This document pertains to one of these projects that General Atomics Electromagnetic Systems is executing. This project sought to improve EPA's understanding of a specific embodiment of a treatment approach called supercritical water oxidation (SCWO) which has been developed at GA-EMS since 1992.

1.3 BACKGROUND ON SCWO

Supercritical water oxidation (SCWO) makes use of the unique properties that water exhibits at conditions of above 374 °C, and 3210 psia for the destruction of organic waste compounds and toxic wastes (Figure 1). The typical SCWO reactor operating temperature and pressure are 600 – 650 °C and 3400 psia, respectively. At these conditions supercritical water (SCW) has intermediate properties between liquid and vapor including lower fluid density, and order of magnitude reduction of the dielectric constant that makes water behave as organic compounds (Tester et al. 1993). Various oxidants have been used including high-pressure air, pure oxygen, hydrogen peroxide and other oxidizing agents. The organic waste, SCW and oxidant are miscible which creates an excellent condition for oxidation with negligible mass transfer limitation during SCWO decomposition of organics. SCW has the low viscosity and the high density of a liquid, with no surface tension, good fluidity and no diffusion rate determining steps in reaction involving SCW.

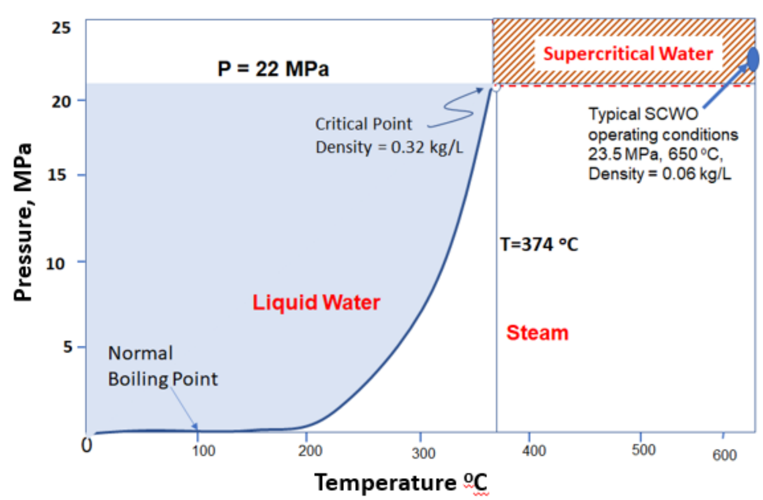

Figure 1: *Phase diagram of water showing the operating conditions for GA SCWO operation condition*

SCW is an excellent solvent for organic compounds (Japas and Franck 1985, Thomason and Modell 1984) and becomes completely miscible with air (Japas and Franck, 1966). Single-phase operation can eliminate mass transfer limitations, with the added benefit of high intrinsic reaction rates provided by the relatively high temperature (400 - 700 ºC). In the presence of SCW, most organic compounds react rapidly with oxygen to form CO_2 and water. Essentially complete mineralization of pollutants occurs within contact times of seconds to minutes (Modell 1985, Savage 1999).

The SCWO environment exhibits many unique characteristics that are favorable for treatment of recalcitrant contaminants. The degradation rates are likely to be controlled by reaction kinetics instead of the mass transfer rate and the solution loses its dielectric constant and the solubility of dissolved salts reduced considerably. Thus, SCWO can be an attractive destruction option when incineration is not economical or releases partially degraded products, or the waste is too toxic to treat biologically ((Modell 1982, Wightman 1981, Yang and Eckert 1988). Over the past four decades, SCWO technology has proven to be an effective method for treating different chemicals – from concentrated chlorinated hydrocarbons to chemical weapon agents. To combat corrosive conditions, the second and third generations use corrosive resistant materials, such as titanium, and add alkali to neutralize halogenated acids (Marrone and Hong 2009). There have been studies into the SCWO treatment of contaminated soils (Kocher et al. 1995), various types of sludge (Blaney et al. 1995, Cooper et al. 1997, Goto et al. 1998), solid particulates (Kodra and Balakotaiah 1994), and ion-exchange resins from nuclear power plants (Dubois et al. 1996, Pisharody et al. 1996).

Sludge is one of the complex wastes for which SCWO might be best suited since it contains sufficiently high organic levels such that auxiliary fuel is not needed or greatly diminished. Unlike other disposal approaches, such as incineration and landfilling, SCWO does not require a dewatering step. For aqueous wastes containing 1 to 20 wt% organics, supercritical water oxidation is less costly than activated carbon treatment and far more efficient than wet oxidation. Kodra and Balakotaiah have shown that in many cases, autothermal operation of the SCWO process for dilute wastewaters is feasible only with the addition of auxiliary fuel (Kodra and Balakotaiah 1994).

The medium for SCWO can be homogeneous, where the reaction occurs in single phase, or heterogeneous including solid-fluid oxidation reactions in SCW. Halogenated organic compounds typically separate halogen atoms under SCWO conditions. For example, oxidation of chlorinated compounds releases potentially corrosive chloride (Savage 1999). Oxidation of trichloroethylene and trichloroethane in the presence of H_2O_2 oxidant produced mainly CO_2 and hydrochloric acid (Foy et al. 1996). Under SCWO conditions, both hydrolysis and oxidation occur at a comparable rate. Oxidant additives shift product distribution from formaldehyde, methanol, and hydrogen to CO and CO_2 assisting complete oxidation.

The reaction mechanism of SCWO appears to involve homogeneous, free radical, gas phase oxidation chemistry in the same temperature regime. Therefore, quantitative, predictive, mechanism-based reaction models for SCWO can be developed based on the gas phase combustion kinetics database which is used to determine elementary reaction steps. However, the chemical equilibrium constants should include the fluid-phase nonidealities.

1.4 PROJECT OBJECTIVES

The overall goal was to evaluate the effectiveness of SCWO in destroying PFAS compounds present in AFFF. Tests were planned to conduct SCWO operations to treat AFFF at different dilutions to effectively degrade and defluorinate the full range of PFAS structures identified in selected AFFF samples.

The primary samples were analyzed using high-resolution mass spectrometry methods to measure the decomposition of a wide range of PFAS structures, including the most commonly measured perfluoroalkyl acids (i.e., PFOS and PFOA) and their polyfluorinated precursors that have been observed during hydrothermal treatment applications. Secondary samples were used to conduct non-target chemical analysis for air and liquid emission streams. Sample analysis included a range of metals in an attempt to estimate corrosion products, if any.

The US EPA used multi-criteria decision-making to assess the broad efficacy, energy requirements, and emissions from several SCWO operations tests. The results can be compared to conventional hazardous waste disposal methods to determine whether SCWO can be an alternative technology for managing various high moisture content PFAS wastes and concentrates, however, this assessment is outside the scope of this report.

For EPA-ORD, the overall goal of this limited scope project was to better understand the dynamics of treating AFFF using the iSCWO technology provided by GA-EMS. The data obtained will inform decision-makers where it may optimally fit in the range of appropriate waste management options, such as destruction of AFFF concentrate and potentially other PFAS-impacted wastewater solids, industrial residuals or waste, and spent media. As part of this collaboration, GA-EMS aims to better understand EPA-ORD's research into this topic area, develop measurement approaches for PFAS and other compounds in the subject waste streams, and improve knowledge of their processes and treatment emission control efficacy. This limited scope study was intended to conduct treatability of AFFF using iSCWO on a best effort basis by both parties and prior test experience with other technology vendors (Krause et al., 2021). The detailed project objectives are listed below.

This project was divided into primary, secondary, and tertiary objectives by conducting multiple iSCWO tests of PFAS-contaminated waste at various concentrations. The reactor performance is determined based on the physical influent and effluent samples acquired and analyzed by a certified commercial laboratory. For all testing, there was a mandate to execute trials with the highest degree of documented safety possible since compounds such as hydrogen fluoride (HF) may be generated as part of the process. EPA participation in the onsite testing was covered under an EPA safety plan.

1.4.1 PRIMARY OBJECTIVE

The primary objective of this project was to investigate the targeted PFAS destruction in the input material to the SCWO unit through measurement of PFAS levels in pre-SCWO influent and post-SCWO effluent and other measures as per the QAPP. The GA-EMS team operated their iSCWO demonstration plant in accordance with all safety management and standard operating procedures to ensure an accurate dilute AFFF feed to the system. The EPA-ORD team was responsible for sample collection, submittal to the third-party analytical labs using appropriate quality assurance measures, and managing the analytical results for evaluation. These samples, under this objective, were used for the "primary lab analysis." Additional, or "secondary samples", were collected to address the secondary objective.

1.4.2 SECONDARY OBJECTIVE

The second objective was to understand the extent to which PFAS can be destroyed and the effectiveness of SCWO to eliminate any degradation products (aka, fluorinated hydrocarbons, or FH). An EPA-ORD objective was to process, in part or in total, the "secondary samples" for a special analysis by EPA using other non-target analysis approaches, such as high-resolution LC-QTOF-MS. In addition, exploratory gas stack sampling of the GA-EMS iSCWO process using sorbent materials and evacuated canisters for targeted and non-targeted PFAS compounds analyses was performed to examine potential PFAS emissions.

1.4.3 TERTIARY OBJECTIVE

In addition to the chemistry and analytical requirements to support PFAS destruction, the third objective was intended to obtain process performance information on both the suitability and process cost of iSCWO technology for the treatment of PFAS containing liquid/slurry waste (note that other tests to demonstrate slurry feed capability have been performed by GA-EMS and could be combined with data from this study for applicability). The analyses included in this study will provide information on whether the treatment conditions and the reactor selected for the iSCWO are suitable to treat AFFF. This assessment includes ease or possibility of the reaction and the issues of corrosion and plugging.

1.5 GENERAL ATOMICS SYSTEM

The GA-EMS iSCWO process combines organic materials with water and an oxidant at approximately 650 °C (1200 °F) and 3,336 psia in a continuous-flow reactor configuration. At these supercritical conditions, water exhibits unique properties, combining physical properties of both liquid and gas, which improve its waste destruction capabilities. Gases and organics that are insoluble in water at ambient conditions are miscible with supercritical water, thus allowing the oxidant to react quickly with the organic material. The organic materials are oxidized to yield mainly carbon dioxide (CO_2) and water, while allowing nitrogen (N_2) and inorganic mineral species, depending on the presence of their precursors in the feed materials, to pass through.

A typical iSCWO configuration consists of feed and effluent tankage, several feed pumps, a startup heater, a pressure vessel reactor, a high-pressure (HP) air compressor, a pressure letdown system, and a gas-liquid separator (GLS) as shown in Figure 2.

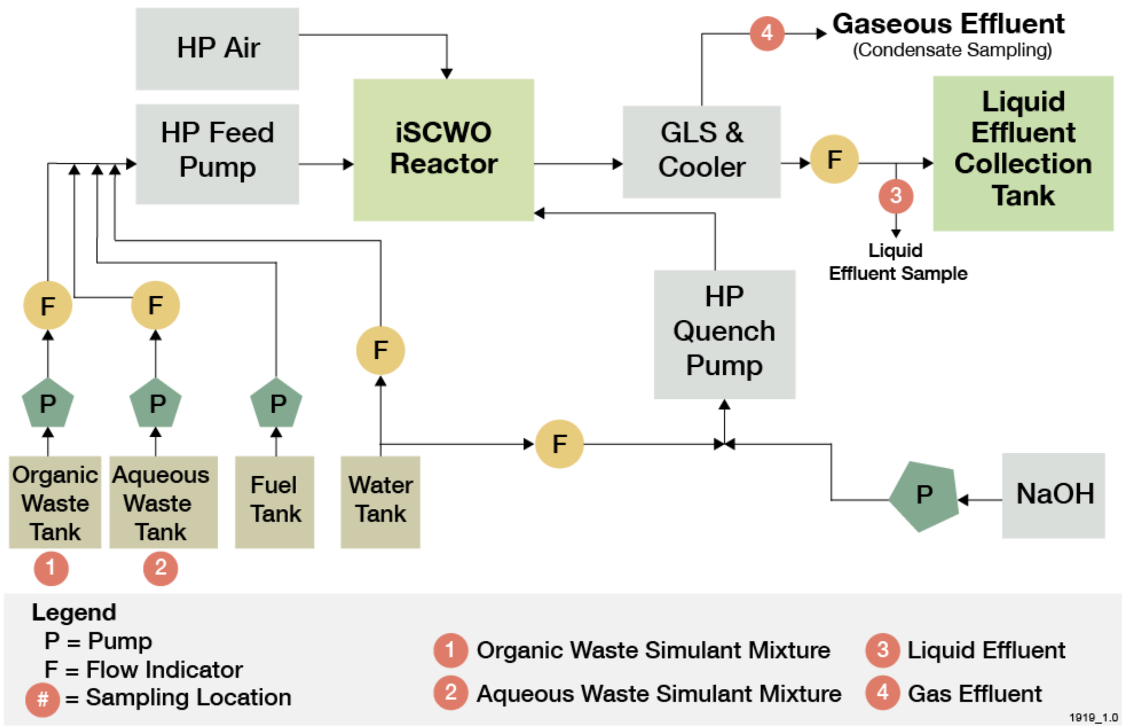

Figure 2: *Block Flow Diagram for the Industrial Demostrial-scale Plant*

The feed system typically consists of batch waste feed tanks, a softened water feed tank, and an auxiliary fuel feed tank. Each feed tank has its own low-pressure (LP) pump that delivers liquid at controlled flow rates to a single HP feed pump, which pumps the fluids to the pressurized iSCWO reactor vessel. The GA iSCWO demonstration-plant system incorporates a high pressure (HP) feed pump which is capable of pumping up to about three gallons per minute (GPM) of liquid at 3,336 psia. The waste feed/auxiliary, fuel/softened water mixture is injected into the reactor through a feed nozzle, which is also used to inject air to supply O_2 as the oxidant. The feed nozzle is designed to ensure effective mixing of the oxidant with the fuel/water/waste feed at the inlet of the reactor.

The iSCWO reactor consists of a pressure-bearing outer shell surrounding an easily removable, pressure-balanced liner. The liner can be fabricated from various corrosion-resistant alloys to allow tailoring of the reactor corrosion protection system to a wide range of feed characteristics. A titanium liner was used for the testing based on typical practice. The reactor temperature is monitored by two temperature probes, one of which is a single-point thermocouple and the other a three-point thermocouple. To maintain a low reactor wall temperature, quench water is introduced into the gap between the liner and the reactor. When the quench water reaches the bottom of the reactor, it is introduced to the exiting liquid reaction product to establish a liquid effluent temperature well below the critical point and assist in flushing out the iSCWO products. The quenched liquid effluent exits through the bottom of the reactor and then enters a capillary tube section of the pressure letdown system. The length of the capillary tube is sized to achieve a nominal reactor operating pressure of 3,336 psia at a flow rate of 3 GPM. After the capillary tube, the effluent passes through an expansion chamber to a GLS where the effluent is separated into its gas and liquid constituents at atmospheric pressure. The gaseous effluent comprises N_2, steam, CO_2, and O_2, and vents to the atmosphere through a demister. Canister samples of emission gases are analyzed in this study. The liquid effluent passes through a heat exchanger for cooldown before being discharged to the liquid effluent collection tank.

The GA iSCWO design utilizes a vessel reactor as opposed to an elongated pipe reactor. This configuration provides several advantages when processing liquid wastes such as PFAS solutions:

- Vessel reactor geometry facilitates uniform temperature and reaction
- Large reactor cross section reduces tendency for solids plugging
- Low reactor surface to volume ratio so that much smaller area requires corrosion protection
- Low surface to volume ratio and compact geometry minimizes replacement of reactor liner if necessary
- Large reactor cross section provides large amount of fluid back-mixing at nozzle, allowing injection of cold feeds and bypassing problems of preheat exchanger corrosion and plugging
- Vessel geometry and large cross section enable use of low alloy pressure vessels

As shown in Figure 2, an option to add alkali as sodium hydroxide (NaOH) with the quench stream is available. Alkali addition is useful to neutralize acid-forming wastes and was used for this demonstration program to neutralize the HF formed by the AFFF feed waste. A 35 wt% NaOH solution was used for this purpose.

Figure 3 and Figure 4 show views of the two skids that make up the iSCWO industrial demonstration scale plant used for this testing. A fully automated human-machine interface (HMI) controls the iSCWO industrial demonstration plant. All data channels for temperature, pressure, flow, and online analytical instruments are scanned and stored at a prescribed time interval, typically every 2 seconds, on a data acquisition system (DAS). The data can be accessed at any time, even when operating, and was used for analysis and data plots. The operator view of the main control screen is shown in Figure 5. Of the four temperatures shown within the reactor, TE-601 is the single thermocouple and TE-600, -602, and -603 are the triple thermocouples that span the top half of the reactor.

Figure 3: *Industrial Dememostration Plant iSCWO Main Skid*

Figure 4: *Industrial Dememostration Plant iSCWO two Stage Compressor Skids*

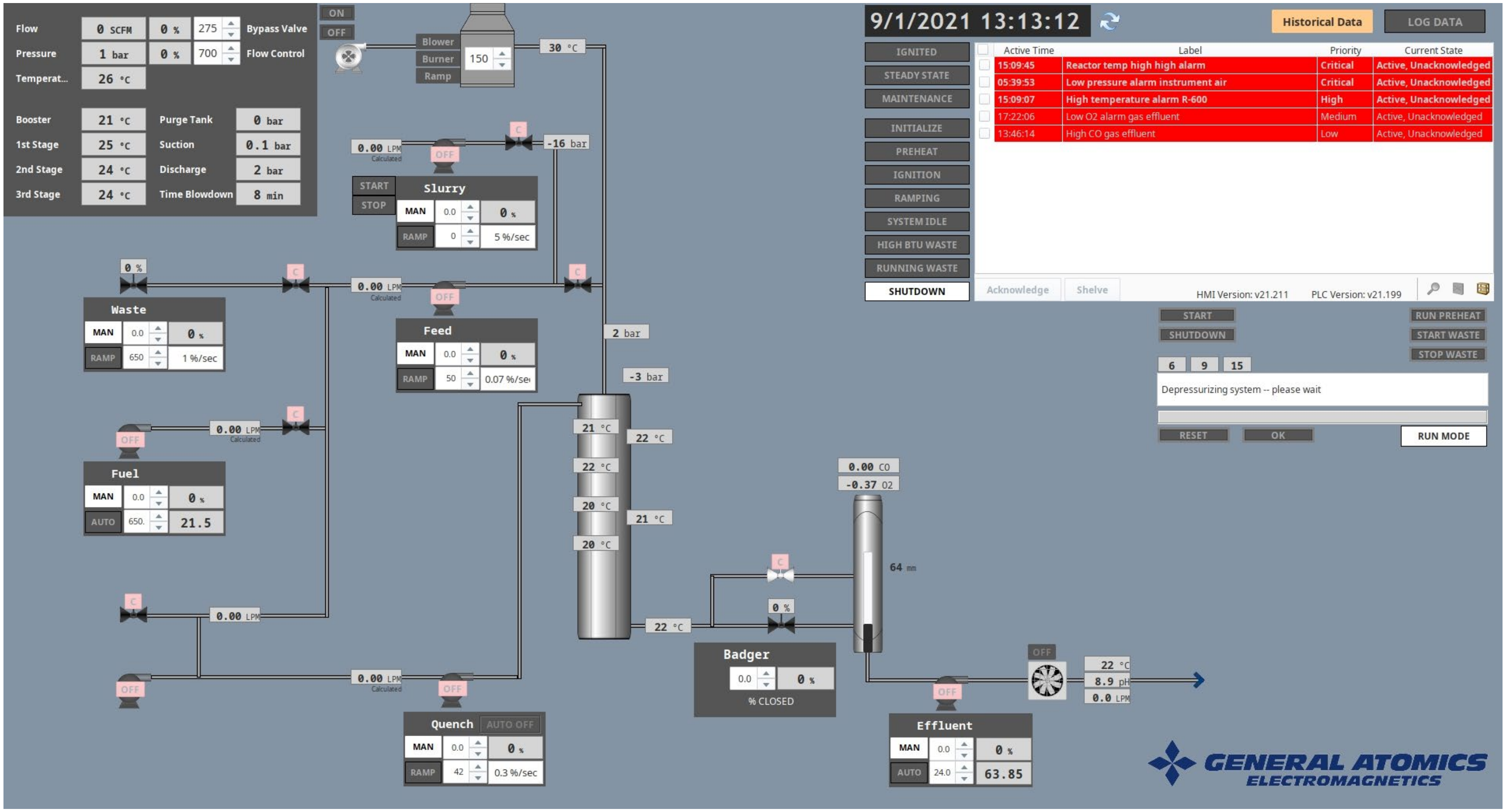

Figure 5: *Industrial Dememostration Plant iSCWO HMI Screen*

2. METHODS

2.1 FEED AND SYSTEM PREPARATION

A U.S. military specification (Milspec) aqueous film-forming foam Aer-O-Water (AFFF) 3% (National Foam) was obtained for these tests (Figure 2). This product is still in use by fire departments because of its superior flexibility, fire performance on hydrocarbon fuels, and environmental responsibility due to the reduced sulfur content. The aqueous film is produced by the fluorocarbon surfactant reducing the surface tension of the foam solution to a point where the surface tension of the fuel can support the solution. The surface tension of the fuel directly influences the effectiveness of the aqueous film.

Figure 6: *Three 5-gallon Buckets used for SCWO Test*

Legacy fluorotelomer AFFFs that were manufactured in the U.S. (1970 – 2016) contain some long-chain PFAS and encompass all other brands of AFFF besides 3M Lightwater (Schultz et al. 2006). Many AFFF contain polyfluorinated precursors that are shown to degrade to perflurooctanoic acid (PFOA) and other polyflurocarboxylic acids (PFCAs) in the natural environment (Weiner et al. 2013) or may contain trace quantities of PFOA as an unavoidable byproduct of the manufacturing process. Legacy fluorotelomer-based AFFF foams have historically included predominantly short-chain (C6) PFAS with formulations ranging from about 50–98% short-chain and the balance as long-chain PFAS. Many of the long-chain PFAS content of these foams have the potential to break down in the environment to PFOA and other PFCAs, but not to perfluorooctane sulfonate (PFOS) or other PFSAs (Weiner et al. 2013). Table 1 shows the AFFF composition provided by the EPA-ORD as described within the safety data sheet (SDS).

Table 1: Aer-O-Water AFFF Composition from SDS

Component	CAS Number	Concentration (%)
Water	7732-18-5	60-70
Diethylene Glycol Monobutyl Ether	112-34-5	10-20
Ethylene Glycol	107-21-1	5-15
Synthetic Detergent	Proprietary	1-10
Fluoroalkyl Surfactants	Proprietary	1-5
Surfactant	Proprietary	<1

The AFFF concentrate was mixed with water in a 350-gallon cone-bottom tank for feed to the iSCWO system. An antifoaming agent, Sigma-Aldrich Antifoam C Emulsion, was added to avoid formation of foam within the mixing tank. The mixed proportions are shown in Table 2.

Table 2: Feed Preparation overview of the four tests

Test	Water Vol (L)	AFFF Vol (L)	Water:AFFF Ratio	Antifoam (mL)
Workup	821	3.8	217	8.25
A	1316	8.3	159	11.7
B	1436	9.5	152	11.4
C	1361	40	34	11.7

The contents of the tank were continually circulated using a centrifugal pump at a consistent pressure of approximately 40 psi, and purge-fed to the high-pressure feed pump suction line through a remote-controlled flow valve. This allowed for a consistent, controllable feed to the iSCWO system.

2.2 iSCWO TESTING

Although General Atomics has many decades of treating different waste streams, preparatory work was needed since treating large volumes of high concentration AFFF is first-of-a-kind testing. The tests were intended to understand the functionality, operability, and performance of the iSCWO system for PFAS. Four tests were completed during the test week. Each test required the same setup: pressurizing the system, heating up and reaching supercritical conditions, operating at steady state with water and diesel to flush out startup byproducts, initiating feed, collecting samples every 30 minutes for 120 minutes, shutting off the waste stream, continuing to feed clean water and fuel to flush out any feed residue from the feed lines, and finally shutting down by turning off the fuel supply, depressurizing and cleaning up the system. In this study's planning stage and development, the Quality Assurance Project Plan trial runs consisting of three tests (Test A, Test B, and Test C) that utilize different AFFF influent starting concentrations were planned. The tests were intended to incorporate from high dilution rate (~3,000-fold) to a moderate dilution rate (~300-fold), and Test C will be diluted AFFF influent (~30-fold dilution). The exact dilution rate was projected to depend on initial workup testing and ensuring a safe environment. The tests were performed in the sequence A, B, and C, and detailed planning of influent dilution and pretreatment approach, projected run times, amount of waste generated, between-run cleaning procedures, safety, standard in process instrumentation, and other testing data to be provided.

A top image of the feed tank with the foam suppressor is shown in Figure 7. A circulating centrifugal pump kept the mixture homogenous before the liquid AFFF mixture was fed to the high-pressure pump (P-100 in Figure 7.

Testing was carried out the week of June 20 – 24, 2021 at the General Atomics facility in San Diego, CA. Table 3 shows the key conditions for each run. Note that the amount of NaOH added to neutralize the HF was increased throughout the week to accommodate the higher PFAS content. The pH values never went below 7.0 indicating that NaOH added was in excess. Further work can be performed to optimize the caustic addition balance.

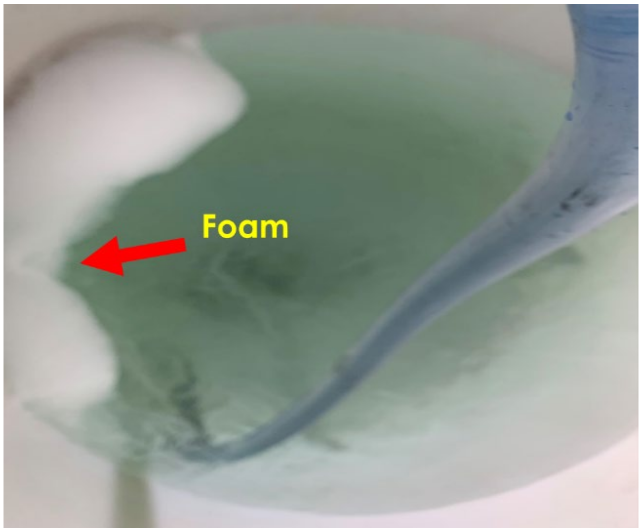

Figure 7: *Top view of diluted AFFF feed tank with foam suppressor and operating circulation pump showing foam formation*

Table 3: EPA AFFF Test Key Conditions Summary

Test	Workup	A	B	C
Date	6/21/21	6/22/21	6/23/21	6/23/21
Time During Steady State AFFF Feed	1544-1731	1443-1650	1200-1404	1621-1834
Reactor temperature, °C	649.8	649.4	645.9	650
Reactor pressure, psia	3220	3249	3191	3249
Dilute AFFF feed flow, lpm (calc)	7.84	7.73	8.50	7.87
Dilute AFFF feed flow, lpm (tank)	6.24	7.97	9.74	8.84
Feed water flow, lpm	0.39	0	0	0
Diesel fuel flow, lpm	1.03	1.02	1.05	0.98
Total feed flow, lpm (gpm)	9.26 (2.45)	8.75 (2.3)	9.54 (2.5)	8.85 (2.3)
Total Organic Carbon Feed, mg/min	736,286	729,850	752,477	704,668
Air oxidant flow, scfm	650 [a]	650 [a]	697	699
Quench water flow, lpm (gpm)	13.6 (3.6)	14.5 (3.8)	13.7 (3.6)	15.8 (4.2)
35 wt% NaOH flow, grams/min	0.51	1.52	1.01	5.05
Liquid effluent (avg), lpm	12.4	12.2	12.5	11.5
% O_2 in gas effluent	7.88	8.44	10.05	11.77

[a] - estimated – estimated; gpm-gallons per minute; lpm – Liters per minute; scfm – standard cubic feet per minute; calc – flow rate determined through pump curves and comparison with flow meters; tank – flow rate based on tank liquid level reduction over time using gross markings.

GA-EMS collected sufficient processing data to enable the team to determine mass flow rates of targeted and non-targeted PFAS in both the liquid and gas effluent. These data were required as part of the quality objectives outlined in the associated project test plan

Figures 8, 9 and 10 show the temperatures, pressures, flows, and gas effluent oxygen for each performance test. The dilute AFFF feed flow was calculated by using the high-pressure feed pump curve that was established using values from an inline water flowmeter just prior to initiating AFFF feed.

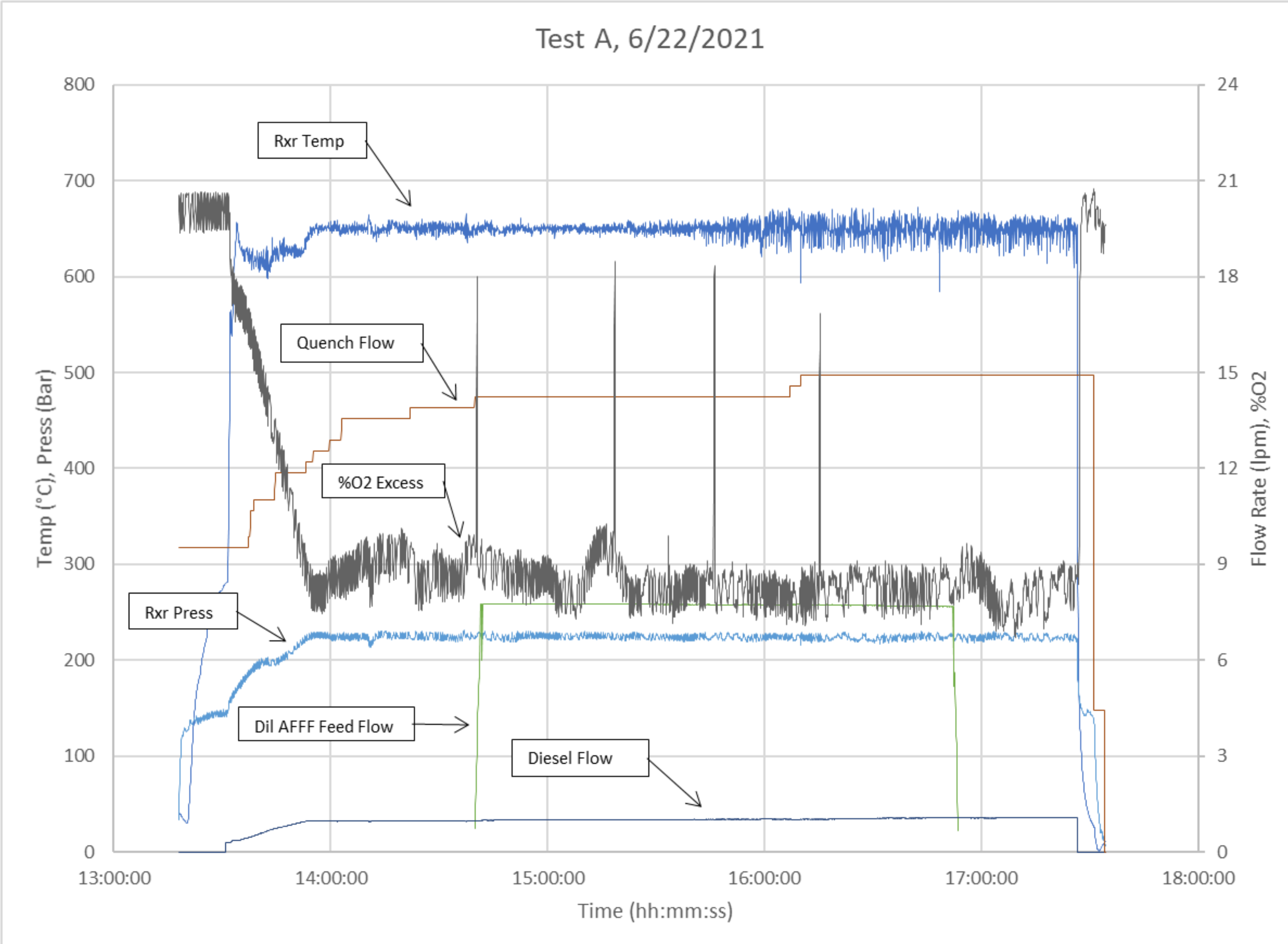

Figure 8: *Test A, 6/22/2021: Temperature, pressure, flow rates and excess O_2 in gas effluent.*

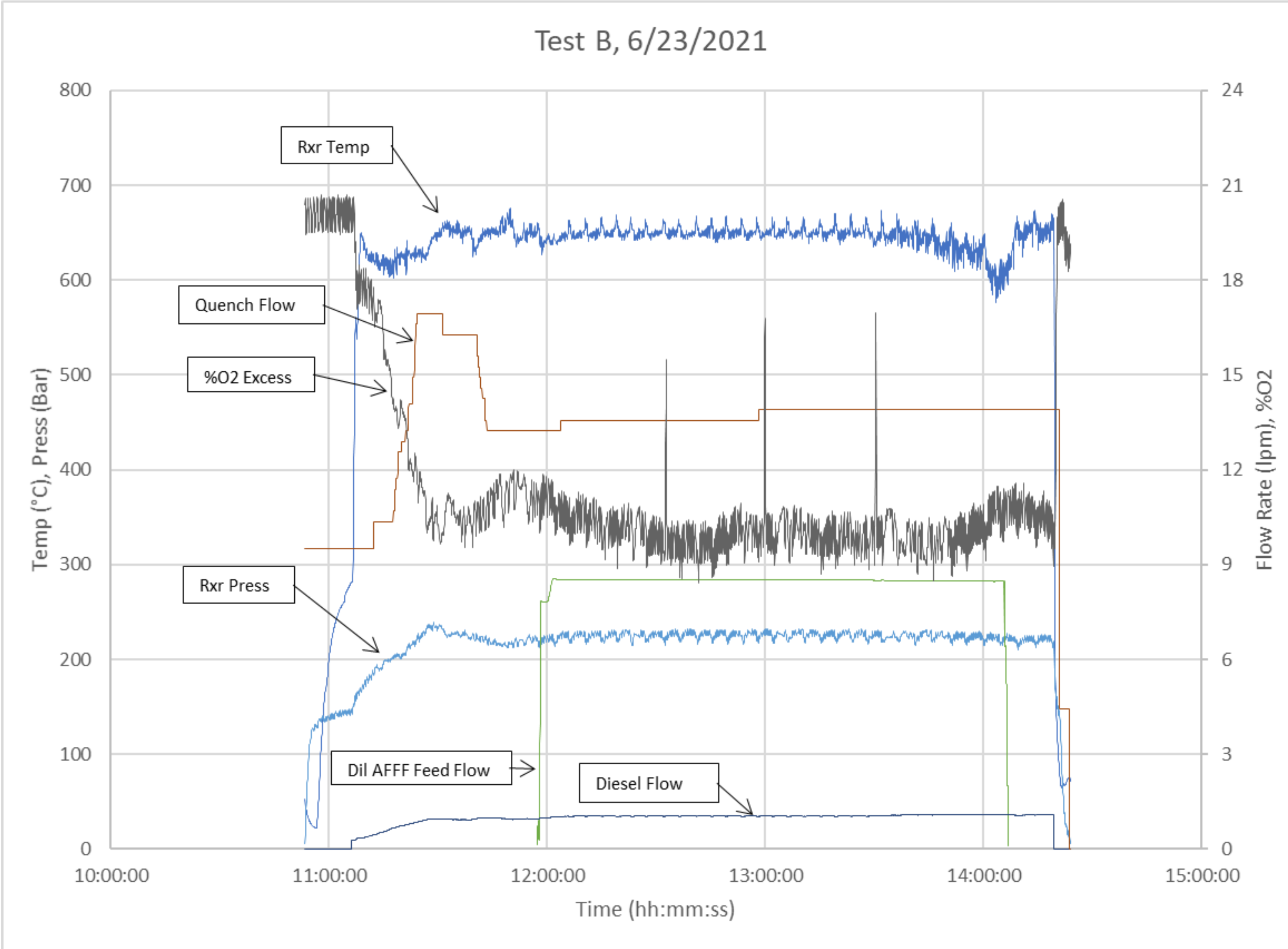

Figure 9: *Test B, 6/23/2021: Temperature, pressure, flow rates and excess O_2 in gas effluent.*

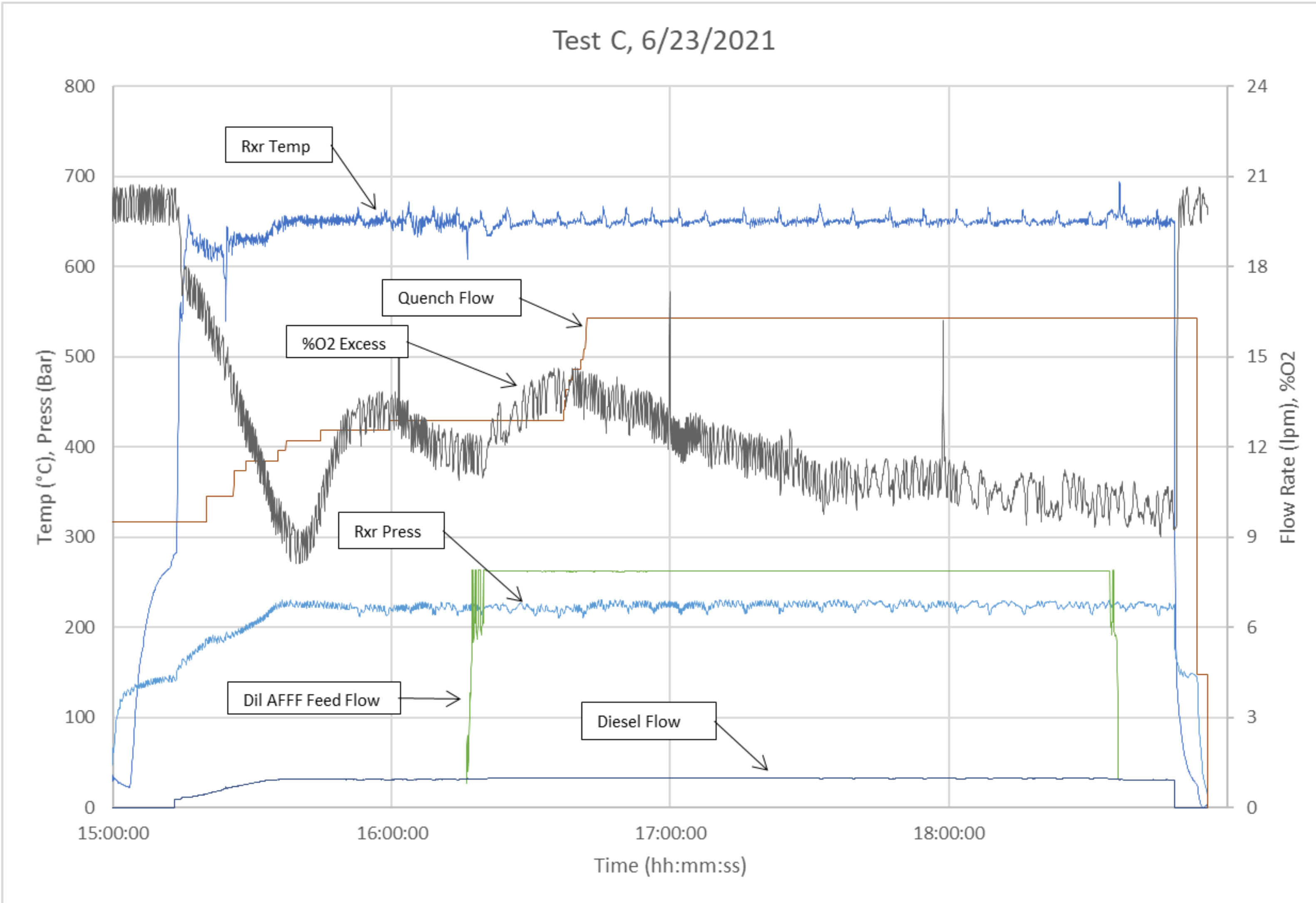

Figure 10: *Test C, 6/23/2021: Temperature, pressure, flow rates and excess O_2 in gas effluent.*

2.3 SAMPLE COLLECTION AND ANALYSIS

Sampling locations were shown previously in Figure 4. Liquid and gas sampling was performed and managed by EPA-ORD. Gas-phase samples collected with canisters and adsorption columns were shipped to EPA/Research Triangle lab, NC, while liquid samples were distributed and analyzed at EPA/Cincinnati, EPA/RTP and an external contract laboratory (Jacobs Laboratory).

2.4 Total Organic Carbon

Total organic carbon samples were analyzed at the EPA Andrew W. Breidenbach Environmental Research Center in Cincinnati, Ohio. The concentration of organic carbon was determined using a carbonaceous analyzer that converts the organic carbon in a sample to CO_2. There are two different approaches for the oxidation of organic carbon in water samples to carbon dioxide gas: (a) combustion in an oxidizing gas and (b) UV promoted or heat catalyzed chemical oxidation with a persulfate solution. Carbon dioxide, which is released from the oxidized sample, is detected by a conductivity detector or by a nondispersive infrared (NDIR) detector. The amount of CO_2 in a sample is directly proportional to the concentration of carbonaceous material in the sample. EPA Method 415.3 was followed for this measurement. The highest calibration standard was 100 ppm, and some of the samples were diluted to bring them to within the calibration range. The MDL for the method is 0.08 ppm. Effluent liquid samples were collected using 250 ml polypropylene bottles.

2.5 Chemical Oxygen Demand (COD)

COD (chemical oxygen demand) is a common method for evaluating organic pollutants. The analyses were performed following Standard Method 5220D, similar to EPA Method 410. COD is an indirect measurement of the amount of organic matter in a sample by determining the amount of dichromate, a chemical oxidant, that is required to oxidize the organic compounds in solution. The chloride ion can cause interference.

2.6 Elemental Analysis (ICP-AES)

In this study, inductively coupled plasma optical emission spectrometry (ICP-OES) was used for the determination of dissolved metal and sulfur ion concentrations before and after the SCWO treatment. U.S. EPA Method 200.7 was selected as the standard method for the analysis of total chromium in either drinking water or wastewater for regulatory compliance and can be measured using ICP-OES (Juliasih et al.). Standards were prepared in 5% (V/V) HNO_3 from a single elemental solution (Sigma Aldrich Co.). Seven standard solutions were prepared to ensure a broader range of experimental designs. This method offers a lower detection limit for the occurrence monitoring for the program. Water samples not used up from the adsorption studies were collected into separate containers and given a few drops of 0.15% double distilled HNO_3 as preservative. A method detection limit of 6 ppb passed the criteria set by the U.S. EPA Method 200.7.

2.7 Total Organic Fluoride (TOF)

An effort was made to evaluate PFAS destruction efficiency based on total organic fluorine (TOF), total fluoride, and total inorganic fluoride (TIF) in both the feed and effluent streams without regard to any specific PFAS compounds (i.e., includes targeted and non-targeted PFAS compounds). TOF methods defluorinate the PFAS, by combustion or chemical reduction, and the fluoride is quantitated using ion chromatography or a fluoride selective electrode. The difference between the inorganic fluoride and the total fluoride in the sample after defluorination is the total organic fluoride. Another method to determine the organic fluorine extracts the organofluorine molecules from the sample and then defluorinates them to produce an extractable organic fluorine measurement. TOF methods have not yet been validated or approved for complex matrices, such as AFFF, and can have some difficulties with complex matrices and high inorganic fluoride concentrations.

2.8 Targeted PFAS Analysis

EPA Method 537.1 was used to for quantitative analysis of targeted PFAS. The method uses a solid phase extraction (SPE) liquid chromatography/tandem mass spectrometry (LC/MS/MS) method for the determination of selected PFAS in drinking water. Water samples (250-ml) are mixed with PFAS surrogates and passed through an SPE cartridge containing polystyrenedivinylbenzene (SDVB) to extract the method analytes and surrogates. The compounds are eluted from the solid phase sorbent with a small amount of methanol. The extract is concentrated to dryness with nitrogen in a heated water bath, and then adjusted to a 1-mL volume with 96:4% (vol/vol) methanol:water and addition of the internal standards. A 10-µL injection is made into an LC equipped with a C18 column that is interfaced to an MS/MS. The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under identical LC/MS/MS conditions. The concentration of each analyte is determined by using the internal standard technique. Surrogate analytes are added to all industrial demostration-scale influent and effluent and QC samples to monitor the extraction efficiency of the method analytes.

2.9 Non-Targeted PFAS Analysis

Effluent aqueous samples collected from the bottom of the gas-liquid separator were extracted and analyzed for targeted and nontargeted PFAS, using ultra-high performance liquid chromatography (UPLC)–tandem mass spectrometry (MS/MS) or Q-TOF. For the identification of unknown compounds based on prediction of chemical formula from accurate ion mass measurement and characteristic isotopic pattern, high performance liquid chromatography coupled with high resolution TOF (or Q-TOF) mass spectrometry at the University of Cincinnati Environmental Analysis Service Center.

The purpose of these tests was to develop a method for examining the process outlet from treating fluorinated foam (AFFF) used for this study ("Aer-O-Water® National Foam 3% concentration) that contains legacy carboxylic and sulfonic surfactants, and fluorotelomer. SCWO could potentially result in a partial decomposition of PFAS resulting in large quantities of non-targeted PFAS.

2.10 Sample Extraction for Non-Targeted PFAS Analysis

A 250 mL water sample is fortified with surrogates, then extracted with weak anion exchange (WAX) solid phase extraction (SPE) cartridges. PFAS are eluted off the cartridge with 1% ammonium hydroxide in 90%:10% MTBE/MeOH solution. The eluate is concentrated by evaporation to a given volume, fortified with internal standard (IS), then adjusted to 250 µL in methanol:10 mM formic acid in Milli-Q water 70:30 (v/v). The sample extracts are analyzed on UPLC MS/MS operated in the negative electrospray ionization mode (ESI-). Target compounds are quantified by internal standard technique using a multipoint calibration curve.

Samples are collected in 250-mL high density polyethylene (HDPE) bottles cleared of any residual PFAS. Field blanks (FB) are collected in the field, by transferring MilliQ water to 250-mL HDPE bottles. In general, 10% of the samples collected are to be processed as sample duplicates and matrix spike samples (LFSM), as determined on a project specific basis. Samples were shipped overnight to the laboratory, at temperatures ≤10 °C. Samples were stored at ≤6 °C until extraction. Samples are extracted within 14 days after collection. To each 250-mL bottle, including all samples, field blanks, laboratory blanks, and laboratory spikes, add 1.5 g of Trizma pre-set crystals (pH 7.0) and mix, then add 0.25 g each of citric acid and sodium citrate and mix. Trizma with added citric acid buffers samples to pH ~4.and removes free chlorine.

2.11 Process Monitoring Sampling

Gas sampling was a critical objective for this test to allow for identification of any potential PFAS, HF or other contaminant emissions within the gas exhaust stream. As part of the tertiary objective, stack emission flow measurements was planned to use a pitot tube (P or S-Type) was offered by EPA to General Atomics to confirm gas stack velocity conducted at the stack. Due to resource limitation, and dry air throughput confirmation using flowmeters, this measurement was deemed not necessary This study used measurement for stack gas velocity was taken in previous tests That data was combined into an overall gas throughput for mass balancing (McDonough et al., 2022)

One method used to confirm the absence of various gases, including HF, was the use of Draeger tubes. Appropriate air volumes were pulled from the exhaust stack through different Draeger tubes to provide immediate analysis of the gas stream for was for HF, then CO, nitrous fumes and SO_2. The purpose was mainly for safety, to give operators a quick glance at gas outputs so process changes can be made, and to check on compliance with criteria pollutant emissions. This allowed the iSCWO operators to make adjustments, if necessary, to ensure a safe environment.

2.12 Sorbent Tube Sampling

Sorbent tube analyses were used with methods outlined in EPA QAPP J-AMCD-0032693-QP-1-0 and SOP J-AMCD-IO-SOP-3981-0. A summary of the method follows. Use Universal Sorbent tubes from Markes International. These sorbent tubes have three sorbents (Carboxen 1003, Tenax, and Carbograph 1, Product number: C3-AAXX-5266). Sorbent tubes should be conditioned within two weeks prior to sampling using a four-step conditioning procedure: 30 min at 100 ºC, 30 min at 200 ºC , 30 min at 300 ºC , and 30 min at 330 ºC at flow rate of around 75 mL/min helium. After conditioning, tubes should be capped with brass Swagelok caps and stored in a cool environment. Prior to field sampling, sorbent tubes need to be blanked on the instrument used for analysis, using the same method. If tubes have low levels of compounds, this is noted so that we do not attribute these to come from the SCWO system and can do a background subtraction during data analysis. EPA standard operating protocols were used following similar published studies (Peng and Batterman 2000, Woolfenden 1997).

Sorbent tubes were intermittently inserted inline with iSCWO air sampling line, which includes a condenser to remove water vapor, and provided a flow rate between 50-100 mL/min through the tube. The flow rates were recorded in the lab notebook. The flow rates were controlled by a small sample pump and checked with a calibrated flow meter. Several different volumes were collected (300 mL, 500 mL, 1 L, 1.5 L etc.). The volumes/sample times were recorded in the lab notebook. Due to unknown breakthrough volumes, two sorbent tubes in tandem were used once at each sampling event to determine whether or not any molecules broke through to the second tube.

2.13 Canister Sampling

All emission samples were collected from the vacuum side of any sampling pump. The emission sample was withdrawn from the stack with a bypass vacuum pump at a nominal sample flow of 0.1 – 1.0 lpm or relies on the canister's vacuum to draw in the sample. The canister sample is withdrawn from the sampling manifold between the impingers and the sample pump. (Figure 6). A critical orifice (~40 cc/min) controls sample flow rate into the evacuated canister. A total of 3 canisters were collected at the sampling location along with field blanks. Nominally, a ~4 L whole air (background) sample was collected over a 2 h sampling period.

EPA-ORD provided the sampling system, canisters, and gas sample collection. ORD's MOP Collection of Whole Air Emissions Samples for Targeted and Nontargeted PFAS Analysis Using SUMMA Canisters was followed. It was understood that method deviations or modifications may be required and were documented and provided as part of the data report. In addition, EPA-ORD staff were on-site during testing to independently observe and document activities. EPA-ORD's canister sampling method was used to collect nonpolar, volatile PFAS compounds for targeted and nontargeted analyses. Samples were collected from a sampling manifold where the process emissions are extracted from the stack with a dedicated sampling system after each sorbent tube extraction was completed. A midget impinger train containing 0.1 N NaOH was used to scrub acid gases for these tests. The sampling system consists of an unheated sample probe (¼" OD SS), an unheated section of ¼" OD SS tubing from the probe to the impingers, and an unheated section of ¼" OD SS tubing from the impinger system to the sampling manifold (also comprised of ¼" OD SS tubing).

3. RESULTS

The performance of the SCWO process was determined based on the mass balance of influent and effluents stream rates, and compositions. All dilutions and internal alkaline feed to neutralize the formation of acids have been considered.

3.1 Composition Analysis of Diluted Feed AFFF

Aer-O-Water® from National Foam tested for this study is an aqueous film forming foal (AFFF) used at 3% concentration to extinguish a fire in hydrocarbon fuels. The feed solution of diluted AFFF solution was analyzed for twenty-four perfluoroalkyl compounds were assessed using a modified EPA Method 537.1, and 15 compounds were detected (Table 3). Two fluorotelomer sulfonates (6:2FTS) and (4:2FTS) made up most of the composition, representing 70.82 % and 24% of the detected PFAS for a sub-total of 94.82% between these two compounds. The two compounds that the EPA has established maximum contaminant level (MCL) advisory levels PFOA and PFOS make up 0.89% of the diluted sample. The six common compounds, PFOS, PFOA, PFNA, PFHxS, PFHpA and PFDA, contribute 1.43%.

Figure 11 depicts a breakdown of the composition of the diluted AFFF sample. The two most abundant compounds quantified through the EPA Method 537.1 for the 1:200 diluted feed were 6:2FTS and 8:2FTS at 2645.4 ng/L and 889.3 ng/L in the 1:200. Previous studies have shown that AFFF samples consisted of other PFAS that are not in the target analysis list and non-PFAS compounds including many fatty acid based surfactants tetradecyle and dodecyle sulfates.

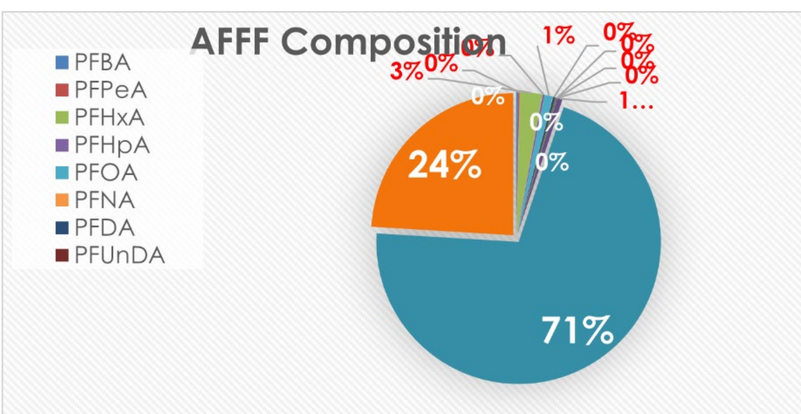

Figure 11. *Composition of 3% aqueous film-forming foam (AFFF) sample. Twenty-four PFAS were quantified from the diluted feed liquid through U.S. EPA Method 537.1. The three fluorotelomers 6:2FTS, 8:2FTS and 4:2FTS contribute 71%, 24% and 3% of the total analyzed PFAS.*

Table 3 *PFAS identified in the AFFF mixure using LC/MS/MS analysis of diluted feed solution to the ISCWO reactor.*

No.	Compound Name	Abbreviation	Formula	Molecular Weight gm/mol	Conce. ng/L	Relative contribution to total PFAS content
1	Perfluorobutanoic acid	PFBA	$C_4HF_7O_2$	214.039	8.803	0.24%
2	Perfluoropentanoic acid	PFPeA	$C_5HF_9O_2$	264.047	5.204	0.14%
3	**Perfluorohexanoic acid**	PFHxA	$C_6HF_{11}O_2$	314.055	93.420	2.50%
4	Perfluorohexanoic acid	PFHpA	$C_7HF_{13}O_2$	364.063	9.443	0.25%
5	Perfluorooctanoic acid	PFOA	$C_8HF_{15}O_2$	414.071	33.305	0.89%
6	Heptadecafluorononanoic acid	PFNA	$C_9PF_{17}C_2$	464.078	1.189	0.03%
7	Perfluorodecanoic acid	PFDA	$C_{10}HF_{19}O_2$	514.086	9.759	0.26%
8	Perfluoroundecanoic acid	PFUnDA	$C_{11}HF_{21}O_2$	564.094	0.366	0.01%
9	Perfluorododecanoic acid	PFDoDA	$C_{12}HF_{23}O_2$	614.102	4.685	0.13%
10	Perfluorotridecanoic acid	PFTrDA	$C_{13}HF_{25}O_2$	664.109	0.203	0.01%
11	Perfluorotetradecanoic acid	PFTeDA	$C_{14}HF_{27}O_2$	714.117	1.656	0.04%
12	Perfluorohexadecanoic acid	PFHxDA	$C_{16}HF_{31}O_2$	814.133	0.716	0.02%
13	Perfluorooctadecanoic acid	PFODA	$C_{18}HF_{35}O_2$	914.149	0.000	0.00%
14	Perfluorobutanesulfonate	PFBS	$C_4HF_9O_3S$	300.1	0.010	0.00%
15	Perfluoropolyether	PFPeS	$C_5HF_{11}SO_3$	372.09	0.000	0.00%
16	Perfluorohexane sulfonic acid	PFHxS	$C_6HF_{13}SO_3$	400.12	0.001	0.00%
17	Perfluoroheptane sulfonic acid	PFHpS	$C_7HF_{15}SO_3$	450.12	0.000	0.00%
18	Perfluorooctane sulfonic acid	PFOS	$C_8HF_{17}O_3S$	500.13	0.000	0.00%
19	Perfluorononanesulfonic acid	PFNS	$C_9HF_{19}O_3S$	550.14	0.000	0.00%
20	Perfluorodecane sulfonic acid	PFDS	$C_{10}HF_{21}O_3S$	600.15	0.000	0.00%
21	Perfluorooctanesulfonamide	PFDoDS	$C_{12}HF_{25}O_3S$	700.16	0.000	0.00%
22	4:2 Fluorotelomer sulfonic acid	4:2FTS	$C_6H_5F_9O_3S$	350.135	22.694	0.61%
23	6:2 Fluorotelomer sulfonic acid	6:2FTS	$C_8H_5F_{13}O_3S$	450.15	2645.458	70.82%
24	8:2 Fluorotelomer sulfonic acid	8:2FTS	$C_{10}H_5F_{17}O_3S$	550.166	898.302	24.05%

3.2 Destruction Efficiency of Organics – TOC analysis results

The concentration of organic carbon in the feed and effluent stream and respective flow rates were used to calculate the DRE of TOC as a measure of the degradation process. It should be noted that the majority of organic carbon entering the iSCWO reactor is coming from diesel fuel. Because the concentration is so high within the feed matrix, only a calculation for the TOC was used rather than sample analysis. In addition, since the AFFF was dilute for these tests, an assumption was made that the AFFF provided no TOC to the feed input matrix. Excluding the TOC from the AFFF would have the effect of decreasing the calculated DRE.

Samples were analyzed following SOP 027.00: Determination of Total Carbon (TC), Inorganic Carbon (IC), and Non-Purgeable Organic Carbon (NPOC) in Water, Wastewater, Aqueous Extracts, and Solids; and Total Nitrogen (TN) in Water, Wastewater, and Aqueous Extracts by a Shimadzu TOC-VCPH, High-temperature TOC Analyzer. The organic carbon compound is oxidized by combustion to carbon dioxide. The carrier gas (transporting the CO_2) is cooled and dehumidified and passed through a halogen scrubber into the cell of the NDIR (Non-Dispersive Infrared) detector where the CO_2 is detected. The NDIR outputs a detection signal which generates a peak. The calculation for DRE is as follows:

$$Destruction\ and\ Removal\ Efficiency, DRE = \frac{W_{in} - W_{out}}{W_{in}}$$

where W_{in}, and W_{out} are mass feed rate of a particular pollutant, such as total organic carbon, total organic fluorine, or any specific fluorinated compound.

Tables 4, 5 and 6, and Figures 12, 13, and 14 show the elimination efficiencies of TOC. For Test A, the TOC levels of the effluent stream ranged from 573 mg/L at 30 min sampling to 90 mg/L at the 120 min sampling time with removal efficiencies improving from 99.4% to 99.99%. After the first 30 minutes of operation, the system was considered steady and excellent removal efficiencies were achieved. The TOC levels in effluent streams of Tests B and C decreased to below 10 mg/L and DRE increased to >99.99%.

It should be noted that immediately after startup, a small amount of TOC residue (unburned diesel fuel) is found within the GLS reservoir until it eventually gets flushed out. This is a normal condition and is only observed during the initial stages of operations. Long-term, steady state samples typically show TOC values ≤5 ppm.

Table 4: Total organic carbon of feed and effluent streams of SCWO treatment Test A

Test	Sample Type	Sample Name	Sampling Time, min	TOC, mg/L	DRE (%)
1	Influent	A-Rep1	0	79,512.50	
	Influent	A-Rep2			
1	Effluent	A-T1_Rep1	30	443.7	99.44%
	Effluent	A-T1_Rep2		307.9	99.61%
	Effluent	A-T1_Rep3		573.4	99.28%
1	Effluent	A-T2_Rep1	60	446.2	99.44%
	Effluent	A-T2_Rep2		411.3	99.48%
	Effluent	A-T2_Rep3		436.3	99.45%
1	Effluent	A-T3_Rep1	90	211	99.73%
	Effluent	A-T3_Rep2		330.4	99.58%
	Effluent	A-T3_Rep3		206.9	99.74%
1	Effluent	A-T4_Rep1	120	90.93	99.89%
	Effluent	A-T4_Rep2		200.5	99.75%
	Effluent	A-T4_Rep3		87.28	99.89%

Table 5: Total organic carbon of feed and effluent streams of SCWO treatment Test B

Test	Sample Type	Sample Name	Sampling Time, min	TOC, mg/L	DRE (%)
2	Influent Influent	LB-01 B-02	0	78,771.17	
2	Effluent Effluent Effluent	B-01-Rep1 B-01-Rep2 B-01-Rep3	0 0 0	121.55 120.75 119.1	99.85% 99.85% 99.85%
2	Effluent Effluent Effluent	B-02-Rep1 B-02-Rep2 B-02-Rep3	30 30 30	11.74 11.375 10.945	99.99% 99.99% 99.99%
2	Effluent Effluent Effluent	B-03-Rep1 B-03-Rep2 B-03-Rep3	60 60 60	10.045 10.505 10.12	99.99% 99.99% 99.99%
2	Effluent Effluent Effluent	B-04-Rep1 B-04-Rep2 B-04-Rep3	90 90 90	9.068 9.024 9.076	99.99% 99.99% 99.99%
2	Effluent Effluent Effluent	B-05-Rep1 B-05-Rep2 B-05-Rep3	120 120 120	8.468 8.66 8.8	99.99% 99.99% 99.99%

Table 6: Total organic carbon of feed and effluent streams of SCWO treatment Test C

Test	Sample Type	Sample Name	Sampling Time, min	TOC, mg/L	DRE (%)
3	Influent	Inf-C-01	0	79623.5	
	Influent	Inf-C-02			
3	Effluent	C_01-Rep1	0	5.15	99.99%
	Effluent	C-01-Rep2		40.1	99.95%
	Effluent	C-01-Rep3		43.475	99.95%
3	Effluent	C-02-Rep1	30	14.116	99.98%
	Effluent	C-02-Rep2		11.876	99.99%
	Effluent	C-02-Rep3		11.428	99.99%
3	Effluent	C-03-rep1	60	9.228	99.99%
	Effluent	C-03-rep2		14.128	99.98%
	Effluent	C-03-rep3		9.44	99.99%
3	Effluent	C-04-rep1	90	4.194	99.99%
	Effluent	C-04-rep2		4.938	99.99%
	Effluent	C-04-rep3		5.002	99.99%
3	Effluent	C-05-Rep1	120	4.194	99.99%
	Effluent	C-05-Rep2		4.938	99.99%
	Effluent	C-05-Rep3		5.002	99.99%

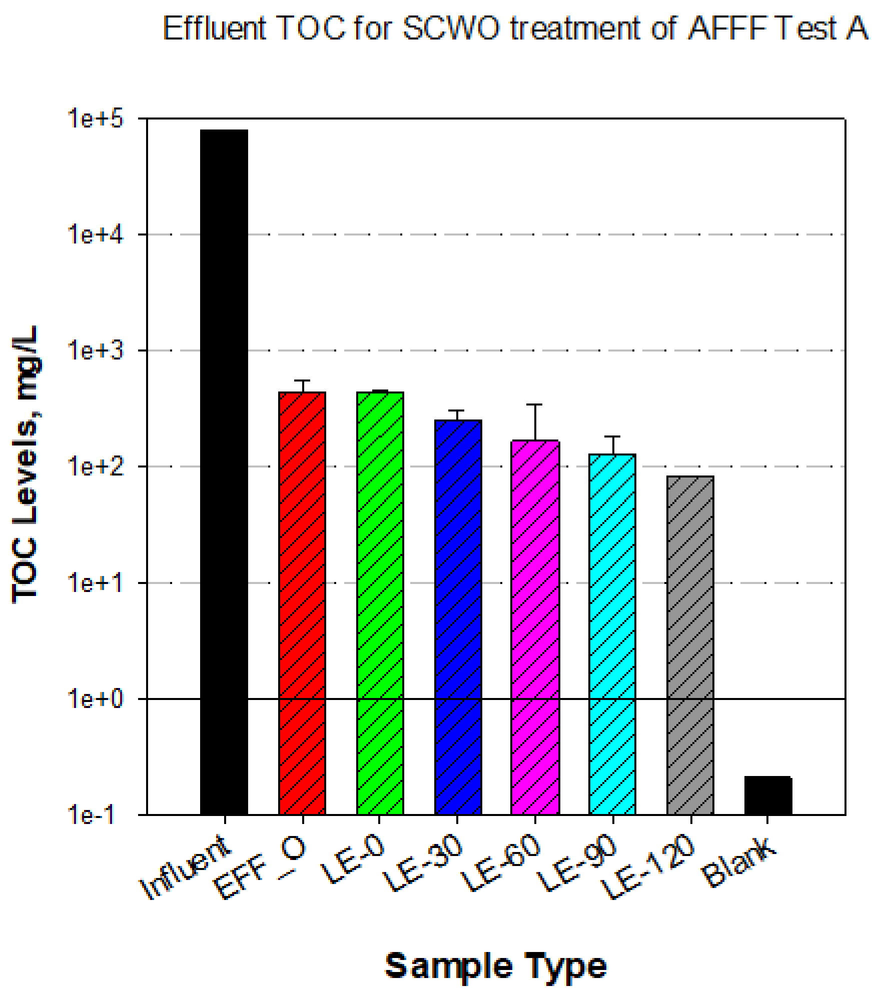

Figure 12: *TOC analysis results of iSCWO reactor Test A of liquid influent and effluent sample collected at run time of 30, 60, 90 and 120 min. following system steady state operation*

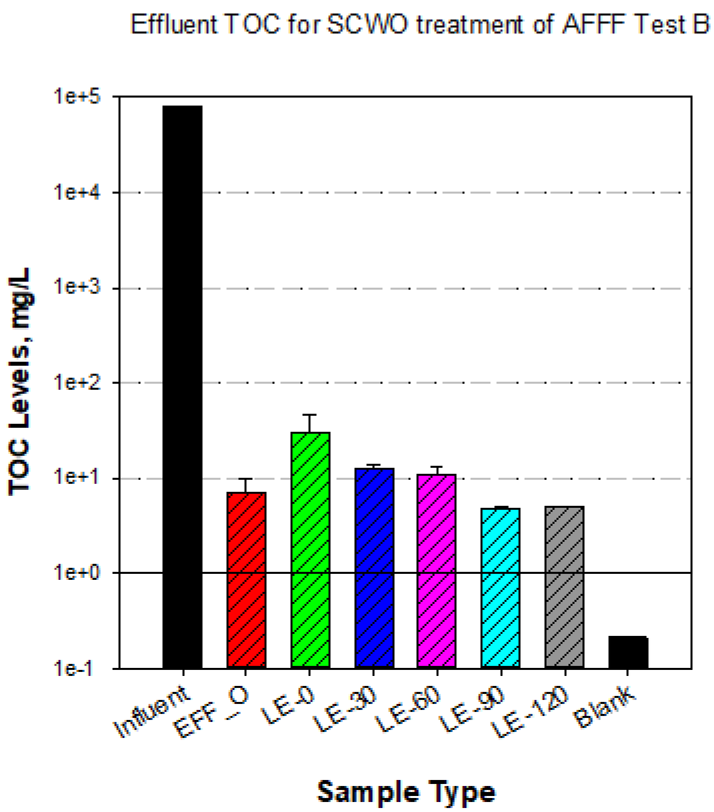

Figure 13 : *TOC analysis results of iSCWO reactor Test B of liquid influent and effluent sample collected at run time of 30, 60, 90 and 120 min. following system steady state operation*

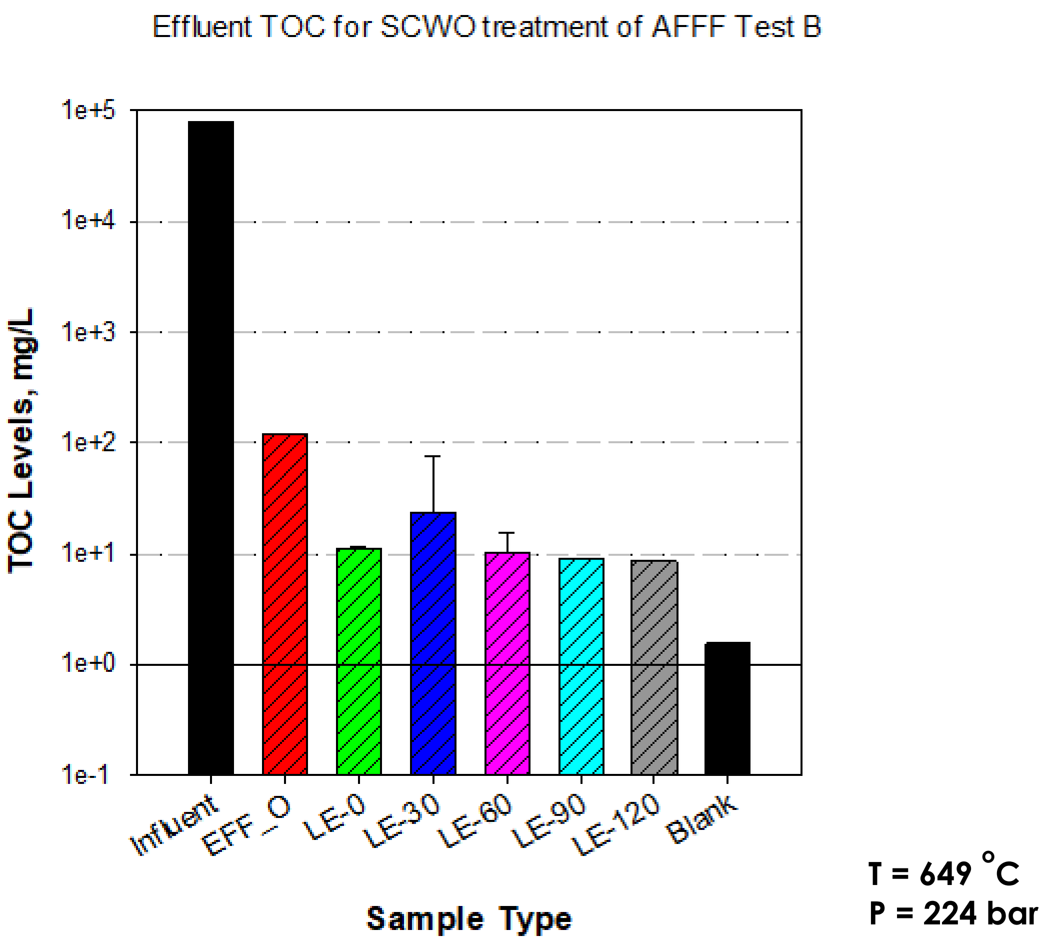

Figure 14: *TOC analysis results of iSCWO reactor Test C of liquid influent and effluent sample collected at run time of 30, 60, 90 and 120 min. following system steady state operation*

3.3 Chemcal Oxygen Demand (COD) Analysis

The COD was determined for samples from the influent and effluent in the gas liquid separator to determine the extent of organic compounds destroyed during the SCWO treatment. COD is not a specific analysis, but can give an indication of the efficacy of a treatment. The results are shown in Table 7. The COD results for samples taken during the AFFF tests were generally below the COD values of the blanks taken before the introduction of the AFFF solution. Only the 30 minute sample during Test A had a COD value higher than the diesel fuel only system blank. For all tests after 90 minutes of runtime, the COD level became consistent and showed over 98% reduction of COD, with up to 99.9% decrease of COD. Since the diesel fuel only blanks had the same to higher levels of COD, it cannot be determined if the remaining COD from the AFFF runs is from the diesel or from the AFFF. It is apparent that the treatment of AFFF with SCWO can reduce the COD levels down to background concentrations.

Table 7: Chemical Oxygen Demand (COD mg/L) for SCWO test samples

Sample	Test A	Test B	Test C
System blank (diesel only)	30	150	250
influent	3800	3800	16000
Separator at 30 min	100	35	100
Separator at 75 min	0	69	24
Separator at 90 min	23	87	20
Separator at 120 min	22	85	20

3.4 Targeted PFAS Destruction Analysis

Up to 24 PFAS compounds were identified and targeted for tracking. Analyses were performed at two separate EPA-ORD laboratories (Research Triangle Park (RTP) and AWBERC) to compare results. Both laboratories used EPA Method 537.1 in accordance with the EPA QA/QC. The individual PFAS concentrations (ng/L) from the AFFF feed in Test A are shown in Figure 15. Note there was no PFOS within the AFFF mixture tested.

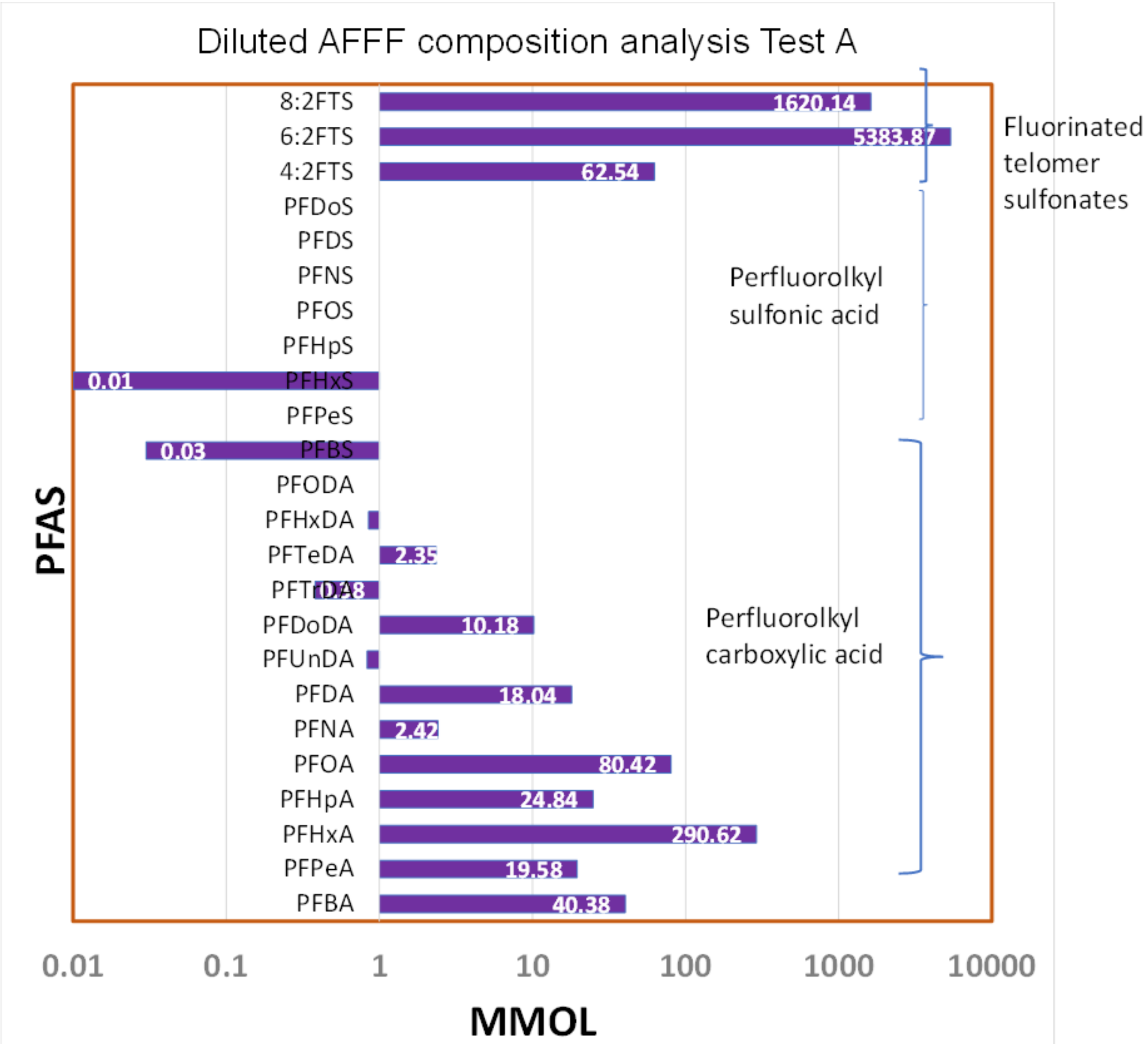

Figure 15: *Feed Sample PFAS Composition for Test A*

After analyses, the PFAS concentrations were combined into a total targeted PFAS concentration for both influent and effluent shown in Figure 16, then averaged across sample time as presented in Table 8. These values were then converted to mass flow rates (µg/min) and used for calculating the %DRE of targeted PFAS. In all tests the destruction efficiency was > 99.99%.

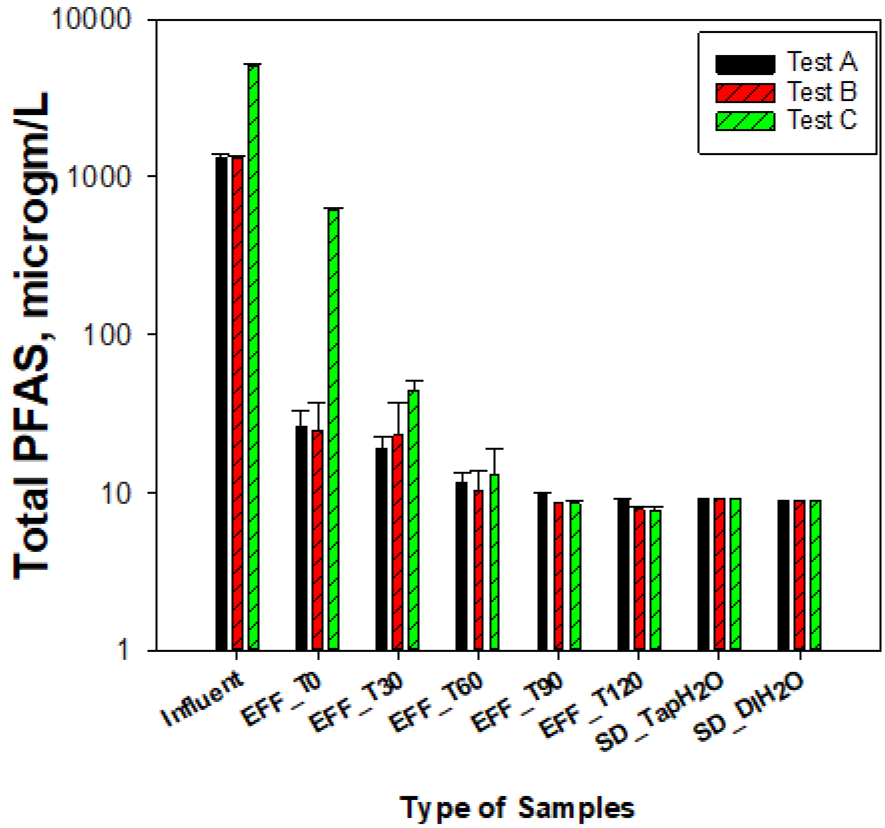

Figure 16: *The total PFAS concentration from Tests A, B, C iSCWO reactor influent and effluent stream from gas-liquid separator at selected times of 30, 60, 90 and 120 min, including San Diego tap water and DI water. EFF = Efflient, T0, T30, T60, T90 = samples collected at time 0, 30, 60, 90 and 120 min., SD-DIH2O = San Diego filtered water.*

Note in Figure 16 that the San Diego tap water (SD_Tap H2O) value is higher than the effluent values from 90 minutes and later. In addition, samples taken at time zero (EFF_T0) show high PFAS values which does not make sense since feed had not even started, suggesting potential contamination of samples during analyses. The iSCWO, primarily set up for processing standard industrial wastes, uses tap water for the quench process which is not exposed to the high temperatures within the reactor.

There appears to be sufficient targeted PFAS compound within the SD tap water that is causing a contamination issue with the reactor outlet fluids preventing an accurate measure of destruction efficiency at the reactor outlet. To resolve this problem, for testing purposes, a deionized or reverse osmosis (RO) water source would be recommended to be used as quench water to allow for a more accurate measure of PFAS destruction. In the field, however, the deionized or RO water source can be used initially until enough liquid effluent can be collected then recycled back to the iSCWO. This will ensure the lowest possible PFAS concentration that could exit the system

Table 8: Targeted PFAS summary with fluoride concentration

Test	Influent Sum of Targeted PFAS (ppt)	Effluent Sum of Targeted PFAS (ppt)	%DE Targeted PFAS	Influent Fluoride⁑ (ppm)	Effluent Inorganic Fluoride⁑ (ppm)	Theoretical fluoride from targeted PFAS* (ppm)	Theoretical Targeted PFAS from Fluoride* (ppm)
A	3,128,300	51.56	99.9984	0.81	173.61	1.86	289.35
B	3,294,600	82.03	99.9975	0.78	235.29	1.96	392.16
C	13,640,000	30.32	99.9998	1.5	482.21	8.07	803.68
		Average DRE:	**99.9985**				

-PFAS remaining - PFBA, 6:2 FTS, and sulfonates (C4-8) - in water or system?
-Effluent values include dilution factor of about 1.5 due to higher flow of liquid in the separator (~12 Lpm out vs ~8 Lpm in)
-Effluent values from 120 minutes sampled

* Assumes 60% fluorine content in associated targeted PFAS
⁑ ~0.8 ppm fluoride typical in SD tap water

Note that the calculated %DRE increased substantially in test C, with higher PFAS concentration in the feed matrix, compared with tests A and B, but is still limited to the PFAS content found in the SD tap water (used for reactor quench). Based on this assessment, there is likely an opportunity to achieve higher destruction efficiencies either through higher AFFF feed applications and/or using treated water (deionized, RO or iSCWO processed liquid effluent) as the reactor quench.

Table 8 also provides information on conversion of organic fluorine to inorganic fluoride. Influent fluoride is very close to the typical level found in the San Diego municipal water. In the effluent, there is up to 300 times more fluoride present resulting from the displacement of the organic fluorine. The theoretical fluoride anticipated from the targeted PFAS is only one-hundredth of the actual fluoride found in the effluent, suggesting that most of the PFAS within the AFFF is not accounted for in the list of targeted compounds. As a check, the theoretical PFAS was calculated based on the effluent total fluoride concentration assuming that fluorine makes up 60% of the molar stoichiometry (estimated from chemical balance). This calculation indicates that other PFAS make up to 100-times more content than the 24 targeted PFAS listed in Figure 16.

3.5 Total Organic Fluorine Analysis

The TOF results for these experiments are shown in Table 9. These results were determined by subtracting the inorganic fluoride from the organic fluoride and show the problems that can occur with TOF experiments with AFFF and complex matrices with high organofluorine and/or inorganic fluoride content. The amount of fluoride in the effluent is many times the calculated TOF of the influent. This shows that the method used to measure TOF was not effective for AFFF. The AFFF was analyzed for both organic fluorine using total oxidizable precursor (TOP) and extractable organic fluorine (EOF) to attempt to detmine how much fluoride should be produced in the effluent with 100% destruction of the PFAS. Both the TOP (415 ppm at 1X concentration) and the EOF (~6,000 ppm at 1X) still did not show near the same concentration as was measured in the effluent of the SCWO system.

Using the values in Table 9, the 1X concentration of organic fluorine in AFFF should be between 16,000 and 35,000 ppm. If about 60% of the AFFF PFAS mass is fluorine (an amount based on the targeted PFAS) then the AFFF would have between 1.6 and 3.5% PFAS content. Table 1 shows that the SDS for the AFFF states that the fluorinated surfactant content is between 1 and 5%, so the 1.6 – 3.5% range is realistic. When using the measured concentrations to determine the TOF concentration of the 1X AFFF, the lower fluoride concentration from Test C (34X dilution) compared with the higher fluoride concentrations produced in the more dilute Test A and B (159X and 152X, respectively) do indicate that more defluorination likely occurs with lower concentrations. Unfortunately, a method to verify the TOF concentration of the AFFF was not found, and the TOF measurements cannot be used to directly determine a destruction efficiency.

Table 9: Total organic fluorine

Test	Influent Free Fluoride (ppm)	Influent TOF (ppm)	Effluent Free Fluoride (ppm)	Effluent TOF (ppm)
A	0.72	19	167	17.8
B	0.7	23.7	234	24.1
C	1.1	96.4	479	45.4

Effluent values include dilution factor of about 1.5 due to higher flow of liquid in the separator (~12 Lpm out vs ~8 Lpm in)
Effluent values from 120-minute samples
San Diego water has about 0.8 ppm fluoride added

3.6 Fluoride Ion Concentration Analysis

Liquid samples from influent and effluent streams collected from the three tests at selected process times were analyzed by ion chromatography (IC) (ThermoScientific) using Thermo Scientific™ Dionex™ IonPac™ AS18-Fast-4µm anion-exchange column. This column provides high capacity, high efficiency, and reasonable resolution of inorganic anions in various sample matrices. This is the hydroxide-selective column of choice for the fastest compliance monitoring of inorganic anions in drinking water and wastewater samples following U.S. EPA Methods 300.0 (A) and 300.1. Dionex AS-AP autosampler supports any Thermo Scientific Dionex ICS ion chromatography system with simultaneous or sequential injections. Automating via the Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS) software is easy. The samples were also analyzed for free fluoride ions in aqueous solutions quickly,, accurately, and economically with the Thermo Scientific™ Orion™ Fluoride Electrodes.

The two analytical methods, IC and F- probe, responded very close for the fluoride concentration up to 100 mg/L, but for higher concentrations, the F- probe was more linear to the calibration concentrations (Figure 22(a) and 22(b)). Effluent samples showed two orders magnitude increase in fluoride concentration compared to influent samples (5.73±1.19 mg/L) by more than two orders of magnitude (Tables 13-15). The concentration of fluoride ions increased with the increase in PFAS concentrations of the feed (Figure 19). These results indicate SCWO effect on high levels of mineralization of organics fluoro compounds.

3.7 VOCs

Air sampling using sorbent tubes (Carboxen 1003, Tenax, and Carbograph 1) with thermal desorption and gas chromatography, is a versatile method for identifying and quantifying trace levels of volatile organic compounds (VOCs). Sorbent tubes were conditioned within two weeks prior to sampling using a four-step conditioning procedure: 30 min at 100 ºC, 30 min at 200 ºC , 30 min at 300 ºC , and 30 min at 330 ºC at a flow rate of around 75 mL/min helium. After conditioning, tubes were capped with brass Swagelok caps and stored in a cool environment. Sorbent tubes were installed in the air sampling line down-stream from the GLS and provided a flow rate between 50-100 mL/min through the tube.

Thermal desorption was used to provide high sensitivity, appropriate choices of sorbents and method parameters to accommodate a wide range of compounds at high humidity. An automated short-path system was used to minimize artifacts, losses and carry-over effects.The samples from the gas emission stream from the GLS are presented in the Table 1. All the compounds detected in this table fit meet the following criteria:

1) Total Score (match to NIST libraries) > 90
2) High resolution filtering score > 90
3) Average peak areas more than 100x greater in AFFF air samples than backgrounds (IPA), field blanks, & ambient air (check flow) samples.

The result is a condensed form of all the detected organic compounds without quantification. The main takeaway result is that although there are lots of trace organic compounds detected there are no fluorinated compounds. The trace organic compounds can be directly related to the diesel fuel used to maintain temperature within the iSCWO reactor. Should avoidance of these compounds be desired, other forms of fuel can be considered such as alcohols, other solvents or natural gas.

3.8 Non-Targeted Analysis of PFAS in Liquid Effluent

Supercritical water oxidation (SCWO) oxidizes organic wastes into gaseous forms, water, salts and minerals (Gloyna and Li 1995). Studies have shown that complete oxidation of specific organics with SCWO goes in tandem with tailored conditions, typically involving elevated operating temperatures, long residence times, high oxidizer-to-waste ratios, or a combination of those (Bermejo and Cocero 2006). However, PFAS have shown to be resistant to other destruction approaches, such as thermal treatments. The common thermal degradation operations may not completely degrade PFAS since fluorine radicals quickly terminate chain branching reaction to as efficient flame retardants, inhibiting the reaction propagation. Such resistance to degradation is likely to form pollutants of incomplete combustion (PICs). PICs may be larger or smaller than the original fluorinated PFAS of concern, where CF_2 radicals are preferred and relatively stable, suggesting the possibility of reforming fluorinated alkyl chains. However, there is limited published information destruction of PFAS and the formation of PICs. Following a degradation process, the remaining C-F fragments may recombine to produce a variety of fluorinated PICs with no analytical method or calibration standards or result in adequate PFAS destruction but unmeasured and unquantified PICs.

The targeted PFAS analysis using LC-MS/MS technology and the implantation of TOC and TOF of influent and effluent streams indicated high removal efficiencies for the SCWO process. However, the EPA Method 537 and ASTM 7979 are designed to monitor a limited and discrete number of PFAS compounds. A non-target screening is necessary for the detection of partially degraded PFAS and other novel compounds that do not have available reference standards (Hollender et al. 2017). Although non-target screening using high-resolution mass spectrometry (HRMS) has shown to be useful for identifying unknowns (Krauss et al. 2010), the technique has proved to be useful for PFAS along with Kendrick Mass Defect analysis (Liu et al. 2015). However, there are few studies focusing on PFAS analysis in water treatment processes (Pan et al. 2016). Therefore, SCWO has to meet several criteria with respect to process conditions to satisfy the oxidation of different organics effectively. Note that some qualitative interpretation is provided regarding NTA, and only some NTA work is comparable.

Eight samples and a blank control for the water used to prepare feed mixtures and effluent samples from Tests A, B, C collected after run times of 90 and 120 min underwent solid phase extraction. In this study, LC/Q-TOF analysis was conducted with an Agilent 6546 LC-Q/TOF system with an automated PFAS annotation and putative identification using Agilent's in-silico fragment predicator, Molecular Structure Correlator (MSC) and exact mass, and fragmentation pattern analyzer for acquiring untargeted data for screening PFAS degradation products. The non-target analysis data allows for retrospective detection of possible products as we learn more about the reaction pathways. Analytical results from the SCWO treated effluent water were intended to fill the gap of the possible partial degraded products in the process discharge stream. None of the longer carbon chain PFAS compounds were detected. The compounds we observed were all in the original targeted list, which include shorter carbon chain trace compounds including $C_6HF_{11}O_2$, Perfluorohexanoic, Perfluoropentanoic acid, $C_5HF_9O_2$, Heptafluorobutyric acid, $C_4HF_7O_2$, $C_6HF_{13}OS$, Perfluorohexanesulfonic acid, C_4HF_9OS, Perfluorobutanesulfonic acid.

An example of successive reaction steps PFAS of hydrothermal decomposition from effluent liquid samples support defluorination reactions is shown in Figure 17. The observed PFAS present at trace levels could produce the observed due to this process or from the presence of the PFAS in the starting material.

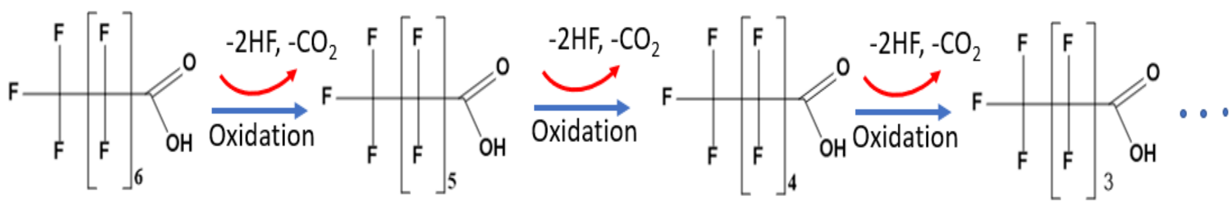

Figure 17: *Proposed oxidative degradation of PFOA in SCWO to successive shorter-chain perfluorocarboxylate and releasing HF with each reaction step. A similar reaction degradation was observed with PFOS in a previous studies (Singh et al, 2019, Cui, Gao and Deng, 2020; Bentel et al, 2019).*

3.9 Corrosion Evaluation Through Liquid Phase Analyses

Along with the evaluation of PFAS destruction efficiency, there was an effort to examine the potential for corrosion by way of analyzing for corrosion products. The iSCWO reactor is comprised of various metals including titanium and Hastelloy that are considered corrosion resistant to most chemicals at SCWO conditions. When PFAS oxidizes to form CO_2 and H_2O, the free fluoride ion will combine with free hydrogen to form HF which can be a very corrosive chemical (e.g., used to etch glass).

Table 10 – 12 and Figure 18 shows the elemental analyses for selected elements that were detected above control and show changes during the process within the influent and effluent matrices. There are several things to note with regard to the different species:

1. The sodium (Na) ion will increase in the liquid effluent as a result of adding NaOH to the quench line in an effort to neutralize HF. The increase in Na is a direct correlation to the concentration of PFAS in the feed line. Optimization can be explored in future work to minimize the NaOH addition while maintaining a neutral pH in the liquid effluent. Note that some feeds containing low PFAS concentrations (such as groundwater) can be self-neutralizing as fluorides will strongly interact and bond with certain cations.

2. Sulfur (S) content will increase in the liquid effluent as various sulfonic acid PFAS compounds are broken down. In this case, S will be in the form of sulfate (SO_4^{2-}) and complex with Na to form Na_2SO_4.
3. Silicon (Si) concentrations show a slight increase from feed to effluent. Although there is some uncertainty as to the cause, one likely suspect is from the addition of the silicone-based antifoam. This is a polymeric chemical that contains silicon. When added to the feed, the polymeric chains may be causing the silicon to go undetected. Once through the reactor, the polymers are destroyed which releases the free silicon.
4. Metals that would be found within the liquid effluent, should corrosion be an issue, would include titanium, chromium, iron, molybdenum and nickel. As shown in Tables 10, 11 and 12, the concentrations of these elements are very low, even decreasing as AFFF concentration increases through the consecutive tests. Results from the metals analyses indicate that there were no corrosion products in the liquid effluent corroborating the goal of using titanium and Hastelloy as reactor materials that are considered corrosion resistant.

Table 10: Selected elemental analysis of feed water and effluent stream samples from Test A. (Sample labels in Appendix II)

SAMPLE	Cr	Fe	Mo	Ni	Na	P	S	Si	Ti
GA-EPA-LI-A	<0.018	<0.037	0.005	<0.003	230.6	0.029	97.200	3.738	<0.008
GA-EPA-LI-B	<0.018	<0.037	0.005	<0.003	227.7	0.030	98.780	3.665	<0.008
GA-EPA-LEB-A	0.190	<0.037	0.158	0.242	946.2	0.080	365.600	18.340	0.009
GA-EPA-LEB-B	0.192	<0.037	0.158	0.255	978.0	0.080	372.400	18.390	0.019
LE-A-01-A	0.254	0.040	0.234	0.114	1848.0	0.508	526.100	23.060	0.180
LE-A-01-B	0.254	<0.037	0.224	0.113	1641.0	0.419	496.000	21.880	0.037
LE-A-01-C	0.257	0.037	0.237	0.117	1788.0	0.521	528.500	22.590	0.267
LE-A-02-A	0.203	<0.037	0.176	0.077	1816.0	0.516	520.200	23.890	0.117
LE-A-02-B	0.184	0.058	0.175	0.093	1740.0	0.521	522.800	22.540	0.336
LE-A-02-C	0.185	<0.037	0.174	0.069	1854.0	0.520	523.100	22.070	0.098
GA-EPA-San Diego DIW	<0.018	<0.037	0.005	<0.003	237.2	0.008	78.670	3.109	<0.008
GA-EPA-San Diego TAPW	<0.018	<0.037	0.004	<0.003	98.4	<0.008	72.810	2.903	<0.008
LE-A-03-A	0.136	0.040	0.169	0.055	1834.0	0.445	479.900	19.920	0.124
LE-A-03-B	0.132	<0.037	0.168	0.054	1814.0	0.461	477.000	19.930	0.112
LE-A-03-C	0.146	0.042	0.169	0.059	1960.0	0.450	475.900	21.890	0.200
GA-EPA-San Diego DIW	<0.018	<0.037	0.004	<0.003	247.4	0.009	73.940	2.983	<0.008
10xGAEPA SanDiegoDIW	<0.018	<0.037	0.004	<0.003	236.4	0.010	74.390	2.859	<0.008
10xGAEPASanDiegoTapW	<0.018	<0.037	0.004	<0.003	102.9	0.008	73.870	2.875	<0.008
LE-A-04-A	0.104	<0.037	0.161	0.046	1653.0	0.378	441.200	17.650	0.116
LE-A-04-B	0.096	<0.037	0.160	0.047	1610.0	0.373	437.800	17.020	0.088
LE-A-04-C	0.100	<0.037	0.157	0.046	1544.0	0.365	427.900	16.640	0.086
LE-A-04-C AnDup	0.100	<0.037	0.161	0.049	1608.0	0.373	442.000	17.300	0.104
07092021 Lab Blank	<0.018	<0.037	<0.004	<0.003	0.4	<0.008	<0.033	<0.114	<0.008
10x070921 BlkSpk	0.244	0.452	0.103	0.272	1.0	0.098	0.268	<0.114	0.101
070921 BlkSpk	2.419	4.520	1.010	2.609	6.2	1.026	2.621	0.953	1.030
070921 BlkSpkAnDup	2.403	4.490	1.010	2.616	6.0	1.030	2.651	0.932	1.027
-	-	-							
D10xGA-EPA-LI-A	<0.018	<0.037	<0.004	<0.003	26.8	<0.008	9.565	0.318	<0.008
D10xGA-EPA-LI-B	<0.018	<0.037	<0.004	<0.003	26.6	<0.008	9.445	0.328	<0.008
D10xGA-EPA-LEB-A	<0.018	<0.037	0.015	0.026	126.7	<0.008	34.910	1.561	<0.008
D10xGA-EPA-LEB-B	<0.018	<0.037	0.016	0.028	126.6	<0.008	35.750	1.563	<0.008
D10xLE-A-01-A	0.028	<0.037	0.023	0.012	313.2	0.020	49.350	1.981	0.014
D10xLE-A-01-B	0.020	<0.037	0.021	0.012	264.5	0.018	46.010	1.855	<0.008
D10xLE-A-01-C	0.028	<0.037	0.024	0.013	310.6	0.021	49.250	1.974	0.022
D10xLE-A-02-A	<0.018	<0.037	0.018	0.010	312.4	0.025	49.690	2.065	0.020
D10xLE-A-02-B	<0.018	<0.037	0.017	0.009	302.3	0.023	48.960	1.999	0.029

SAMPLE	Cr	Fe	Mo	Ni	Na	P	S	Si	Ti
D10xLE-A-02-C	<0.018	<0.037	0.017	0.008	300.7	0.022	49.090	1.949	<0.008
10xGAEPASanDiegoDIW	<0.018	<0.037	<0.004	<0.003	26.1	<0.008	7.072	0.279	<0.008
10xGAEPASanDiegoTapW	<0.018	<0.037	<0.004	<0.003	10.750	<0.008	6.660	0.267	<0.008
D10xLE-A-03-A	<0.018	<0.037	0.016	0.006	261.100	0.021	46.260	1.805	0.014
D10xLE-A-03-B	<0.018	<0.037	0.016	0.006	264.800	0.021	47.110	1.808	0.008
D10xLE-A-03-C	<0.018	<0.037	0.016	0.006	256.800	0.017	45.760	1.756	0.011
D10xLE-A-04-A	<0.018	<0.037	-	<0.003	-	-	-	-	<0.008
D10xLE-A-04-B	<0.018	<0.037	<0.008	<0.003	<0.096	<0.005	-	<0.404	<0.008
D10xLE-A-04-C	<0.018	<0.037	-	<0.003	<0.404	<0.001	<0.005	<0.134	<0.008
-	-	-	-	-	-	-	-	-	-
	Cr	**Fe**	**Mo**	**Ni**	**Ca**	**Co**	**Cu**	**Mg**	**Ti**
Established 2016MRL	**0.018**	**0.037**	**0.004**	**0.003**	**0.404**	**0.001**	**0.005**	**0.134**	**0.008**
Lowest Cal Std	**1.0**	**1.0**	**1.0**	**1.0**	**5.0**	**1.0**	**1.0**	**5.0**	**0.5**
Highest Cal Std	**20**	**200**	**20**	**20**	**2000**	**20**	**20**	**2000**	**10**
Correlation**	**0.999**	**0.999**	**0.999**	**0.999**	**0.999**	**0.999**	**0.999**	**0.999**	**0.999**
Re-slope	-	-	-	-	-	-	-	-	-

Table 11: Selected elemental analysis of feed water and effluent stream samples from Test B

SAMPLE	Units	Cr	Fe	Mo	Na	Ni	S	Si	Ti
08042021-Lab Blank	mg/L	<0.018	<0.037	<0.004	<0.191	<0.003	<0.033	<0.114	<0.008
10x0842021_BlkSpk	mg/L	0.272	0.536	0.108	0.415	0.283	0.253	<0.114	0.107
08042021-Blk Spike	mg/L	2.667	5.350	1.060	5.320	2.739	2.752	1.086	1.069
LI-B-01-A	mg/L	<0.018	<0.037	0.005	238.500	<0.003	102.600	3.659	<0.008
LI-B-01-B	mg/L	<0.018	<0.037	0.004	242.200	<0.003	103.100	3.514	<0.008
LE-B-01-A	mg/L	0.204	0.101	0.222	980.400	0.473	369.900	16.540	1.130
LE-B-01-B	mg/L	0.197	0.079	0.219	950.400	0.567	365.200	15.880	0.768
LE-B-01-C	mg/L	0.198	0.061	0.219	999.900	0.436	368.600	16.500	0.584
LE-B-02-A	mg/L	0.345	<0.037	0.168	1949.000	0.150	455.200	18.940	0.075
LE-B-02-B	mg/L	0.337	<0.037	0.166	2012.000	0.097	450.800	18.790	0.033
LE-B-02-C	mg/L	0.336	<0.037	0.169	2008.000	0.104	453.900	18.960	0.060
LE-B-03-A	mg/L	0.313	<0.037	0.266	2272.000	0.134	618.800	26.120	0.025
LE-B-03-B	mg/L	0.312	<0.037	0.270	2276.000	0.195	629.600	26.070	0.028
LE-B-03-C	mg/L	0.299	<0.037	0.269	2137.000	0.118	621.400	25.280	0.050
LE-B-04-A	mg/L	0.230	<0.037	0.264	2095.000	0.063	625.400	27.100	0.035
LE-B-04-B	mg/L	0.213	<0.037	0.264	2043.000	0.060	627.700	25.290	0.023
LE-B-04-C	mg/L	0.234	<0.037	0.264	2083.000	0.070	632.100	27.060	0.057
LE-B-05-A	mg/L	0.178	<0.037	0.255	2023.000	0.104	625.800	26.860	0.051
LE-B-05-B	mg/L	0.168	<0.037	0.251	1975.000	0.079	610.600	25.260	0.117
LE-B-05-C	mg/L	0.164	<0.037	0.245	1950.000	0.076	593.600	25.360	0.138
-	-	-	-	-	-	-	-	-	-
	-	**Cr**	**Fe**	**Mo**	**Na**	**Ni**	**S**	**Si**	**Ti**
Established 2016MRL	-	**0.018**	**0.037**	**0.004**	**0.191**	**0.003**	**0.033**	**0.114**	**0.008**
Lowest Cal Std	mg/L	**1.0**	**1.0**	**1.0**	**5.0**	**1.0**	**5.0**	**2.0**	**0.5**
Highest Cal Std	mg/L	**20**	**200**	**20**	**100**	**20**	**200**	**100**	**10**
Correlation**	-	**0.999**	**0.999**	**0.999**	**0.999**	**0.999**	**0.999**	**0.999**	**0.999**
Re-slope	-	-	-	-	-	-	-	-	-

Table 12: Selected elemental analysis of feed water and effluent stream samples from Test C

SAMPLE	Units	Cr	Fe	Mo	Na	Ni	S	Si	Ti
LI-C-01-A	mg/L	<0.018	<0.037	<0.004	29.050	<0.003	17.020	0.362	<0.008
LI-C-01-B	mg/L	2.453	4.960	<0.004	28.980	2.601	16.810	0.352	1.002
LE-C-01-A	mg/L	2.440	4.920	<0.004	28.180	2.598	16.520	0.340	0.994
LE-C-01-B	mg/L	<0.018	<0.037	<0.004	28.950	<0.003	16.770	0.345	<0.008
LE-C-01-C	mg/L	<0.018	<0.037	<0.004	28.600	<0.003	16.690	0.360	<0.008
LE-C-02-A	mg/L	<0.018	<0.037	0.031	275.000	<0.003	55.030	2.292	<0.008
LE-C-02-B	mg/L	<0.018	<0.037	0.031	258.900	<0.003	54.990	2.186	<0.008
LE-C-02-C	mg/L	<0.018	<0.037	0.031	263.800	<0.003	53.930	2.182	<0.008
LE-C-03-A	mg/L	0.199	<0.037	0.022	209.900	0.063	41.990	1.731	0.012
LE-C-03-B	mg/L	0.195	<0.037	0.022	208.600	0.067	41.940	1.731	0.014
LE-C-03-C	mg/L	0.196	<0.037	0.022	207.100	0.072	42.390	1.722	0.009
LE-C-04-A	mg/L	0.120	<0.037	0.022	203.800	0.039	39.360	1.627	<0.008
LE-C-04-B	mg/L	0.122	<0.037	0.023	205.900	0.037	40.280	1.627	<0.008
LE-C-04-C	mg/L	0.117	<0.037	0.021	191.500	0.029	38.280	1.521	<0.008
LE-C-05-A	mg/L	0.088	<0.037	0.018	196.900	0.139	39.040	1.523	0.015
LE-C-05-B	mg/L	0.087	<0.037	0.017	187.900	0.086	36.990	1.477	0.026
LE-C-05-C	mg/L	0.082	<0.037	0.018	198.500	0.078	38.700	1.556	0.013
08132021 Lab Blank	mg/L	0.067	<0.037	<0.004	<0.191	0.044	<0.033	<0.114	0.013
Dil10x 081321 BlkSpk	mg/L	0.071	<0.037	0.099	0.473	0.041	0.262	<0.114	0.025
081321 BlkSpk	mg/L	0.068	<0.037	0.988	4.939	0.040	2.644	1.014	0.017
081321 BlkSpk AnD	mg/L	-	-	0.991	4.939	-	2.646	1.009	-
-	-	Cr	Fe	-	-	Ni	-	-	Ti
	-	**0.018**	**0.037**	Mo	Na	**0.003**	S	Si	**0.008**
Established 2016MRL	-	**1.0**	**1.0**	**0.004**	**0.191**	**1.0**	**0.033**	**0.114**	**0.5**
Lowest Cal Std	mg/L	**20**	**200**	**1.0**	**5.0**	**20**	**5.0**	**2.0**	**10**
Highest Cal Std	mg/L	**0.999**	**0.999**	**20**	**100**	**0.999**	**200**	**100**	**0.999**
Correlation**	-	**1.017**	**1.019**	**0.999**	**0.999**	**1.064**	**0.999**	**0.999**	**0.918**
Re-slope	-	-	-	-	-	-	-	-	-

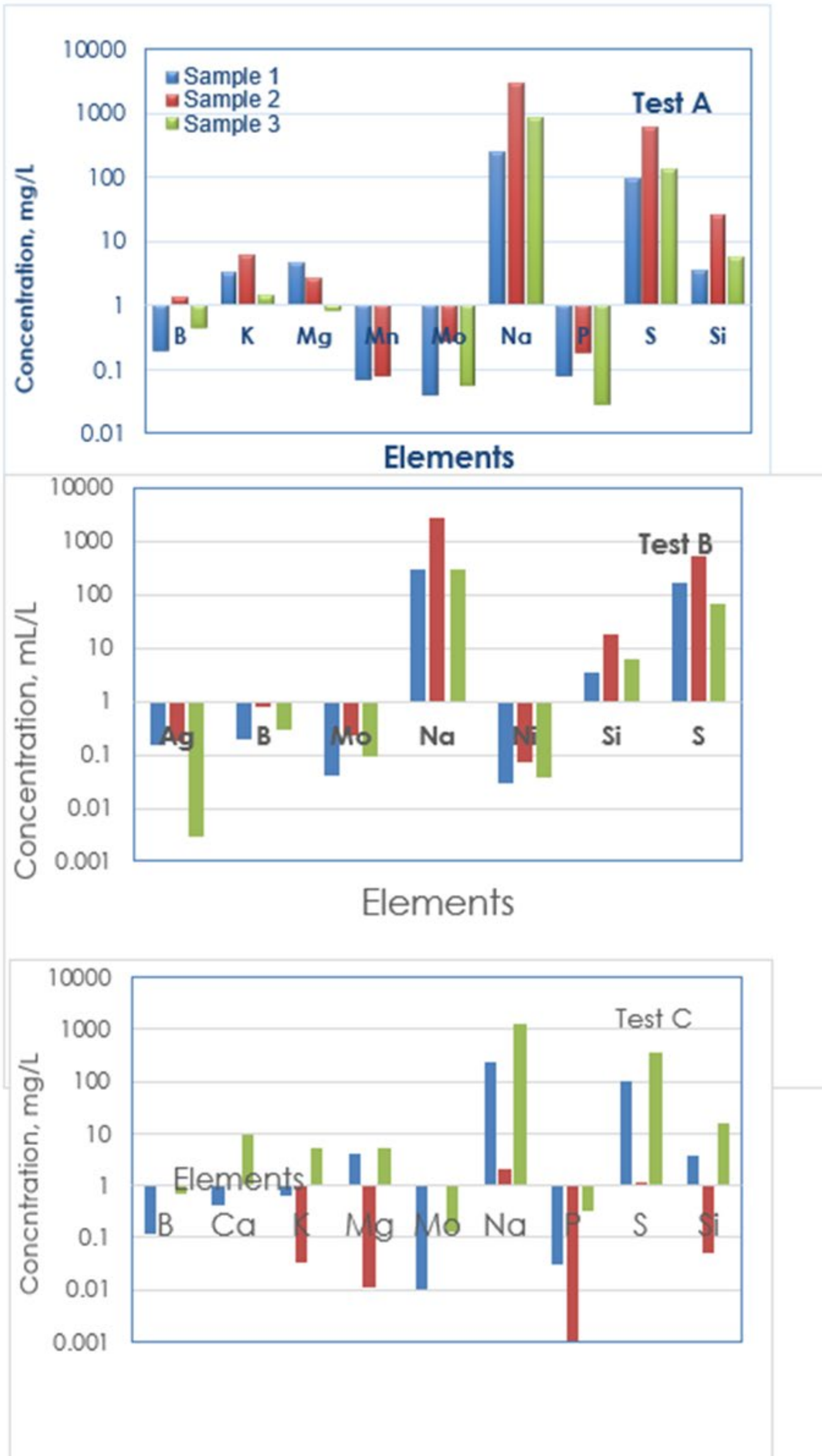

Figure 18: *Elemental Composition of influent and effluent stream Samples for Test run A, B, and C The color bars represent duplicate*

Table 13 Flouride ion concentration measured using ion Chromatography and F- prob approaches for influent and efflent sample from Test A

S/N	SAMPLE NAME	F^- using IC (ppm)	S^{2-} using IC (ppm)	F^- using ISE (ppm)
1	GA-EPA-LI-A	0.94	0.00	2.26
2	GA-EPA-LI-B	0.93	0.00	2.11
3	LEB-A-01	5.11	0.00	10.50
4	LEB-A-02	4.84	0.00	10.50
5	LE-A-01-A	24.93	0.00	77.20
6	LE-A-01-B	18.96	0.00	51.30
7	LE-A-01-C	32.92	0.00	76.80
8	LE-A-02-A	27.69	0.00	97.10
9	LE-A-02-B	31.84		98.40
10	LE-A-02-C	34.17	0.00	98.70
11	LE-A-03-A	34.91	0.00	96.40
12	LE-A-03-B	35.43	0.00	97.30
13	LE-A-03-C	32.62	0.00	97.10
14	LE-A-04-A	33.51	0.00	91.90
15	LE-A-04-B	36.61	0.00	92.30
16	LE-A-04-C	37.96	0.00	92.30

Table 14 Flouride ion concentration measured using ion Chromatography and F- prob approaches for influent and efflent sample from Test B

S/N	SAMPLE NAME	F^- using IC (ppm)	S^{2-} using IC (ppm)	F^- using ISE (ppm)
17	AFFF	8.83	0.00	25.60
18	LI-B-01-A	1.53	0.00	2.09
19	LI-B-01-B	1.57	0.00	2.12
20	LEB-B-01-A	7.65	0.00	14.00
21	LEB-B-01-B	5.65	0.00	14.10
22	LEB-B-01-C	8.90	0.00	14.00
23	LE-B-02-A	21.06	0.00	52.50
24	LE-B-02-B	19.17	0.00	52.10
25	LE-B-02-C	21.09	0.00	53.50
26	LE-B-03-A	32.58	0.00	114.00
27	LE-B-03-B	43.71	0.00	116.00

28	LE-B-03-C	41.52	0.00	117.00
29	LE-B-04-A	40.75	0.00	135.00
30	LE-B-04-B	42.73	0.00	134.00
31	LE-B-04-C	40.99	0.00	134.00
32	LE-B-05-A	43.13	0.00	139.00
33	LE-B-05-B	36.68	0.00	138.00
34	LE-B-05-C	44.59	0.00	139.00

Table 15 Flouride ion concentration measured using ion Chromatography and F- prob approaches for influent and efflent sample from Test C

S/N	SAMPLE NAME	F^- using IC (ppm)	S^{2-} using IC (ppm)	F^- using ISE (ppm)
35	LI-C-01-A	3.19	0.00	2.88
36	LI-C-01-B	3.29	0.00	2.85
37	LI-C-01-A*	3.07	0.00	2.85
38	LI-C-01-B*	3.45	0.00	2.85
39	LI-C-01-C*	3.74	0.00	2.85
40	LEB-C-01-A	19.55	0.00	30.30
41	LEB-C-01-B	22.36	0.00	33.40
42	LEB-C-01-C	26.22	0.00	33.80
43	LE-C-02-A	56.90	0.00	303.00
44	LE-C-02-B	57.74	0.00	303.00
45	LE-C-02-C	59.04	0.00	305.00
46	LE-C-03-A	66.59	0.00	300.00
47	LE-C-03-B	54.53	0.00	299.00
48	LE-C-03-C	87.48	0.00	299.00
49	LE-C-04-A	84.06	0.00	296.00
50	LE-C-04-B	73.42	0.00	295.00
51	LE-C-04-C	71.07	0.00	296.00
52	LE-C-05-A	95.99	0.00	292.00
53	LE-C-05-B	70.57	0.00	294.00
54	LE-C-05-C	68.80	0.00	295.00

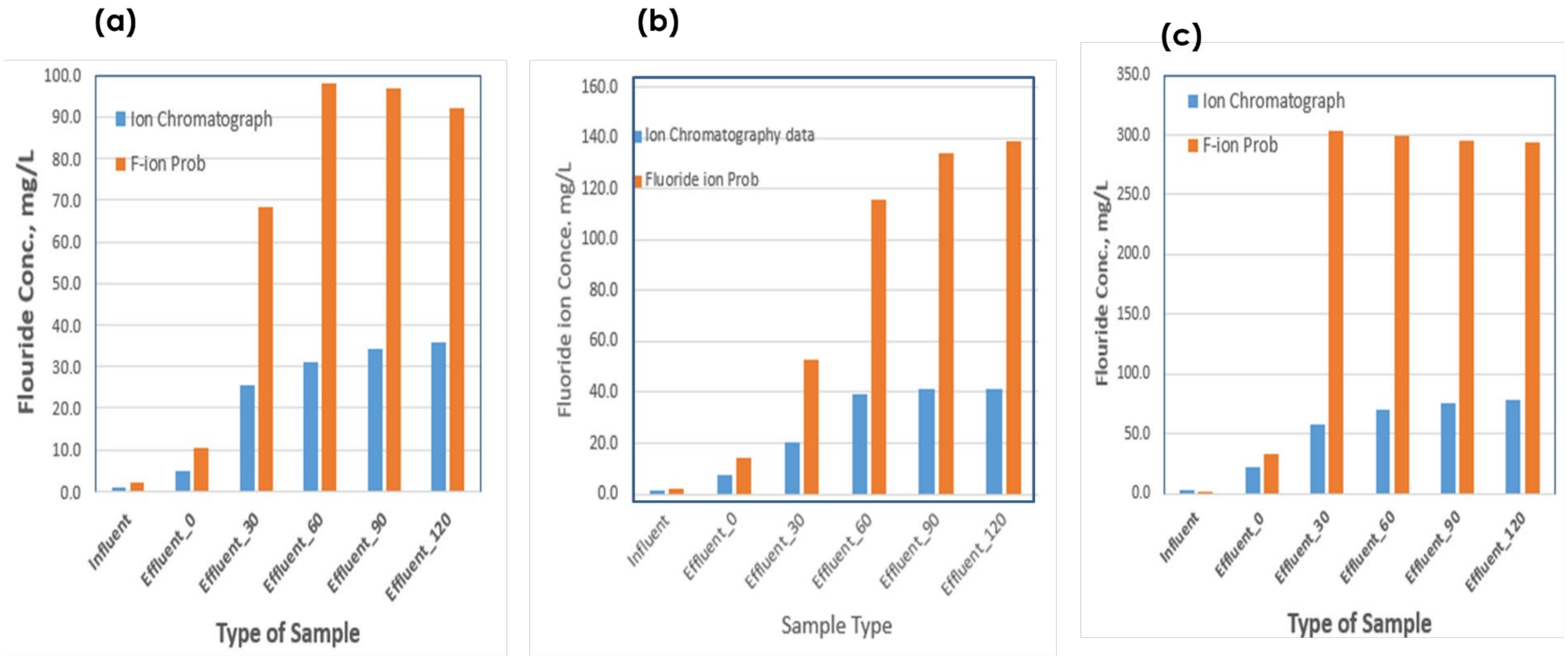

Figure 19: *Fluoride ion concentration measured with IC and fluoride ion probe for feed and effluent samples collected at times 0, 30, 60, 90, 120 min (a) Test A, (b) Test B, and (c) Test C.*

3.10 Gas Analyses from Evacuated Canisters

Eighteen 6-liter Silonite canisters were received on July 1st, 2021 from the General Atomics Facility in San Diego, California. The canisters contained gas phase samples collected from the emissions of three performance tests conducted between June 22-23, 2021, including background samples that were taken from the surrounding air away from the test area. Samples were logged in, pressurized with zero grade air to 30 psia, and analyzed between July 17th-July 21st, 2021. A Markes Unity concentration unit equipped with a green-house gas trap affixed to a 7890 Agilent GC and a Markes time of flight detector was used to concentrate gas samples from each 6-liter canister and quantified select PFAS amenable to gas phase collection and TO-15 type analysis.

Samples were analyzed by concentrating 15 and 200-ml sample gas across a Peltier cooled Green House gas (GHG) trap (-30°C) that traps the PFAS gases prior to desorption onto a GC

time of flight system for separation and detection. Fifteen ml of sample were taken from each Silonite canister to stay well below the tetrafluoromethane breakthrough maximum volume of 25 ml. All reported tetrafluoromethane values used the 15 ml sample pull volume for quantification. The remaining target analytes used the 200 ml sample pull volume for quantification. Dilution (pressurized to 30 psia) and volume normalizations (only 15 ml were taken for tetrafluoromethane) were applied to the data to adjust the reported part per billion (ppbv) target concentrations to the correct levels (Tables 13, 14, 15).

All reported samples were quantified using a multi-level 30-component PFAS calibration performed in mid-July. Daily calibration checks and blanks were run along with the samples each day and were reported along with the data. The calibration range for tetrafluoromethane ranged between 50 and 200 ppbv (estimated MDL of 3 ppbv). The other PFAS targets were calibrated at a range typically between 0.5 and 20 ppbv (except hexafluoroethane that ranged between 2.5 and 50 ppbv). Most of the 30-component calibration PFAS mix targets have correlation coefficients greater than 0.995 over each calibration range. Nearly all the reported 30 PFAS targets daily calibration check standards performed throughout (4 were done) this study were within method calibration recovery limits of 70-130% except for trichlorofluoromethane that was biased slightly low.

Method detection limits (MDL) were not reported with this data set because this method is still being developed and can only be estimated at this time. All quantification target ion values having greater than a 3/1 signal/noise ratio threshold were reported as positive target hits if retention time and fragmentation criterion was met. Since detection limits for this method have not been formally performed, they were not applied to this data set to invalidate positively identified targets that met the 3:1 signal/noise ratio and met the qualifier fragmentation ratio/retention time criterion. All electronic data were double checked and corrected to make sure that integrations were performed appropriately. Because the GC was equipped with electronically programmable control (EPC), retention times did not shift much through the analysis period. This was critical in the positive identification of target analyte components.

Sample concentrations were reported in parts per billion (ppbv) in an attached spreadsheet. PFAS target compounds were not detected above background level in this data set. Composited target PFAS results can be found in Appendix I.

Target View deconvolution library searches were performed using the 200 ml sample pull volumes for each test canister. The data were reported in a separate attached Excel file due to the number of samples (samples separated by tab). Target View displays all detected qualitative compound match probability information with associated abundances generated from the Target View software.

Under the test procedure, no specific compounds were found to exceed the background levels indicating that the air emissions from the iSCWO system would be considered clean shown in Tables 13, 14, and 15, corresponding to Tests A, B, and C.

3.11 Thermal Desorption Tube Data

The thermal desorption tubes did not show any molecules that were likely fluorinated. There were other organic molecules that were tentatively identified by the GC/MS software and NIST database. The possible organic molecules are listed in Appendix I, Table 18.

Table 16: GA-EPA SCWO test Gas Emission Canister Analysis Test A

Canister ID	Batch Blank Canister 37187	Background Canister 37156	**GA Test A Sample 1** Canister 36864	**GA Test A Sample 2** Canister 37198	**GA Test A Sample 3** Canister 37147
Compound Name	GA06072121-LB10 ppbv	GA-062221-AFFF Bkgd ppbv	GA 062221-AFFF Sample 1 ppbv	GA-062221-AFFF Sample 2 ppbv	GA-062221-AFFF Sample 3 ppbv
tetraflouromethane	ND	ND	ND	ND	ND
hexafluoroethane	ND	ND	ND	ND	ND
chlorotrifluoromethane	ND	ND	ND	ND	ND
fluoroform	ND	ND	ND	ND	ND
octafluoropropane	ND	ND	ND	ND	ND
difluoromethane	ND	ND	ND	ND	ND
pentafluoroethane	ND	0.10	0.09	0.09	0.16
octafluorocyclobutane	ND	ND	ND	ND	ND
fluoromethane	ND	ND	ND	ND	ND
tetraflouroethylene	ND	ND	ND	ND	ND
hexafluoropropylene	ND	ND	ND	ND	ND
1,1,1-trifluoroethane	ND	ND	ND	ND	ND
hexafluoropropene oxide	ND	ND	ND	ND	ND
chlorodifluoromethane	ND	0.10	0.05	0.05	0.05
1,1,1,2-tetrafluoroethane	ND	ND	0.09	ND	ND
perfluorobutane	ND	0.05	0.05	0.05	0.05
1H heptafluoropropane	ND	0.05	ND	0.05	0.11
octafluourocyclopentene	ND	ND	ND	ND	ND
trichlorofluoromethane	ND	0.39	0.38	0.33	0.33
dodecafluoro-n-pentane	ND	ND	ND	ND	ND
1H nonafluorobutane	0.01	0.05	0.05	0.09	0.11
tetradecafluorohexane	ND	ND	ND	ND	ND
1H perflluoropentane	ND	ND	ND	ND	ND
heptafluoropropyl 1,2,2,2-tetrafluoroethyl ether (E1)	ND	ND	ND	ND	ND
hexadecaflluroheptane	ND	ND	ND	ND	ND
1H perfluorohexane	ND	ND	ND	ND	ND
perfluorooctane	ND	ND	ND	ND	ND
1H perfluoroheptane	ND	ND	ND	ND	ND
1H Perfluorooctane	ND	ND	ND	ND	ND
2H-Pefluoro-5-methyl-3,6-dioxanonane (E2)	ND	ND	ND	ND	ND

Table 17: GA-EPA SCWO summary of gas emission canister sampling analysis three tests summary. Test B

Can ID	Background Canister 37188	GA Test B Sample 1 Canister 36850	GA Test B Sample 2 Canister 37157	GA Test B Sample 3 Canister 36840
Compound Name	GA-062321-AFFF Background ppbv	GA-062321-AFFF Sample 1 ppbv	GA-062321-AFFF Sample 2 ppbv	GA-062321-AFFF Sample 3 ppbv
tetraflouromethane	ND	ND	ND	ND
hexafluoroethane	ND	ND	ND	ND
chlorotrifluoromethane	ND	ND	ND	ND
fluoroform	ND	ND	ND	ND
octafluoropropane	ND	ND	ND	ND
difluoromethane	ND	ND	ND	ND
pentafluoroethane	ND	ND	ND	ND
octafluorocyclobutane	ND	ND	ND	ND
fluoromethane	ND	ND	ND	ND
tetraflouroethylene	ND	ND	ND	ND
hexafluoropropylene	ND	ND	ND	ND
1,1,1-trifluoroethane	ND	ND	ND	ND
hexafluoropropene oxide	ND	ND	ND	ND
chlorodifluoromethane	ND	ND	ND	ND
1,1,1,2-tetrafluoroethane	ND	ND	ND	ND
perfluorobutane	ND	ND	ND	ND
1H heptafluoropropane	ND	ND	ND	ND
octafluourocyclopentene	ND	ND	ND	ND
trichlorofluoromethane	ND	ND	ND	ND
dodecafluoro-n-pentane	ND	ND	ND	ND
1H nonafluorobutane	ND	ND	ND	ND
tetradecafluorohexane	ND	ND	ND	ND
1H perflluoropentane	ND	ND	ND	ND
heptafluoropropyl 1,2,2,2-tetrafluoroethyl ether (E1)	ND	ND	ND	ND
hexadecaflluroheptane	ND	ND	ND	ND
1H perfluorohexane	ND	ND	ND	ND
perfluorooctane	ND	ND	ND	ND
1H perfluoroheptane	ND	ND	ND	ND
1H Perfluorooctane	ND	ND	ND	ND
2H-Pefluoro-5-methyl-3,6-dioxanonane (E2)	ND	ND	ND	ND

Table 18: GA-EPA SCWO summary of gas emission canister sampling analysis three tests summary. Test C

Can ID	Background dup Canister 37200	GA Test 3 Passing Gas Analyzer Box Canister 37184	GA Test C Sample 1 Canister 37168	GA Test C Sample 2 Canister 37151	GA Test C Sample 3 Canister 37166
Compound Name	GA-062321-AFFF Bkgd ppbv	GA-062321-AFFF Test 3 Passing gas analyzer box ppbv	GA-062321-AFFF Test 3 Sample 1 ppbv	GA-062321-AFFF Test 3 Sample 2 ppbv	GA-062321-AFFF Test 3 Sample 3 ppbv
tetraflouromethane	ND	ND	ND	ND	ND
hexafluoroethane	ND	ND	ND	ND	ND
chlorotrifluoromethane	ND	ND	ND	ND	ND
fluoroform	ND	ND	ND	ND	ND
octafluoropropane	ND	ND	ND	ND	ND
difluoromethane	ND	ND	ND	ND	ND
pentafluoroethane	ND	ND	ND	ND	ND
octafluorocyclobutane	ND	ND	ND	ND	ND
fluoromethane	ND	ND	ND	ND	ND
tetraflouroethylene	ND	ND	ND	ND	ND
hexafluoropropylene	ND	ND	ND	ND	ND
1,1,1-trifluoroethane	ND	ND	ND	ND	ND
hexafluoropropene oxide	ND	ND	ND	ND	ND
chlorodifluoromethane	ND	ND	ND	ND	ND
1,1,1,2-tetrafluoroethane	ND	ND	ND	ND	ND
perfluorobutane	ND	ND	ND	ND	ND
1H heptafluoropropane	ND	ND	ND	ND	ND
octafluourocyclopentene	ND	ND	ND	ND	ND
trichlorofluoromethane	ND	ND	ND	ND	ND
dodecafluoro-n-pentane	ND	ND	ND	ND	ND
1H nonafluorobutane	ND	ND	ND	ND	ND
tetradecafluorohexane	ND	ND	ND	ND	ND
1H perflluoropentane	ND	ND	ND	ND	ND
heptafluoropropyl 1,2,2,2-tetrafluoroethyl ether (E1)	ND	ND	ND	ND	ND
hexadecaflluroheptane	ND	ND	ND	ND	ND
1H perfluorohexane	ND	ND	ND	ND	ND
perfluorooctane	ND	ND	ND	ND	ND
1H perfluoroheptane	ND	ND	ND	ND	ND
1H Perfluorooctane	ND	ND	ND	ND	ND
2H-Pefluoro-5-methyl-3,6-dioxanonane (E2)	ND	ND	ND	ND	ND

Table 19: GA-EPA SCWO summary of gas emission canister sampling analysis three tests summary. Test C & QC

Can ID	GA Test 3 Sample 4 Canister 37158	GA Field Blank Canister 36867	GA Extra Trip Blank Canister 37182	GA Ambient Air Canister 37162	GA Field Blank Canister 37193
Compound Name	GA-062321- AFFF Test 3 Sample 4 ppbv	GA-062321-AFFF Field Blank ppbv	GA-062321-AFFF Extra Trip Blank ppbv	GA-062321-AFFF Ambient Air ppbv	GA-062321-AFFF Field Blank ppbv
tetraflouromethane	ND	ND	ND	ND	ND
hexafluoroethane	ND	ND	ND	ND	ND
chlorotrifluoromethane	ND	ND	ND	ND	ND
fluoroform	ND	ND	ND	ND	ND
octafluoropropane	ND	ND	ND	ND	ND
difluoromethane	ND	ND	ND	ND	ND
pentafluoroethane	ND	ND	ND	ND	ND
octafluorocyclobutane	ND	ND	ND	ND	ND
fluoromethane	ND	ND	ND	ND	ND
tetraflouroethylene	ND	ND	ND	ND	ND
hexafluoropropylene	ND	ND	ND	ND	ND
1,1,1-trifluoroethane	ND	ND	ND	ND	ND
hexafluoropropene oxide	ND	ND	ND	ND	ND
chlorodifluoromethane	ND	ND	ND	ND	ND
1,1,1,2-tetrafluoroethane	ND	ND	ND	ND	ND
perfluorobutane	ND	ND	ND	ND	ND
1H heptafluoropropane	ND	ND	ND	ND	ND
octafluourocyclopentene	ND	ND	ND	ND	ND
trichlorofluoromethane	ND	ND	ND	ND	ND
dodecafluoro-n-pentane	ND	ND	ND	ND	ND
1H nonafluorobutane	ND	ND	ND	ND	ND
tetradecafluorohexane	ND	ND	ND	ND	ND
1H perflluoropentane	ND	ND	ND	ND	ND
heptafluoropropyl 1,2,2,2-tetrafluoroethyl ether (E1)	ND	ND	ND	ND	ND
hexadecaflluroheptane	ND	ND	ND	ND	ND
1H perfluorohexane	ND	ND	ND	ND	ND
perfluorooctane	ND	ND	ND	ND	ND
1H perfluoroheptane	ND	ND	ND	ND	ND
1H Perfluorooctane	ND	ND	ND	ND	ND
2H-Pefluoro-5-methyl-3,6-dioxanonane (E2)	ND	ND	ND	ND	ND

4. CONCLUSIONS AND SUMMARY

4.1 Summary

This report presents the performance and treatability study of a full-scale commercial SCWO system for the destruction of PFAS found in a commercially available AFFF. Additional objectives included an examination of the oxidation of total organic compounds, potential corrosion issues, elucidation of targeted PFAS breakdown products and overall suitability for SCWO to process PFAS-contaminated waste streams.

The AFFF and sample collection/analyses were provided by EPA-ORD, and the SCWO system was provided and operated by GA-EMS. Approximately 15 gallons of AFFF were diluted and processed through a standard 3-gpm iSCWO system over four test runs to confirm the effective elimination of PFAS compounds. Each test treated about 250 gallons of contaminated waste.

- Although there are many studies on SCWO oxidation of chlorinated compounds, this test is one of the few recent studies on the elimination of fluorinated compounds with SCWO that includes both gas and liquid effluent collection and analysis using a commercially available, readily deployable system.
- This study was performed in collaboration between the US EPA ORD and GA-EMS to evaluate the effectiveness of SCWO for the destruction of PFAS. GA-EMS has four decades of experience in safely processing and destroying a wide range of organic feed streams, including numerous halogenated wastes similar to PFAS as well as actual PFAS wastes.
- PFAS elimination averaged >99.99% after treating more than 4800 L of contaminated water.
- The GA-EMS iSCWO technology testing is the first full-scale test to effectively treat waste containing high concentrations of PFAS found in AFFF. The total organic feed to the reactor ranged from 704 g/L to 752 g/L at a feed rate ranging from 8.85 L/min to 9.54 L/min.
- These tests were limited to AFFF diluted in softened water treated at temperatures of 650ºC to mineralize PFAS compounds. (Note that 650°C is the standard operating temperature for GA-EMS iSCWO systems to maintain high organic destruction efficiency in short residence times.) The reaction medium was a homogeneous system where the reaction occurs in a single fluid phase
- Average residence times in the reactor were about 10-12 seconds, significantly more than the minimum required 3-4 seconds for destruction of most organic chemicals as determined in previous tests. The results were reproducible and PFAS destruction efficiencies were >99.99% among the three performance tests (note that the preliminary workup test did not include sample analyses). Varying temperature and reactor residence times were not considered in these tests.

- There was no HF detected in effluent streams. The addition of NaOH at the reactor discharge neutralized acids such as HF that could be generated within the reactor. The judicious selection of corrosive resistant materials for reactor design has been used.

- There was no corrosion observed, and the ICP-AES tests of the effluent stream did not show a significant presence of titanium, chromium, or nickel, which is commonly found in the alloys of the reactor material. This confirms the effectiveness of the unique reactor design, the proper selection of corrosion-resistant materials, and the selected operating conditions consistent with the iSCWO system design.

- Fluorine appears mainly as a fluoride ion in the liquid effluent. Trace amounts of fluorinated by-product compounds were also found in the liquid effluent, but it is unclear if this is coming from the contaminated quench water that was shown to have higher PFAS levels than the liquid effluent. Limited data is available on the SCWO degradation pathways for the different PFAS.

- Previous studies showed that hydrolysis and oxidation occur under SCWO conditions, and the rates are comparable. Air was used as an oxidant for this study. However, the addition of other oxidants shifts products distribution to more complete oxidation products.

- Although analyses of non-targeted PFAS compounds were not confirmed, there was no indication that they would not be destroyed similarly as the targeted PFAS compounds.

- No known PFAS compounds were detected in the gas effluent during these tests.

4.2 Recommendations for Implementation

Dependant on the source parameters, SCWO is a potentially viable technology for treating PFAS contaminated water, including remaining unspent AFFF. There are several systems currently available for sale. As shown in Table 20, there are a few required utilities for sites for the systems to function once purchased & transported. The transportable system requires only a few days for set up and functions similarly to the stationary system.

Table 20: iSCWO Utility Requirements

Description	Units	Quantity	Notes
HP air compressor power	kW	350	More efficient compressors using ~285 kW may be available
Main skid power	kW	25	Single connection to programmable logic controller (PLC) cabinet
Diesel fuel, ultra-low sulfur	gpm	0.3	Other fuels can be considered
Propane (startup)	gal	14	Used for ~30 minutes during startup; other fuels can be considered
Softened water	gpm	10	For startup, shutdown and quench; water recycling possible
35 wt% NaOH solution	g/min	<5	Sufficient Na^+ to form NaF and Na_2SO_4
Instrument air	scfm	15	Clean, dry air for pneumatic valves

In support of sales, GA-EMS offers full scale testing for individualized needs prior to implementing an iSCWO system to validate the efficacy and retain data for intellectual property. The following are suggested topics that are specific to PFAS wastes.

- The tests discussed in this report are first-of-a-kind full-scale tests to demonstrate safe and sustained operations of SCWO for destroying concentrated PFAS. High destruction was achieved for the range of concentrations (AFFF dilution levels 217:1 to 34:1 in water). However, higher AFFF concentrations can be explored provided that the pumping and mixing does not cause excessive foaming or cavitation within the pump chambers. Using an antifoaming agent is recommended until testing with appropriate pumps confirms that no foaming occurs.
- Processing waste streams containing low PFAS concentrations will greatly benefit with the use of a pretreatment concentrating stage such as reverse osmosis (RO). Using this strategy, large volumetric flows can be achieved through a cascading network of RO systems resulting in a low reject flow rate that is amenable to being a feed stream to a SCWO system. This strategy should be further explored for waste streams that have high volumetric flows and low PFAS concentrations.

- The system can be combined with a water recovery system, such as the use of reverse osmosis, to remove solids or dissolved salts, from the effluent. This water can be recycled back to the feeding/quenching operation to minimize water usage.
- Vessel-type SCWO reactors, such as the GA-EMS iSCWO, could be used to process slurries that may contain PFAS. Current remediation systems that use granular activated carbon or ion exchange resins to remove PFAS can be ground into a slurry, then processed through an iSCWO system.

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6. APPENDIX I

Appendix includes partial additional raw data of effluent gas and liquid samples analysis from the SCWO process are presented. A much larger dataset on liquid and gas analysis including adsorbent cartridge analysis, QA/QC tests and canister gas sample analysis are not included in this report.

Data Type	Table
Trace organic compounds sampled from the effluent of SCWO process at the exit of GLS using sorbent tube sampling	18
ICP-OES analysis of effluent wate from Tests A, B, and C,analysis targenting 21 elements	19-21

Table 21: Trace organic compounds sampled from the effluent of SCWO process at the exit of GLS using sorbent tube sampling

Compound	MW	RT, Min	Reference, m/Z	Formula	Total score	SI
Decanedioic acid, didecyl ester	154.17	24.704	56.06209	C30 H58 O4	96.9	845
5-Propyldecane	140.15646	22.897	71.08552	C13 H28	96.5	832
Benzene, (1,3- dimethylbutyl)-	162.14075	24.190	162.1402	C12H18	95.9	810
2,6,10- Trimethyltridecane	154.17207	25.526	85.10117	C16 H34	96.3	817
1-Methylbicyclo (4.4. 0) decane(trans)	152.15646	20.643	152.15591	C11H2O	95.6	784
Bicyclo[3.2.2]nona-6,8-diene, 6,7-dimethyl-	148.12520	22.293	148.12465	C11 H16	98.5	935
Methyl {2'-(benzylamino) carbonyl]-5'-hydroxy-1'-(3''-hydroxypropyl) pentyl}-arbamate	355.07043	24.199	107.08115	C18 H28 N2 O5	93.7	753
Benzene, (1-methylbutyl)-	148.12520	21.758	105.06985	C11 H16	96.2	819
1-butan-2-yl-4- ethyl-benzene	162.14075	24.084	162.14020	C12 H18	95.3	773
Oxalic acid, isohexyl neopentyl ester	147.08096	25.530	72.08889	C13 H24 O4	92.7	632
cis,trans-3-Ethylbicyclo[4.4. 0]decane	166.17209	23.910	166.17154	C12 H22	95.1	770
1-Sec-butyl-4- methylbenzene	148.12520	21.545	120.08890	C11 H16	97.8	908
9- methylheptadecane	159.11735	24.226	140.15591	C18 H38	95.6	792
2,3-Dimethyldodecane	154.17221	24.318	154.17166	C14 H30	93.9	728
Tridecane, 5-propyl-	168.18770	25.452	85.10117	C16 H34	94	699

Compound	MW	RT, Min	Reference, m/Z	Formula	Total score	SI
Cyclohexane, (3-cyclopentylpropyl) -	168.18777	23.928	83.08116	C14 H26	97.8	892
1-Sec-butyl-4-methylbenzene	148.12520	22.113	148.12465	C11 H16	97.5	88
Nonadecane	161.12428	25.380	85.10118	C19 H40	96	816
1-Methylbicyclo (4.4. 0)decane(trans)	152.15646	21.155	152.15591	C11 H20	95.5	782
p-Cymene	134.10950	18.875	134.10895	C10 H14	97.1	870
Benzene, 1-methyl- 3-propyl-	134.10950	18.532	134.10895	C10 H14	97.5	892
6-tert-Butyl-6-methylfulvene	148.12520	23.364	134.10448	C11 H16	99.1	961
2-Methyltricyclo [3.3.3.0(1,5)]undec-2-ene	162.14075	23.707	134.10448	C12 H18	98.1	904
1-Sec-butyl-4- methylbenzene	148.12520	22.524	148.12465	C11 H16	96.6	834
Naphthalene, decahydro-2,3-dimethyl-	166.17209	22.453	152.15150	152.15150	C12 H22	918
Benzene, 2-ethyl- 1,4-dimethyl-	134.10950	19.542	134.10895	C10 H14	C10 H14	902
Nonane, 5-butyl-	126.14085	21.837	126.14030	C13 H28	94.2 713	713
2,3-Diphenyl-2,3-di(N-acetylamino)-butane	162.14075	24.361	120.09324	C20 H24 N2 O2	97.9	912
Perhydrophenalen e, (3aα, 6aα, 9aα,9bβ)-	178.17213	25.665	178.17159	C13 H22	92.3	625

Compound	MW	RT, Min	Reference, m/Z	Formula	Total score	SI
5,10-Dodecadiyn-1-ol	149.13301	25.192	121.09671	$C_{12} H_{18} O$	95	752
1-Propanone, 3-cyclopentyl-1-(2,4-dimethylphenyl)-	151.14859	23.586	148.12465	$C_{16} H_{22} O$	93.9	713
1-Sec-butyl-4-methylbenzene	148.12520	20.159	148.12465	$C_{11} H_{16}$	95.7 7	797
Benzene, 1-methyl-4-propyl-	134.10950	19.145	134.10895	$C_{10} H_{14}$	97.6	899
1-(2,2-dimethylpropyl)-3-methyl-benzene	131.08609	23.615	107.08100	$C_{12} H_{18}$	94	709
Bicyclo[3.2.2]nona-6,8-diene, 6,7-dimethyl-	148.12520	21.397	148.12465	$C_{11} H_{16}$	97.1	864
Undecane, 2,3-dimethyl-	140.15646	22.046	99.11682	$C_{13} H_{28}$	97.1	858
3H-Cyclopenta[1,3]cyclopropa[1, 2]benzene,3a,3b,4,5-tetrahydro-1,3bdimethyl-, [3aS-(3aα,3bβ,7aS*)]-	160.12515	24.118	160.12460	-	98.2	941
Benzene, 1-methyl-2-(1 methylethyl)-	134.10950	20.514	134.10895	-	98.1	916
2,2,4,4,7,7-Hexamethyloctahy dro-1H-indene	193.19553	26.548	193.19498	$C_{15} H_{28}$	91.6	587
(3R,5R)-2-tert-Butoxycarbonyl-1-(4-nitrophenylsulfonyl)-5-octylpyrazolidin-3-ol	162.14075	24.978	180.18713	$C_{22} H_{35} N_3 O_7 S$	92.5	626
2-(trans-2-Methylcyclohexyl) buta-1,3-diene	150.14098	20.167	106.07773	$C_{11} H_{18}$	93.6	746
C1-epimer of [1S,4aS,8aS] -1,2,3,4,4a,7,8,8a -octahydro -1,4a,5,6 -tetramethyl -naphthalene	192.18779	26.122	177.16374	$C_{14} H_{24}$	94.2	741

Compound	MW	RT, Min	Reference, m/Z	Formula	Total score	SI
2,3-Dimethyldecane	126.14085	21.704	127.14816	$C_{12}H_{26}$	97.5	873
Octane, 2-cyclohexyl-	154.17207	21.120	154.17152	$C_{14}H_{28}$	97.5	892
3-Pyridinemethanol, 5-hydroxy-4-[[[[3-hydroxy-5-(hydroxymethyl)-2-methyl-4-pyridinyl] methylene] amino] methyl]-6-methyl-,(E)-	151.14868	22.908	152.15150	$C_{16}H_{19}N_3O_4$	98.2	907
2-Cyclobutylidenspiro[4.4]nonane	176.15642	24.551	134.10448	$C_{13}H_{20}$	99.5	992
Naphthalene, decahydro-2-methyl-	152.15646	21.334	137.13252	$C_{11}H_{20}$	94.9	745
1,2-Dimethyl-1-phenylcyclopropan	146.10951	21.473	146.10896	$C_{11}H_{14}$	98.9	9 960
e isomer	-	-	-	-	-	-
3,3-Dimethyl-1-phenyl-1-pentene	145.10171	23.602	146.10448	$C_{13}H_{18}$	92.3	648
Octane, 2,6,6-trimethyl-	141.16431	20.748	99.11682	$C_{11}H_{24}$	97	850
(3R*,4R*)-3-(2-Nitro-4-methoxyphenyl)-4-(4-hydroxyphenyl) hexane	149.02383	24.974	180.18716	C19 H23 N O4	94.5	848
Benzene, 1-methyl-4-(1-ethylethyl)-	119.08610	20.413	120.08890	C10 H14	96.3	814
heneicosa-1,20-diene	96.09392	22.890	96.09337	C21 H40	91.8	660
Butyric acid, 2-phenyl-, oct-3-en-2-yl ester	134.10950	20.021	134.10895	C18 H26 O2	91	550
4-Nonene, 3,7-dimethyl-6-methylene-, [S-[R*,R*-(E)]]-	137.13307	24.040	136.12468	C12 H22	96.1	867

Compound	MW	RT, Min	Reference, m/Z	Formula	Total score	SI
(Z)-1,7-Octadadiene	55.05480	25.648	192.18724	C18 H34	92.7	634
2-ethylhexyl ester2,4-dicyano-2-Methylbutanoic acid ester	112.12527	24.056	113.12807	C15 H24 N2 O2	92.4	617
4,1,6-[1,2,3]Propanetriyl-1Hindene, octahydro-6-ethyl-	-	-	-	-	-	-
Benzoic acid, 2,4-dihydroxy-3-[(2-hydroxy-4-methoxy-6-propylbenzo yl)oxy] -6-propyl-, methyl ester	194.20338	25.984	194.20284	C22 H26 O8	92.8	664
1-ethyl-2,2,3,3,4,4,5,5-octamethylcyclopentan	111.11739	24.242	114.13585	C15 H30	94.6	772
2-Norpinene-2-ethanol, 6,6-dimethyl-, acetate	148.12520	20.532	134.10448	C13 H20 O2	98	906
1-(1'-Ethyl)-3,3-dimethylcyclopropene	81.07045	25.800	25.800	C7 H24	94.1	750
Adamantane, 1,3-dimethyl-	164.15634	20.923	150.13579	C12 H20	96.1	831
1HCyclopentacyclooctene, decahydro-,cis-	152.15646	21.983	153.15929	C11 H20	96.7	861
3-Octen-2-one, 3-ethyl-4-methyl	151.14868	23.068	152.15150	C11 H20 O	98.9	969
(E,E)-4,8,12-Trimethyl-1,3,7,11-tridecatetraene	69.07043	26.307	187.14809	C16 H26	90.5	570
1,3,5-Trimethyladamantane	178.17213	23.220	164.15131	C13 H22	99.8	987
4-methyl-4-propyl-1-cyclohex-2-enone	110.10955	19.050	82.07326	C10 H16 O	94.5	849
Pentanoic acid, 5-hydroxy-, p-tbutylphenylester	152.15643	22.380	150.14021	C15 H22 O3	94.8	737

Compound	MW	RT, Min	Reference, m/Z	Formula	Total score	SI
Neopentylidenecyc lohexane	152.15646	19.273	137.13252	C11 H20	94.3	745
(3Z)-1,1-dimethyl-3-(2-methylpropylidene)cyclopentane	152.15646	22.163	153.15929	C11 H20	98.6	927
1,2,3-Trimethylperhydro naphthalene	152.15646	19.886	96.08892	C13 H22	95	773
1,8-Nonadiyne	176.15645	24.150	120.08894	C9 H12	98.6	966
(1R,2R)-1-but-1-ynyl-2-ethenylcyclopentane	148.12520	21.316	134.10448	C11 H16	97.5	902
Hexadecane	205.19566	27.175	207.21060	C16 H34	92.1	634
1-(1,1,3-trimethyl-2-butenyl)-1-cyclohexene	178.17213	21.381	164.15131	C13 H22	97.3	875
5-Decyne	138.14088	17.297	138.14034	C10 H18	97.2	861
3-(2-Hydroxy-2-methylpropyl)-5,5-dimethyl-2-cyclohexen-1-one	138.14088	20.180	139.14369	C12 H20 O2	C12 H20 O2	625
2-(1,2-epoxycycloheptyl)-1-pentene	151.14859	25.050	152.15144	C12 H20 O	94.2	809
cis,cis-2,7-Nonadiene	69.07048	25.975	202.17166	C9 H16	90.2	635
Adamantane, 1,3-dimethyl-	164.15643	23.635	150.13588	C12 H20	99.2	979
3-isopropyl-8a,9-dimethyl-4-phenyl(hexahydro)-1Hpyrrolo[3',4' :3,4]pyrrolo[2,1-c]-(1,4)oxazine-1,7,	146.10951	20.155	106.07320	C20 H24 N2	90.3	514
2-[(2-isopropyl-4-keto-quinazolin-3-yl)amino]-1-phenylbutane-1,3-dione	104.06261	25.771	196.21855	C21 H21 N3 O3	94.6	834

Compound	MW	RT, Min	Reference, m/Z	Formula	Total score	SI
2-butyl-5-methylhexa-1,4-diene	152.15649	19.048	152.15594	C11 H20	96.5	851
Tricyclo[4.3.1.13, 8]undecane, 3-methyl	164.15634	22.778	150.13579	C12 H20	99.7	985
Cyclopentene, 3-ethyl-1-pentyl-	137.13307	19.917	138.13594	C12 H22	91.2	558
Cyclohexane, 1,1'- (1-methylethylidene)bis-	124.12517	21.345	124.12463	C15 H28	96.4	819
Octane, 2-methyl-	113.13306	14.405	72.08887	96.7	96.7	847
2-(Hydroxyimino)- 2-(4-methoxyphenyl) ethyl 4-butyramidobenzoate	137.13303	20.122	134.10451	C20 H22 N2 O5	95.5	772
Dibenzo[a,i]biphenylene,5,6,6a,6b,7,8,12b,12c-octahydro-2,11-dimethoxy-	160.12514	23.290	161.12793	C22 H24 O2	90.1	860
Benzene, 2-(butenyl)-5-(1,1-dimethylethyl)-1,3-dimethyl-	216.18774	27.643	216.18719	C16 H24	94.9	824
Ethanone, 2-(acetyloxy)-1-phenyl	105.07049	106.07349	106.07349	C10 H10 O3	90.4	926
1,3-Ditert-butyl-2-methoxy-4-oxo-3-azetidinecarbonitrile	139.14863	21.341	140.15154	C13 H22 N2 O2	91.7	780
(R)-6-ethyl-5-methyloct-3-ene	154.17209	20.041	154.17154	154.17154	902	902
1-Methyl-3,5-bis(cyclohexyl)-1-phospha-3,5-diazacyclohexane	222.23479	27.028	222.23424	C16 H31 N2 P	91.4	647
2-cyano-3-dimethylamino-N-(9,10-dioxo-9,10-dihydro-anthracen-2-yl)-acrylamide	123.11747	19.398	124.12037	C20 H15 N3 O3	94.4	721
4,4-Dimethyl-(exo,trans)-etracyclo[5.2.1.0(2,6).0(3,5)]dec-8-ene	160.12512	21.916	146.10445	C12 H16	98	928
(2R)-2-methyl-2-(4-methylpent-3-enyl)oxirane	109.10179	20.344	110.10458	C9 H16 O	97.5	912
(1R*,2S*,5S*)-2-Methyl-4-oxabicyclo[3.2.0]heptan-1-ol	128.08375	15.909	113.05978	C7 H12 O2	93.8	723

Table 22. ICP-AES Sample analysis of effluent stream analysis Test A

ANALYSIS TECHNIQUE:	ICP-AES	**Calibration date:**	03/10/20
ANALYSIS METHOD:	30M_2021-Rev-A	**Calibration Standards:**	L:\Priv\CtrHill\WA 2-38\WA 2-38 ICP-AES\FY2017-2018 ICP-AES Data Reports
ANALYSIS BATCH:	TO 148-02	**Task Order#**	68HERC21F0148
PROJECT:	TO 148	**Sample Dates:**	
UNITS:	mg/L	**Analyst:**	MA
Comments:		SOP# K-LRTD-SOP-1185-1	
Sample results are reported as mg/L		(NRMRL-LRPCD-11-1, G-LMMD-SOP-1185-1)	
Standards results are reported as intensity values.			

SAMPLE	TIME	B	Ca	Cr	Cu	K	Mo	Na	Ni	P	S	Se	Si
GA-EPA-LI-A	7/9/2021 11:42:47AM	0.122	<0.404	<0.018	0.010	0.594	0.005	230.600	<0.003	0.029	97.200	<0.010	3.738
GA-EPA-LI-B	7/9/2021 11:47:34AM	0.122	<0.404	<0.018	0.010	0.639	0.005	227.700	<0.003	0.030	98.780	<0.010	3.665
GA-EPA-LEB-A	7/9/2021 11:52:22AM	0.867	3.405	0.190	<0.005	3.220	0.158	946.200	0.242	0.080	365.600	0.015	18.340
GA-EPA-LEB-B	7/9/2021 11:57:08AM	0.865	3.460	0.192	0.006	3.200	0.158	978.000	0.255	0.080	372.400	0.016	18.390
LE-A-01-A	7/9/2021 12:01:53PM	1.001	1.020	0.254	0.030	6.090	0.234	1848.000	0.114	0.508	526.100	0.064	23.060
LE-A-01-B	7/9/2021 12:06:37PM	0.991	1.433	0.254	0.023	6.690	0.224	1641.000	0.113	0.419	496.000	0.050	21.880
LE-A-01-C	7/9/2021 12:11:23PM	0.979	1.066	0.257	0.032	7.810	0.237	1788.000	0.117	0.521	528.500	0.061	22.590
LE-A-02-A	7/9/2021 12:16:07PM	1.012	0.994	0.203	0.032	8.420	0.176	1816.000	0.077	0.516	520.200	0.060	23.890
LE-A-02-B	7/9/2021 12:20:53PM	0.935	0.921	0.184	0.034	8.130	0.175	1740.000	0.093	0.521	522.800	0.063	22.540
LE-A-02-C	7/9/2021 12:25:37PM	0.937	0.885	0.185	0.032	8.320	0.174	1854.000	0.069	0.520	523.100	0.063	22.070
GA-EPA-SenDiego DIW	7/9/2021 12:44:18PM	0.191	<0.404	<0.018	0.006	0.690	0.005	237.200	<0.003	0.008	78.670	<0.010	3.109
GA-EPA-SenDiego TapW	7/9/2021 12:49:08PM	0.116	66.430	<0.018	0.163	5.010	0.004	98.410	<0.003	<0.008	72.810	<0.010	2.903
LE-A-03-A	7/9/2021 12:53:46PM	0.822	0.979	0.136	0.050	5.910	0.169	1834.000	0.055	0.445	479.900	0.051	19.920
LE-A-03-B	7/9/2021 12:58:32PM	0.828	0.912	0.132	0.030	7.100	0.168	1814.000	0.054	0.461	477.000	0.047	19.930
LE-A-03-C	7/9/2021 1:03:18PM	0.903	1.055	0.146	0.026	8.120	0.169	1960.000	0.059	0.450	475.900	0.047	21.890
GA-EPA-SenDiego DIW	7/9/2021 1:08:33PM	0.185	<0.404	<0.018	0.006	0.953	0.004	247.400	<0.003	0.009	73.940	<0.010	2.983
10xGAEPASeDiegoDIW	7/9/2021 1:16:50PM	0.176	<0.404	<0.018	0.009	0.738	0.004	236.400	<0.003	0.010	74.390	<0.010	2.859
10xGAEPASenDiegoTapW	7/9/2021 1:22:00PM	0.114	67.220	<0.018	0.207	5.230	0.004	102.900	<0.003	0.008	73.870	<0.010	2.875
LE-A-04-A	7/9/2021 1:26:45PM	0.727	0.817	0.104	0.058	5.460	0.161	1653.000	0.046	0.378	441.200	0.037	17.650
LE-A-04-B	7/9/2021 1:31:31PM	0.699	0.702	0.096	0.029	5.940	0.160	1610.000	0.047	0.373	437.800	0.040	17.020
LE-A-04-C	7/9/2021 1:36:18PM	0.684	0.685	0.100	0.022	6.070	0.157	1544.000	0.046	0.365	427.900	0.040	16.640
LE-A-04-C AnDup	7/9/2021 1:41:05PM	0.706	0.715	0.100	0.021	6.350	0.161	1608.000	0.049	0.373	442.000	0.041	17.300
07092021 Lab Blank	7/9/2021 2:04:15PM	<0.020	<0.404	<0.018	<0.005	<0.317	<0.004	0.439	<0.003	<0.008	<0.033	<0.010	<0.114
10x070921 BlkSpk	7/9/2021 2:08:59PM	0.046	0.468	0.244	0.269	0.674	0.103	0.965	0.272	0.098	0.268	0.246	<0.114
070921 BlkSpk	7/9/2021 2:13:39PM	0.423	5.025	2.419	2.570	6.070	1.010	6.170	2.609	1.026	2.621	2.445	0.953
070921 BlkSpkAnDup	7/9/2021 2:18:10PM	0.419	5.005	2.403	2.580	6.000	1.010	6.013	2.616	1.030	2.651	2.459	0.932
D10xGA-EPA-LI-A	7/9/2021 2:31:52PM	<0.020	<0.404	<0.018	<0.005	<0.317	<0.004	26.830	<0.003	<0.008	9.565	<0.010	0.318
D10xGA-EPA-LI-B	7/9/2021 2:36:35PM	<0.020	<0.404	<0.018	<0.005	<0.317	<0.004	26.610	<0.003	<0.008	9.445	<0.010	0.328
D10xGA-EPA-LEB-A	7/9/2021 2:41:18PM	0.075	<0.404	<0.018	<0.005	0.355	0.015	126.700	0.026	<0.008	34.910	<0.010	1.561
D10xGA-EPA-LEB-B	7/9/2021 2:45:59PM	0.075	<0.404	<0.018	<0.005	<0.317	0.016	126.600	0.028	<0.008	35.750	<0.010	1.563
D10xLE-A-01-A	7/9/2021 2:50:43PM	0.087	<0.404	0.028	<0.005	0.523	0.023	313.200	0.012	0.020	49.350	<0.010	1.981
D10xLE-A-01-B	7/9/2021 2:55:32PM	0.091	<0.404	0.020	<0.005	0.513	0.021	264.500	0.012	0.018	46.010	<0.010	1.855
D10xLE-A-01-C	7/9/2021 3:00:23PM	0.083	<0.404	0.028	0.005	0.560	0.024	310.600	0.013	0.021	49.250	<0.010	1.974
D10xLE-A-02-A	7/9/2021 3:05:15PM	0.088	<0.404	<0.018	0.005	0.577	0.018	312.400	0.010	0.025	49.690	<0.010	2.065
D10xLE-A-02-B	7/9/2021 3:10:07PM	0.087	<0.404	<0.018	0.005	0.539	0.017	302.300	0.009	0.023	48.960	<0.010	1.999
D10xLE-A-02-C	7/9/2021 3:14:57PM	0.087	<0.404	<0.018	<0.005	0.559	0.017	300.700	0.008	0.022	49.090	<0.010	1.949
10xGAEPASeDiegoDIW	7/9/2021 3:33:43PM	<0.020	<0.404	<0.018	<0.005	<0.317	<0.004	26.050	<0.003	<0.008	7.072	<0.010	0.279
10xGAEPASenDiegoTapW	7/9/2021 3:38:26PM	<0.020	6.611	<0.018	0.029	0.566	<0.004	10.750	<0.003	<0.008	6.660	<0.010	0.267
D10xLE-A-03-A	7/9/2021 3:43:06PM	0.075	<0.404	<0.018	<0.005	0.443	0.016	261.100	0.006	0.021	46.260	<0.010	1.805
D10xLE-A-03-B	7/9/2021 3:47:57PM	0.077	<0.404	<0.018	<0.005	0.509	0.016	264.800	0.006	0.021	47.110	<0.010	1.808
D10xLE-A-03-C	7/9/2021 3:52:49PM	0.075	<0.404	<0.018	<0.005	0.404	0.016	256.800	0.006	0.017	45.760	<0.010	1.756
D10xLE-A-04-A	7/9/2021 3:57:35PM	<0.020	<0.404	<0.018	<0.005	<0.317	<0.004	0.367	<0.003	<0.008	<0.033	<0.010	<0.114
D10xLE-A-04-B	7/9/2021 4:02:11PM	<0.020	<0.404	<0.018	<0.005	<0.317	<0.004	0.229	<0.003	<0.008	<0.033	<0.010	<0.114
D10xLE-A-04-C	7/9/2021 4:06:47PM	<0.020	<0.404	<0.018	<0.005	<0.317	<0.004	<0.191	<0.003	<0.008	<0.033	<0.010	<0.114

		B	Ca	Cr	Cu	K	Mo	Na	Ni	P	S	Se	Si
Established 2016MRL		0.020	0.404	0.018	0.005	0.317	0.004	0.191	0.003	0.008	0.033	0.010	0.114
Lowest Cal Std	mg/L	1.0	5.0	1.0	1.0	5.0	1.0	5.0	1.0	2.0	5.0	0.5	2.0
Highest Cal Std	mg/L	20	2000	20	20	100	20	100	20	100	200	10	100
Correlation**		0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999
Re-slope													

Where:

For QC2 , QC Ag, QC Al+Fe 100, QC S50, QC P10

Yellow	QC Sample
Dark blue	Raw Data (w/o applying MRL)
Bright green	90≤R%≤110
Orange	85≤R%≤89 or 111≤R%≤115
Red	85 > R% >115
Peach	Low Level QC Check
Light blue	Dilution Required
Grey	QC CCV Failed
Black	Sampling error
Light orange	0.800>Re-slope>1.200

For QC2xDil100, QC2xDil10, QC1 and other Sp R%

Light green	70≤R%≤130
Dark orange	50≤R%≤70 or 131≤R%≤150
Dark red	50 > R% >150
Brown	RPD>20%
Dark green	20%<RPD

^ ***** No Data (due to extremely high IEC element concentration level)

C .000 Extremely High Concentration (reanalysis of the necessary dilution is required for accepting QA acceptable Data)

Table 23. ICP-AES Sample analysis of effluent stream analysis Test B

ANALYSIS TECHNIQUE:	ICP-AES	**Calibration date:**	03/10/20
ANALYSIS METHOD:	30M_2021-Rev-A	**Calibration Standards**	L:\Priv\CtrHill\WA 2-38\WA 2-38 ICP-AES\FY2017-2018 ICP-AES Data Reports
ANALYSIS BATCH:	TO 142-66 + TO 148-04	**Task Order#**	68HERC21F0148
PROJECT:	TO 148	**Sample Dates:**	
UNITS:	mg/L	**Analyst:**	MA
Comments:		SOP# K-LRTD-SOP-1185-1	
Sample results are reported as mg/L		(NRMRL-LRPCD-11-1, G-LMMD-SOP-1185-1)	
Standards results are reported as intensity values.			

SAMPLE	TIME	Ag	Al	As	B	Ba	Ca	Cd	Co	Cr	Cu	Fe
08042021-Lab Blank	8/4/2021	0.015	<0.096	<0.005	<0.020	<0.005	<0.404	<0.0005	<0.001	<0.018	<0.005	<0.037
10x0842021_BlkSpk	8/4/2021	0.026	0.561	0.255	0.052	0.266	0.533	0.1098	0.552	0.272	0.277	0.536
08042021-Blk Spike	8/4/2021	0.114	5.540	2.525	0.512	2.636	5.399	1.0720	5.352	2.667	2.670	5.350
LI-B-01-A	8/4/2021	0.016	<0.096	<0.005	0.128	<0.005	<0.404	<0.0005	<0.001	<0.018	0.009	<0.037
LI-B-01-B	8/4/2021	0.016	<0.096	<0.005	0.126	<0.005	<0.404	<0.0005	<0.001	<0.018	0.008	<0.037
LE-B-01-A	8/4/2021	0.015	<0.096	<0.005	0.854	<0.005	3.186	<0.0005	0.003	0.204	0.012	0.101
LE-B-01-B	8/4/2021	0.015	<0.096	<0.005	0.819	0.006	3.756	<0.0005	0.003	0.197	0.011	0.079
LE-B-01-C	8/4/2021	0.015	<0.096	<0.005	0.858	0.007	3.360	<0.0005	0.002	0.198	0.013	0.061
LE-B-02-A	8/4/2021	0.015	<0.096	<0.005	0.955	<0.005	1.200	<0.0005	<0.001	0.345	0.027	<0.037
LE-B-02-B	8/4/2021	0.015	<0.096	<0.005	0.945	<0.005	1.166	<0.0005	<0.001	0.337	0.026	<0.037
LE-B-02-C	8/4/2021	0.015	<0.096	<0.005	0.950	<0.005	1.162	<0.0005	<0.001	0.336	0.029	<0.037
LE-B-03-A	8/4/2021	0.015	<0.096	<0.005	1.367	<0.005	0.968	<0.0005	<0.001	0.313	0.029	<0.037
LE-B-03-B	8/4/2021	0.015	<0.096	<0.005	1.361	<0.005	0.986	<0.0005	<0.001	0.312	0.029	<0.037
LE-B-03-C	8/4/2021	0.014	<0.096	<0.005	1.331	<0.005	0.951	<0.0005	<0.001	0.299	0.026	<0.037
LE-B-04-A	8/4/2021	0.015	<0.096	<0.005	1.411	<0.005	1.128	<0.0005	<0.001	0.230	0.021	<0.037
LE-B-04-B	8/4/2021	0.015	<0.096	<0.005	1.333	<0.005	1.058	<0.0005	<0.001	0.213	0.022	<0.037
LE-B-04-C	8/4/2021	0.015	<0.096	<0.005	1.409	<0.005	1.145	<0.0005	<0.001	0.234	0.023	<0.037
LE-B-05-A	8/4/2021	0.015	<0.096	<0.005	1.401	<0.005	1.242	<0.0005	<0.001	0.178	0.023	<0.037
LE-B-05-B	8/4/2021	0.015	<0.096	<0.005	1.306	<0.005	1.122	<0.0005	<0.001	0.168	0.024	<0.037
LE-B-05-C	8/4/2021	0.016	<0.096	<0.005	1.296	<0.005	1.103	<0.0005	<0.001	0.164	0.025	<0.037

SAMPLE	K	Mg	Mn	Mo	Na	Ni	P	Pb	S	Sb	Se	Si	Sr	Ti	V
08042021-Lab Blank	<0.317	<0.134	< 0.007	<0.004	<0.191	<0.003	<0.008	<0.039	<0.033	<0.010	<0.010	<0.114	<0.005	<0.008	<0.016
10x0842021_BlkSpk	0.440	0.598	0.111	0.108	0.415	0.283	0.107	0.265	0.253	0.264	0.261	<0.114	0.110	0.107	0.268
08042021-Blk Spike	5.500	5.585	1.100	1.060	5.320	2.739	1.061	2.598	2.752	2.600	2.562	1.086	1.090	1.069	2.670
LI-B-01-A	0.552	4.154	< 0.007	0.005	238.500	<0.003	0.035	<0.039	102.600	<0.010	<0.010	3.659	<0.005	<0.008	<0.016
LI-B-01-B	0.557	4.247	< 0.007	0.004	242.200	<0.003	0.032	<0.039	103.100	<0.010	<0.010	3.514	<0.005	<0.008	<0.016
LE-B-01-A	2.870	1.458	< 0.007	0.222	980.400	0.473	0.199	<0.039	369.900	<0.010	0.018	16.540	0.039	1.130	<0.016
LE-B-01-B	2.910	1.447	< 0.007	0.219	950.400	0.567	0.194	<0.039	365.200	<0.010	0.016	15.880	0.044	0.768	<0.016
LE-B-01-C	3.040	1.406	< 0.007	0.219	999.900	0.436	0.196	<0.039	368.600	<0.010	0.016	16.500	0.040	0.584	<0.016
LE-B-02-A	4.940	1.316	< 0.007	0.168	1949.000	0.150	0.598	<0.039	455.200	0.027	0.063	18.940	0.014	0.075	<0.016
LE-B-02-B	4.780	1.250	< 0.007	0.166	2012.000	0.097	0.607	<0.039	450.800	0.027	0.065	18.790	0.014	0.033	<0.016
LE-B-02-C	4.960	1.291	< 0.007	0.169	2008.000	0.104	0.617	<0.039	453.900	0.028	0.074	18.960	0.014	0.060	<0.016
LE-B-03-A	6.220	2.740	< 0.007	0.266	2272.000	0.134	0.673	<0.039	618.800	0.025	0.069	26.120	0.010	0.025	<0.016
LE-B-03-B	6.220	2.765	< 0.007	0.270	2276.000	0.195	0.678	<0.039	629.600	0.025	0.071	26.070	0.010	0.028	<0.016
LE-B-03-C	5.790	2.753	< 0.007	0.269	2137.000	0.118	0.653	<0.039	621.400	0.026	0.072	25.280	0.010	0.050	<0.016
LE-B-04-A	6.320	2.538	< 0.007	0.264	2095.000	0.063	0.508	<0.039	625.400	0.020	0.055	27.100	0.011	0.035	<0.016
LE-B-04-B	6.260	2.378	< 0.007	0.264	2043.000	0.060	0.508	<0.039	627.700	0.017	0.052	25.290	0.011	0.023	<0.016
LE-B-04-C	6.600	2.691	< 0.007	0.264	2083.000	0.070	0.513	<0.039	632.100	0.018	0.051	27.060	0.012	0.057	<0.016
LE-B-05-A	6.680	2.392	< 0.007	0.255	2023.000	0.104	0.435	<0.039	625.800	0.014	0.045	26.860	0.013	0.051	<0.016
LE-B-05-B	6.480	2.397	< 0.007	0.251	1975.000	0.079	0.426	<0.039	610.600	0.014	0.044	25.260	0.012	0.117	<0.016
LE-B-05-C	6.570	2.564	< 0.007	0.245	1950.000	0.076	0.428	<0.039	593.600	0.013	0.044	25.360	0.012	0.138	<0.016

		Ag	Al	As	B	Ba	Ca	Cd	Co	Cr	Cu	Fe
Established 2016MRL		0.008	0.096	0.005	0.020	0.005	0.404	0.0005	0.001	0.018	0.005	0.037
Lowest Cal Std	mg/L	0.05	1.0	0.5	1.0	0.5	5.0	0.5	1.0	1.0	1.0	1.0
Highest Cal Std	mg/L	1	200	10	20	10	2000	10	20	20	20	200
Correlation**		0.999	0.999	0.999	0.999	0.999	0.999	0.9998	0.999	0.999	0.999	0.999
Re-slope												

	K	Mg	Mn	Mo	Na	Ni	P	Pb	S	Sb	Se	Si	Sr	Ti	V
Established 2016MRL	0.317	0.134	0.007	0.004	0.191	0.003	0.008	0.039	0.033	0.010	0.010	0.114	0.005	0.008	0.016
Lowest Cal Std	5.0	5.0	1.0	1.0	5.0	1.0	2.0	0.5	5.0	0.5	0.5	2.0	0.5	0.5	0.5
Highest Cal Std	100	2000	20	20	100	20	100	200	200	10	10	100	10	10	10
Correlation**	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999
Re-slope															

Where:

For QC2 , QC Ag, QC Al+Fe 100, QC S50, QC P10

(light blue)	Dilution Required
(grey)	QC CCV Failed
(dark)	Sampling error
(yellow)	0.800>Re-slope>1.200
^ *****	No Data (due to extremely high IEC element concentration level)
C .000	Extremely High Concentration (reanalysis of the necessary dilution is required for accepting QA acceptable Data)

Table 24. ICP-AES Sample analysis of effluent stream analysis Test C

ANALYSIS TECHNIQUE:	ICP-AES	**Calibration date:**	03/10/20
ANALYSIS METHOD:	30M_2021-Rev-A	**Calibration Standards:**	L:\Priv\CtrHill\WA 2-38\WA 2-38 ICP-AES\FY2017-2018 ICP-AES Data Reports
ANALYSIS BATCH:	TO 148-07	**Task Order#**	68HERC21F0148
PROJECT:	TO 148	**Sample Dates:**	
UNITS:	mg/L	**Analyst:**	MA
Comments:		SOP# K-LRTD-SOP-1185-1	
Sample results are reported as mg/L		(NRMRL-LRPCD-11-1, G-LMMD-SOP-1185-1)	
Standards results are reported as intensity values.			

SAMPLE	TIME	B	Mg	Mo	Na	Ni	P	Pb	S	Si
Dil10x LI-C-01-A	8/13/2021 11	<0.020	1.906	<0.004	29.050	<0.003	<0.008	<0.039	17.020	0.362
Dil10x LI-C-01-B	8/13/2021 11	<0.020	1.837	<0.004	28.980	<0.003	<0.008	<0.039	16.810	0.352
Dil10x LE-C-01-A	8/13/2021 11	<0.020	1.769	<0.004	28.180	<0.003	<0.008	<0.039	16.520	0.340
Dil10x LE-C-01-B	8/13/2021 11	<0.020	1.817	<0.004	28.950	<0.003	<0.008	<0.039	16.770	0.345
Dil10x LE-C-01-C	8/13/2021 11	<0.020	1.807	<0.004	28.600	<0.003	<0.008	<0.039	16.690	0.360
Dil10x LE-C-02-A	8/13/2021 11	0.108	<0.134	0.031	275.000	0.010	0.010	<0.039	55.030	2.292
Dil10x LE-C-02-B	8/13/2021 11	0.103	<0.134	0.031	258.900	0.007	0.009	<0.039	54.990	2.186
Dil10x LE-C-02-C	8/13/2021 11	0.104	<0.134	0.031	263.800	0.007	0.010	<0.039	53.930	2.182
Dil10x LE-C-03-A	8/13/2021 11	0.074	<0.134	0.022	209.900	0.004	<0.008	<0.039	41.990	1.731
Dil10x LE-C-03-B	8/13/2021 11	0.074	<0.134	0.022	208.600	0.004	<0.008	<0.039	41.940	1.731
Dil10x LE-C-03-C	8/13/2021 12	0.076	<0.134	0.022	207.100	0.004	<0.008	<0.039	42.390	1.722
Dil10x LE-C-04-A	8/13/2021 12	0.075	<0.134	0.022	203.800	0.016	<0.008	<0.039	39.360	1.627
Dil10x LE-C-04-B	8/13/2021 12	0.069	<0.134	0.023	205.900	0.010	<0.008	<0.039	40.280	1.627
Dil10x LE-C-04-C	8/13/2021 12	0.067	<0.134	0.021	191.500	0.009	<0.008	<0.039	38.280	1.521
Dil10x LE-C-05-A	8/13/2021 12	0.066	<0.134	0.018	196.900	0.004	0.008	<0.039	39.040	1.523
Dil10x LE-C-05-B	8/13/2021 12	0.066	<0.134	0.017	187.900	0.003	<0.008	<0.039	36.990	1.477
Dil10x LE-C-05-C	8/13/2021 12	0.069	<0.134	0.018	198.500	0.004	<0.008	<0.039	38.700	1.556
08132021 Lab Blank	8/13/2021 1:	<0.020	<0.134	<0.004	<0.191	<0.003	<0.008	<0.039	<0.033	<0.114
Dil10x 081321 BlkSpk	8/13/2021 1:	0.049	0.500	0.099	0.473	0.258	0.100	0.242	0.262	<0.114
081321 BlkSpk	8/13/2021 1:	0.491	5.099	0.988	4.939	2.547	1.016	2.427	2.644	1.014
081321 BlkSpk AnD	8/13/2021 1:	0.490	5.104	0.991	4.939	2.553	1.016	2.429	2.646	1.009

		B	Mg	Mo	Na	Ni	P	Pb	S	Si
Established 2016MRL		0.020	0.134	0.004	0.191	0.003	0.008	0.039	0.033	0.114
Lowest Cal Std	mg/L	1.0	5.0	1.0	5.0	1.0	2.0	0.5	5.0	2.0
Highest Cal Std	mg/L	20	2000	20	100	20	100	200	200	100
Correlation**		0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999	0.999
Re-slope										

Where:

For QC2 , QC Ag, QC Al+Fe 100, QC S50, QC P10

	Dilution Required
	QC CCV Failed
	Sampling error
	0.800>Re-slope>1.200
^ *****	No Data (due to extremely high IEC element concentration level)
C .000	Extremely High Concentration (reanalysis of the necessary dilution is required for accepting QA acceptable Data)

Logged on User: User1
Instrument: ICS-6000
Sequence: Ayu_01102023

Page 1 of 1
1/19/2023 8:47 AM

Peak Integration Report

Sample Name:	**L7**	*Inj. Vol.:*	**10.00**
Injection Type:	**Calibration Standard**	*Dilution Factor:*	**1.0000**
Instrument Method:	**Anions10292022**	*Operator:*	**User1**
Inj. Date / Time:	**10-Jan-2023 / 20:00**	*Run Time:*	**16.00**

No.	Time min	Peak Name	Peak Type	Area µS*min	Height µS	Amount ppm
1	2.68	Fluoride	M *^	10.929	64.756	19.8761
2	4.04	Chloride	BM *^	6.649	70.398	20.1341
5	7.27	Bromide	M *	4.456	21.071	20.0002
6	8.06	Nitrate	M	6.720	31.332	20.6030
		TOTAL:		28.75	187.56	80.61

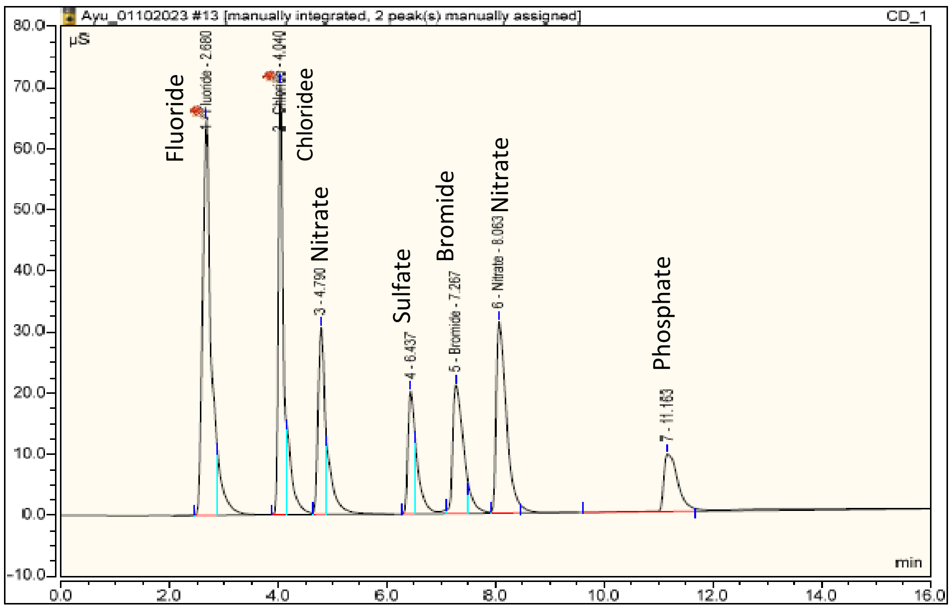

Figure 20: *Ion Chromatography analysis of liquid standard mixture*

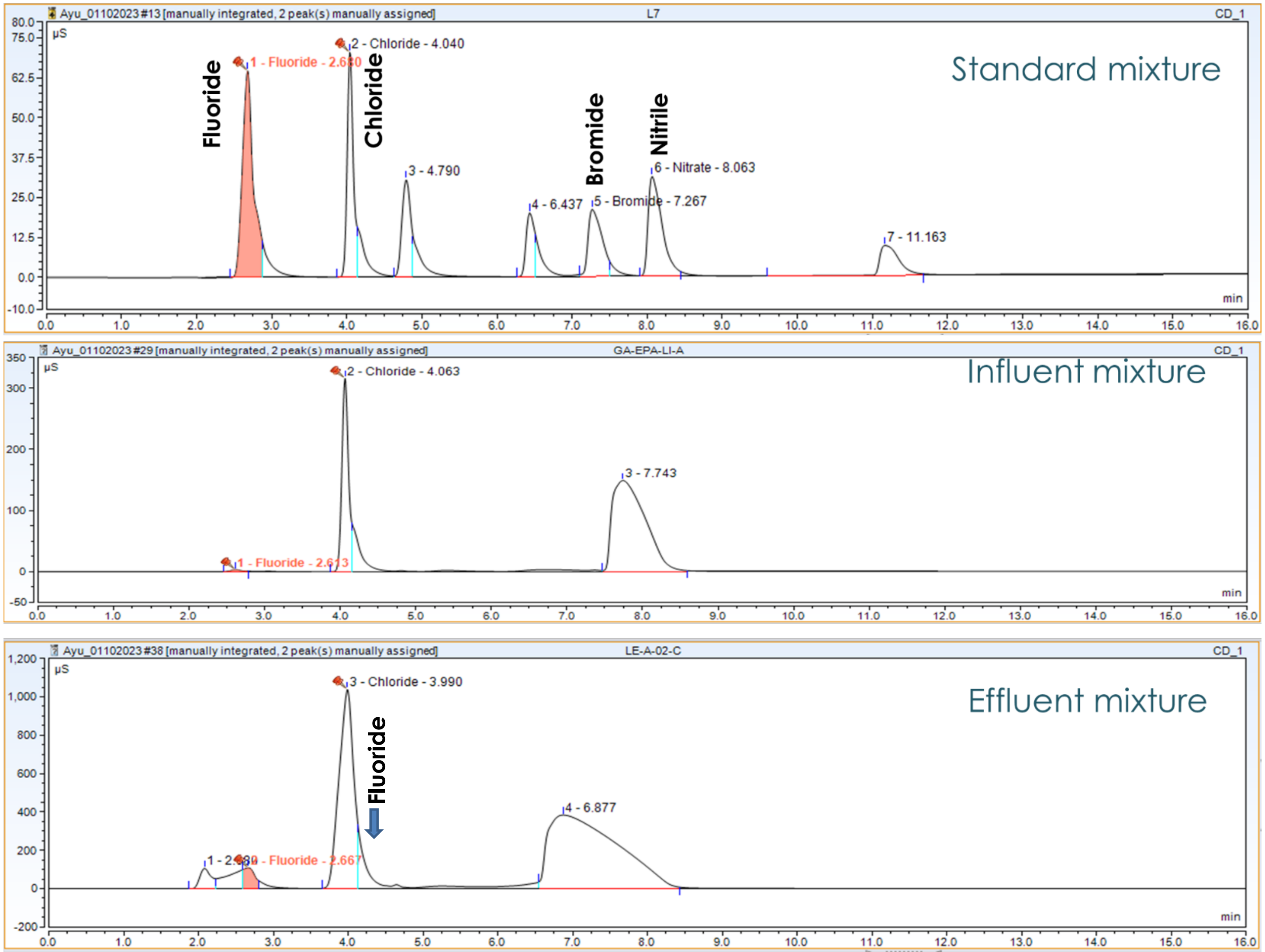

Figure 21: *Comparison of IC chromatograph of influent and effluent samples*

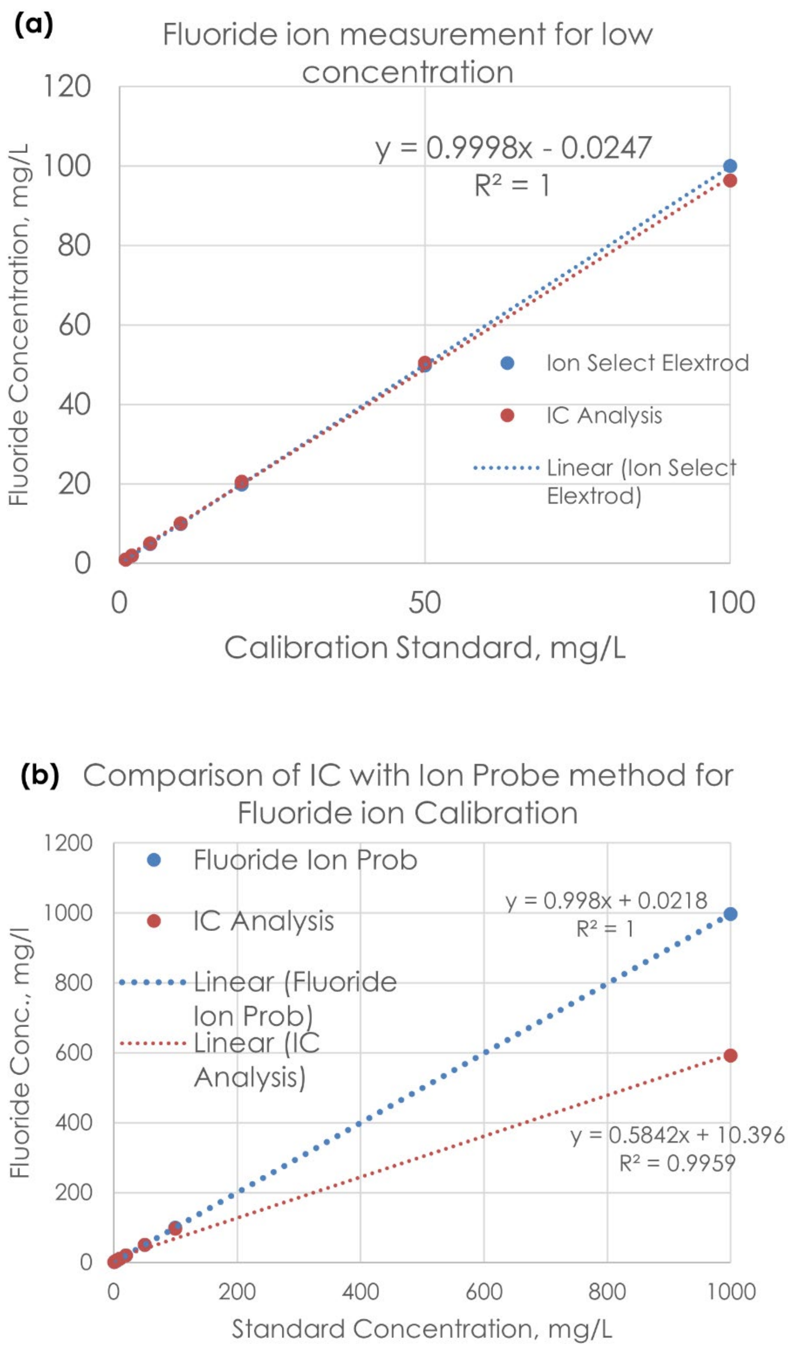

Figure 22: *Calibration of Fluoride Ion measurement using IC and F- ion prob,*

APPENDIX II

The following sample labeling was used for different gas and liquid samples that were stored and labeled with a completed chain of custody (COC) form as per the information contained here and in the Quality Assurance Project Plan (QAPP). There nine (9) types of samples were generated in this project:

1. MC: materials characterization (MC) samples of the AFFF material
2. LI: samples of the liquid influent (LI) into the iSCWO process
3. LE: samples of the liquid effluent (LE) from the iSCWO process
4. LFB: Liquid field blank (LFB) samples (PFAS-free water used to check sample containers)
5. LEB: Liquid equipment blanks (LEB) samples (PFAS-free used water to check sampling gear)
6. GC: Samples of the gas taken by evacuated canister from the iSCWO process
7. GCB: Field blanks for canisters
8. GS: Samples of the gas taken by sorbent approach from the iSCWO process
9. GSB: Field blanks for the sorbent approach

Each sample acquired had a unique sample code that was recorded on the media container label in the COC documentation and on field forms and lab notebooks as needed. The sample code starts with “EPA-GA”, identifying the test.

This is followed by the sample type from the above list. Next in the labeling sequence is the test run designation (e.g., Test A, B, or C) or an “NA” for the MC samples since the test designation is not applicable. The next label entry is the sequential sample number from that test (e.g., 2= second liquid sample from that Test run). The sample code sequence concludes with a “TA” or “EPA” at the end, indicating if TestAmerica or EPA will analyze the sample. For Liquid samples, TA is required to produce a backup sample for each field sample acquired, marked by a final “B”.

6.1 Peer Reviewers

This report was peer reviewed by the following external reviewers:

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