FINAL REPORT

Field Demonstration of Infrared Thermal Treatment of PFAS-impacted Soils from Subsurface Investigations

SERDP Project ER18-1603

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14. ABSTRACT

This study evaluated infrared heating in a small, mobile thermal desorption unit to separate per- and polyfluoroalkyl substances (PFAS) from soil. Indirect infrared heating relies on radiant heating and does not require the production or circulation of a carrier gas to heat the soil. Heated gasses were piped through impacted soil. Once gasses cooled, the PFAS were treated on vapor-phase activated carbon (VGAC). The VGAC was returned to the vender for regeneration and PFAS in the VGAC was destroyed during regeneration. Vapors were monitored using C-18 vapor adsorption cartridges. The test demonstrated that thermal desorption at a temperature above 350°C is a viable technology for removing PFAS from soil. The removal process is temperature dependent and does not work if the temperature is too low. The test also indicated that VGAC is not suited for treating the air stream from this process. To use VGAC, the vapors must be cooled and dried, and the PFAS appears to drop out the vapor phase in this process. This means that a treatment process for the PFAS needs to maintain the temperature of the gasses throughout the process to keep the PFAS mobile.

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ABSTRACT

Introduction and Objectives: This study evaluated infrared heating in a small, mobile thermal desorption unit (TDU) to separate Per- and Polyfluoroalkyl Substances (PFAS) from soil. Indirect infrared heating relies on radiant heating and does not require the production or circulation of a carrier gas to heat the soil. Heated gasses were piped through impacted soil. Once gasses cooled, the PFAS were treated on vapor-phase activated carbon (VGAC). The VGAC was returned to the vender for regeneration and PFAS in the VGAC was destroyed during regeneration. Vapors were monitored using C-18 vapor adsorption cartridges (C-18 cartridges). Project objectives were (1) demonstrate adequate removal of PFAS, such that the soil could be re-used. Reuse was based on meeting Alaska Department of Environmental Conservation soil to groundwater criteria of 1.7 micrograms per kilogram (μ g/kg) of perfluorooctanoic acid (PFOA) and 3.0 μ g/kg of perfluorooctane sulfonic acid (PFOS), (2) demonstrate capture of the PFAS on the VGAC, (3) complete mass balance that accounts for at least 75 percent of the PFAS mass in the treated soil.

Technical Approach: The technology was applied in a small, mobile unit designed to be transported to and used at remote locations. Propane fuel was used to heat vapors in the TDU that were then cooled in a condenser, treated using VGAC, and returned to the TDU. Two soil batches were treated, a Low Concentration Test [PFAS 516 μg/kg, PFOS 429 μg/kg, and PFOA 5.55 μg/kg] and a High-Concentration Test [Total PFAS 2,244 μg/kg, PFOS 1,800 μg/kg and PFOA 22.6 μg/kg]. The soil was heated to over 350 degrees Celsius (°C) to mobilize the PFAS. PFAS concentrations in soil were tracked using daily composite samples. The amounts of materials used and concentrations of PFAS were used to construct a PFAS mass balance to determine capture efficiency and understand the fate of PFAS in the process.

Results: The Low-Concentration Test met temperature requirements (greater than 350°C) and virtually all PFAS was removed by Day 5, with traces remaining at Day 8 (maximum detection of 2.96 µg/kg PFOS, total PFAS of 3.53 µg/kg, 99.6 percent removal of total PFAS). The High-Concentration Test did not meet the temperature requirements (sustained temperatures ranged from 307°C to 346°C) and removal of PFAS was poor. PFOA, a minor constituent, was wellremoved (greater than 98.1 percent), but PFOS was only removed to 68.6 percent and the total PFAS removal was 78.2 percent. Capture on VGAC was less than 1 percent of the total mass for either test, with similar masses of PFAS captured in the condensate and VGAC. C-18 cartridge samples suggest most of the PFAS was not present in the vapor stream before the VGAC in either test, with much smaller than expected amounts detected and only the most volatile compound (perfluorobutanoic acid) detected. The mass balance only accounted for 2 percent of the PFAS in the Low-Concentration Test and 36 percent of the High-Concentration Test. However, the vast majority of the PFAS captured in the High-Concentration Test was the 22 percent retained in the test soil. Based on the layout of the system and where PFAS was and was not observed, we believe the PFAS traveled to the condenser and was retained on the condenser surfaces. This is not yet confirmed by sampling.

Recirculating the vapors appeared to work well. No technical problems were reported. Tracking the moisture in the soil indicated we were able to account for 59 to 83 percent of the soil moisture, suggesting there were not large losses from the system due to fugitive emissions. The loss of PFAS mass is likely due to cooling the vapor stream.

Benefits: The test has a number of positive outcomes. First, the Low-Concentration Test demonstrated that thermal desorption at a temperature above 350°C is a viable technology for removing PFAS from soil. The removal process is temperature dependent and does not work if the temperature is too low. The test also indicated that VGAC is not suited for treating the air stream from this process. To use VGAC, the vapors must be cooled and dried, and the PFAS appears to drop out the vapor phase in this process. This means that a treatment process for the PFAS needs to maintain the temperature of the gasses throughout the process to keep the PFAS mobile. However, under the test conditions, a wet scrubber process is recommended for PFAS removal.

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ABBREVIATIONS AND ACRONYMS

ADEC Alaska Department of Environmental Control

AFFF aqueous film-forming foam
Battelle Battelle Memorial Institute

CH2M HILL, Inc.

CSM Colorado School of Mines

DoD Department of Defense

EIS extracted internal standards ESI+ electrospray ionization+

FtS fluorotelomer sulfate

GAC granular activated carbon

IDW investigation-derived waste Iron Creek Group Holdings, Inc.

LC/MS/MS liquid chromatography—mass spectrometry/mass spectrometry

M moles

MRM multiple reaction monitoring MTGW migration to groundwater

NAS Naval Air Station

NH₃ ammonia

NH₄OH ammonium hydroxide

PAH polynuclear aromatic hydrocarbon PFAS Per- and Polyfluoroalkyl Substances

PFBS perfluorobutane sulfonate PFCA perfluorinated carboxylic acid

PFOA perfluorooctanoic acid PFOS perfluorooctane sulfonate PFSA perfluorinated sulfonic acids

pg picograms

QA quality assurance

rcf relative centrifugal force RSL regional screening level

SERDP Strategic Environmental Research and Development Program

TDU thermal desorption unit

TOPA Total Oxidizable Precursor Assay

TPH-GRO total petroleum hydrocarbons gasoline-range organics

UPLC-MS/MS liquid chromatograph coupled to triple quadrupole mass spectrometer

USEPA U.S. Environmental Protection Agency

VGAC vapor-phase granular activated carbon VOC volatile organic compound

ABBREVIATIONS FOR UNITS OF MEASURE/TIME

°C degree(s) Celsius cm centimeter(s)

μg/kg microgram(s) per kilogram

 $\begin{array}{ll} \mu L & \text{microliter} \\ \mu m & \text{micrometer} \\ g & \text{gram}(s) \\ mg & \text{milligram}(s) \end{array}$

mg/kg milligram per kilogram mg/kg milligram(s) per kilogram

mL milliliter(s)
Mm millimeters

ng/L nanogram(s) per liter

oz. ounce

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EXECUTIVE SUMMARY

INTRODUCTION

Per- and Polyfluoroalkyl Substances (PFAS) are fully fluorinated synthetic organic chemicals that have been used extensively in surface coatings and protectant formulations for packaging products, carpets, leather products, and textiles. PFAS in aqueous film-forming foam (AFFF) are particularly effective in extinguishing hydrocarbon- or solvent-fueled fires, resulting in a large quantity of perfluorinated compound-containing material potentially released to the environment during firefighting training at airports, refineries, chemical manufacturers, and Department of Defense (DoD) installations. Those compounds commonly encountered in water include perfluorinated carboxylic acids (PFCA) and perfluorinated sulfonic acids (PFSA). PFAS contaminants that are target compounds for this work include PFCA, PFSA, fluorotelomers, and a few more complicated compounds.

DoD investigations at sites with a history of AFFF usage indicate many sites will likely require further investigation and possibly remedial action (AFCEC, 2015). Investigation will generate substantial volumes of PFAS-impacted investigation-derived waste (IDW). In response to the Strategic Environmental Research and Development Program (SERDP) fiscal year 2018 Statement of Need Number ERSON-18-L1, Innovative Approaches for Treatment of Waste Derived from PFAS Subsurface Investigations, co-principal investigators from CH2M HILL, Inc. (CH2M) and Battelle Memorial Institute (Battelle), and our co-performer organizations (Iron Creek Group Holdings, Inc. [Iron Creek], SGS AXYS, and the Colorado School of Mines [CSM]), propose to demonstrate the infrared thermal treatment of IDW soil contaminated with PFAS.

The team created two synthetic IDW batches by blending contaminated soil from former Naval Air Station (NAS) Willow Grove with local soil and treating two batches (up to 10 tons). One batch was supplemented with AFFF to supplement concentrations. The testing focused on perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA), because there are regulatory limits for these compounds in soil.

The treatment system is a transportable unit that can be loaded on a flat-bed truck using a forklift. The treatment system was setup in the parking lot at Iron Creek's Estacada, Oregon, fabrication shop, and the synthetic IDW was treated in two batches, each during a 1-week pilot test period. Samples of soil batches, wipe samples from the treatment chamber, vapor cartridges, condensate, and vapor-phase granular activated carbon (VGAC) were collected and analyzed to demonstrate the effectiveness of the treatment process (thermal desorption) and fate of PFAS in the treatment system. The results from the study will be used to optimize design and operation of a mobile treatment system to efficiently and cost-effectively treat PFAS-contaminated soil IDW.

1.1 HISTORY

1.2 REGULATORY ENVIRONMENT

Regulation of PFAS is a recent development and the regulatory environment is rapidly evolving. The USEPA has published tap water advisories for PFOS and PFOA of 70 nanograms per liter (ng/L) (USEPA, 2016A) (USEPA, 2016B). Alaska regulates a migration to groundwater (MTGW) pathway for PFOS and PFOA (Division of Spill Prevention and Response- Contaminated Sites

Program, August 25, 2018), which provides a restrictive set of limits (0.0030 milligrams per kilograms [mg/kg] PFOS and 0.0017 mg/kg PFOA) on soil contamination the is thought to be suitable for re-use (lower than health based standards and protective of groundwater).

1.3 THERMAL DESORPTION TREATMENT

The infrared treatment module indirectly heats contaminated materials that are stored in a reduced-oxygen or oxygen-free environment to minimize oxidation of the contaminated vapor. By indirectly heating the contaminated material, the control of the flow of heat applied to the contaminated material is improved and loss of heating air or other heating gas is reduced. This results in energy-efficient heating of the soil. Objectives

1.4 Performance Objectives

The objectives of this pilot study are as follows:

- 1) Evaluate whether the infrared thermal treatment technology (desorption) can effectively treat PFAS-contaminated soil IDW such that it allows soil re-use.
- 2) Provide documentation of the fate of PFAS in the treatment process.
- 3) Demonstrate that the treated PFAS in the vapors can be captured.

MATERIALS AND METHODS

1.5 TEST SOIL

Test soil was obtained from Former NAS Joint Reserve Base Willow Grove, Building 608, the former Base fire station. The site was being excavated at the time of this study and 4 drums of soil were collected for this study by the remediation contractor.

PFAS results indicate predominantly PFOS (1,227 $\mu g/kg$) in the composite soil sample, with significant concentrations of PFHxS (198.5 $\mu g/kg$) 6:2 fluorotelomer sulfates (FtS) (59.58 $\mu g/kg$), and PFOA (22.82 $\mu g/kg$). Other compounds were detected at less than 20 $\mu g/kg$. The total PFAS concentration was 1,585 $\mu g/kg$. In the post-oxidation soil (TOPA results), the total PFAS was reduced slightly to 1,452 $\mu g/kg$, with PFOS reduced to 960 $\mu g/kg$ (21 percent reduction in concentration) and minor increases in PFCAs.

Two batches of soil were created for treatment, a batch of Willow Grove soil diluted with clean local soil (the Low-Concentration test (Low C test) soil) and a batch containing Willow Grove soil, clean local soil and AFFF (the High-Concentration Test (High C test) soil.

1.6 TEST EQUIPMENT AND OPERATION

The thermal desorption unit (TDU) was operated by heating the air in the treatment bin using propane-fuel infrared heaters. As the vapors exited the treatment bin, they were circulated through a condenser, and then through an vapor-phase granular activated carbon (VGAC) unit. Condensate accumulated in high-density polyethylene drums. Condensate from each batch was tested and the condensate was treated to remove PFAS using granular activated carbon (GAC). The used GAC and VGAC were returned to the vender for regeneration. Vapor was sampled using C-18 cartridges in ports located immediately before the VGAC.

1.7 FIELD TESTING METHODS

Two soil batches were created for the tests. The low-concentration batch included 8,500 pounds of clean fill and 1.5 drums of Willow Grove soil. No AFFF was added to this batch. The high-concentration batch included 7,800 pounds of clean fill and to 2.5 drums of Willow Grove soil, supplemented with 150 mL of electrochemical process AFFF.

During the Low-Concentration Test and High-Concentration Test, pre-, in-, and post-treatment soil samples (five grabs per sample) were collected from the treatment bin, composited, and split into two composite samples. The Colorado School of Mines (CSM) and SGS AXYS (Sidney, British Columbia) analyzed these samples for PFAS. Soil samples were composite samples constructed of five roughly 10-ounce (oz.) grabs of soil. During the run, Days 1 through 8, samples were collected through the sampling ports installed in the TDU.

Wipe samples were collected from the inside of the TDU, outside the bin, before it was loaded with soil and at the completion of each test. The same locations were used for all wipe samples. C-18 cartridges were attached to a side-stream of the vapor. The C-18 cartridges were swapped out daily during treatment to evaluate PFAS desorption over time.

The test used two VGAC units. The units were swapped between tests to estimate what was adsorbed during each test. Before testing and after each test was completed, a composite sample was collected from the VGAC unit using the same method as described for soil samples Condensate was collected from the condensate tank using a spigot installed in the tank. Condensate samples were collected at the end of each run.

Soil samples were analyzed for PFAS and TOPA by SGS AXYS Analytical Services using two analytical approaches. SGS AXYS method MLA-110 was used to determine the concentrations of 29 targeted PFAS and SGS AXYS Method MLA-111 was used measure perfluorinated carboxylates and sulfonates after a TOPA reaction (Houtz and Sedlak 2012) to estimate presence of oxidizable perfluoroalkyl acid (PFAA) precursors in the samples.

The CSM performed PFAS analysis by liquid chromatography Quadrupole Time of Flight Mass Spectrometry (SCIEX X500R).

Battelle Laboratory performed analysis of PFAS using liquid chromatography tandem mass spectrometry in the MRM. Analysis included solid samples, water samples and C-18 cartridges.

RESULTS AND EVALUATION

1.8 OBJECTIVE 1: TREAT PFAS-CONTAMINATED SOIL

Results indicate that the average target temperature of 350°C was met by Day 4 of the Low-Concentration Test and maintained over the test. No PAHs were detected in any of the post-treatment samples indicating that thermal desorption effectively treats PAHs where present as co-contaminants with PFAS.

Based on the ELAP-certified analysis by SGS AXYS, all compounds were removed to less than the detections limits except PFHxS ($0.126~\mu g/kg$) and PFOS ($0.703~\mu g/kg$). Removal of PFOA was to below the detection limit and exceeded 97.4 percent. Removal of PFOS was 99.6 percent and removal of total PFAS was also 99.6 percent. Results from TOPA and the CSM confirmed effective treatment. The data shows that most of the compounds and post-oxidation compounds were removed from soil in 4 days when the average temperature of the test soil was heated to the

target temperature of 350°C. The re-use criteria were met for both PFOA and PFOS.

High concentration results indicate that the average target temperature (350°C) was not met during the High-Concentration Test. Sensors 1 and 2 reached the target temperature on Days 3 through 8 and Day 7, respectively, indicating partial treatment. No PAHs were detected in any of the post-treatment samples, indicating that thermal desorption effectively treats PAHs where present as co-contaminants with PFAS even though the target temperature was not met.

Total PFAS concentrations reported by SGS AXYS, post-oxidation concentrations reported by SGS AXYS, and total PFAS concentrations reported by CSM during the High-Concentration Test indicated partial treatment of the soil. Based on the ELAP-certified analysis by SGS AXYS, 14 PFAS were detected in the post-treatment sample at concentrations from 0.17 µg/kg to 326 µg/kg. Removal of PFOS was 74.0 percent and removal of total PFAS was 73.0 percent. Results for TOPA and the CSM analysis were similar. The data shows that the PFCA were removed better than the PFSA and that the PFOA concentration was reduced to below the ADEC criteria. PFOS concentrations initially decreased, then decreased and stabilized for the last 3 days at concentrations above out goal.

1.8.1 Conclusions

PFAS removal from soil met ADEC soil to groundwater criteria and the treatment goal for the Low-Concentration Test where the temperature was maintained at or above the target temperature of 350°C. PFAS removal in the High-Concentration Test did not meet treatment goals. In this test, the temperature of the treatment unit did not meet the planned temperature. The result was 73 percent removal of PFAS, a similar removal of PFOS, and reduction of only PFOA to less than the ADEC soil to groundwater criteria.

1.9 OBJECTIVE 2: IMPROVE UNDERSTANDING OF PFAS IN THE TREATMENT PROCESS

1.9.1 PFAS Mass Balance

In this section, data collected during the tests is used to evaluate the thermal desorption and vapor treatment processes. This includes presenting the mass balance and the water balance and results. Sample collected during these tests and the amounts of materials used or recovered during the test were used to conduct the mass balance evaluation. In addition, a water balance was conducted to evaluate the amount of the initial soil moisture recovered in the system.

For the low concentration test, an estimated 83 percent of the water in the test soil was recovered. This indicates the system was capturing most of the vapors generated during the test. The mass balance of the system indicated only 2 percent of the total mass present in the soil before the test (0.037 g) was recovered. Most of the recovered mass was in the VGAC (0.019 g) and residual soil (0.014 g) with lesser amount in the condensate (0.003 g). The amount on the interior surface of the TDU was insignificant.

While the treated soil in the Low-Concentration Test met its treatment goal, we were not able to quantify recover sufficiently for a successful mass balance. Our goal of accounting for 75 percent of mass in the for the mass balance was not achieved.

For the high concentration test, 59 percent of the moisture in the system was captured. The mass

balance indicated 36 percent of the total mass present in the soil before the test was recovered, although most of the "recovered" PFAS mass (3.31 g) was retained in the partially treated soil. As with the Low-Concentration Test, recovery of PFAS was far less than the goal of 75 percent and this objective was not met.

1.9.2 Comparison of Total to TOPA Analyses

Post-oxidation results were slightly higher than results from standard analyses, but the increases were not dramatic. The increase in mass was primarily due to an increase in the PFCA post-oxidation.

1.9.3 Treatment by VGAC

The amount of PFAS recovered in the VGAC, as presented in the mass balance, was very small, with 0.025 g of 2.11 g (1.1 percent) recovered in the Low-Concentration Test and 0.002 of 9.17 g (0.02 percent) recovered in the High-Concentration Test (Table 4.14). One objective of the study was to capture 90 percent of the PFAS on the GAC, which did not occur. One explanation for the low recovery of PFAS in the VGAC is the PFAS did not made it to the VGAC.

PFAS was recovered in the condensate, with relatively high concentrations detected in both the Low- and High-Concentration Test samples. The amount of PFAS measured in the condensate implies that significant PFAS made it to the condenser. Very little PFAS was detected in the C-18 cartridges, which confirms that little of the PFAS made it to the VGAC.

1.9.4 Conclusions

The mass balance found far less PFAS than anticipated in treatment residuals. It was anticipated that most of the mobilized PFAS would be found in the VGAC, with a smaller amount in the condensate. Instead, small amounts were found in both the VGAC and condensate. The recovery of PFAS in condensate, VGAC, C-18 cartridges, and wipe samples was less than 3 percent, with 2.1 percent recovered from the Low-Concentration Test and less than 1 percent recovered from the High-Concentration Test. The lack of PFAS detected in vapor confirms that PFAS typically did not make it to the VGAC or the cartridge sampling port. Instead, most of the PFAS appears to have condensed in the condenser, or perhaps in the connecting hose. PFAS does not form a classical liquid when condensed but rather a waxy coating. It appears that a portion of the condensed PFAS dissolved in the condenser liquid, most of the rest likely remains in the condenser, and small portion made it to the VGAC, where it was removed.

1.10 OBJECTIVE 3: EVALUATE VAPOR-PHASE TREATMENT

Very little PFAS was sorbed onto the VGAC. In the Low-Concentration Test, only 1 percent of the initial soil mass was in the VGAC. In the High-Concentration Test, PFAS in VGAC made up less than 0.001 percent of the total mass in the residuals. Only PFBA was detected on the C-18 cartridges (Tables 4.13 and 4.15). VGAC does not appear to be relevant for PFAS treatment in vapors because the PFAS does not appear to stay in the vapor phase.

2.0 CONCLUSIONS AND IMPLICATIONS FOR FUTURE RESEARCH

The objectives of this pilot study are as follows:

- 1) To evaluate whether the infrared thermal treatment technology can effectively treat PFAS-contaminated soil IDW such that it allows soil re-use,
- 2) To provide documentation of the fate of PFAS in the treatment process, and
- 3) Demonstrate that the treated PFAS in the vapors can be captured.

2.1 CONCLUSIONS

The testing confirms that when properly implemented thermal desorption is capable of fully treating PFAS contaminated soil when temperatures are maintained above 350°C. In the Low-Concentration Test, where the proper temperature was maintained, effective PFAS treatment was achieved in 4 days. Treatment time seems more a function of the ability to dry the soil and achieve treatment temperature than the amount of PFAS present.

PFAS recovery was very low, with a few percent of the PFAS mass in the sample retained in the VGAC and condensate. Because excellent treatment was achieved in the Low-Concentration Test and vapors were recycled into the treatment unit, it is unlikely that PFAS passed through the GAC without treatment. Analysis of condensate samples found high concentrations of PFAS, which suggest PFAS made it to the condenser. Taken together, this suggests the PFAS condensed in the condenser. It is likely the PFAS is still in the condenser, attached to the condenser surfaces.

VGAC does not appear to be a good choice for primary PFAS treatment in the vapor phase; it appears that a high temperature must be maintained to keep the PFAS mobile in an air stream. Where test results indicate that some PFAS remained in the air stream, VGAC appeared to effectively remove this residual.

2.2 **RECOMMENDATIONS**

Because the High-Temperature Test soil was not fully treated since the target temperature was not obtained, the study team proposes to re-treat and test the High-Concentration soil before disposal. The proper temperature will be maintained during treatment. This cost will be borne by the project.

To evaluate whether treatment residuals were retained on the interior of the condensation tank, a wipe sample will be collected from the interior of the condenser. This sample will be tested for PFAS. This cost will be borne by the project.

Additional testing is recommended to better evaluate the fate of residuals in the treatment process. A batch similar to the High-Concentration Test could be treated. The test should ensure that the proper temperature is maintained. Because the study confirmed that PFAS can be removed from soil using thermal desorption, limited soil testing would be needed. Instead, treatment residuals should be tested to better understand the fate of the treated PFAS. A series of wipe tests are recommended at multiple locations along the treatment train to evaluate the location of residual PFAS. A mass balance would be used to quantify the recovery of PFAS.

Because the study determined that capture of PFAS from vapor is not an ideal solution for thermal desorption, the condenser system should be replaced with a wet scrubber, with GAC used to treat the scrubber water. Tests would be conducted to assess where residual PFAS remains in the treatment equipment and then remove the residual PFAS from the treatment equipment, to better manage the residuals.

1.0 INTRODUCTION

Per- and Polyfluoroalkyl Substances (PFAS) are fully fluorinated synthetic organic chemicals whose unique water-, oil-, and dirt-repelling properties make them commercially valuable (Kissa, 2001). Because of these properties, they have been used extensively in surface coatings and protectant formulations for paper and cardboard packaging products, carpets, leather products, and textiles. PFAS have been identified as being a component of mist-suppressants in chromium electroplating shops and as surfactants in well drilling/fracking fluids. PFAS in aqueous film-forming foam (AFFF) are particularly effective in extinguishing hydrocarbon- or solvent-fueled fires, resulting in a large quantity of perfluorinated compound-containing material potentially released to the environment during firefighting training at airports, refineries, chemical manufacturers, and Department of Defense (DoD) installations.

While there are thousands of PFAS compounds in industrial use, those commonly encountered in water contamination include perfluorinated carboxylic acids (PFCA) and perfluorinated sulfonic acids (PFSA). These compounds are often the bases of larger molecules, very stable in the natural environment, and are either released directly to the environment or are present as residuals of other compounds degrading. Fluorotelomers are polyfluorinated compounds that are also used as the bases for industrial compounds. While fluorotelomers are not as stable as PFCA and PFSA, some are stable and can be detected as contaminants. Table 1.1 lists common PFAS contaminants that are target compounds for this work. The list includes PFCA, PFSA, fluorotelomers, and a few more complicated compounds that all play a significant role in environmental contamination.

Table 1.1. List of Target Compounds

Compound Acronym	Compound Name		
PFBA	perfluorobuane carboxylic acid		
PFPeA	perfluoropentane carboxylic acid		
PFHxA	perfluorohexane carboxylic acid		
PFHpA	perfluoroheptane carboxylic acid		
PFOA	perfluorooctane carboxylic acid		
PFNA	perfluorononane carboxylic acid		
PFDA	perfluorodecane carboxylic acid		
PFUnA	perfluoroundecanoic acid		
PFDoA	perfluorododecanoic acid		
PFTrDA	perfluorotridecanoic acid		
PFTeDA	perfluorotetradecanoic acid		
PFBS	perfluorobutane sulfonic acid		
PFPeS	perfluoropentane sulfonic acid		
PFHxS	perfluorohexane sulfonic acid		
PFHpS	perfluoroheptane sulfonic acid		
PFOS	perfluorooctane sulfonic acid		
PFNS	perfluorononane sulfonic acid		
PFDS	perfluorodecane sulfonic acid		

Compound Acronym	Compound Name		
PFDoS	perfluorododecanesulfonic acid		
4:2 FTS	4:2 fluorotelomer sulfonic acid		
6:2 FTS	6:2 fluorotelomer sulfonic acid		
8:2 FTS	8:2 fluorotelomer sulfonic acid		
PFOSA	perfluorooctanesulfonamide		
N-MEFOSAA	N-methyl perfluorooctane sulfonamido		
N-ETFOSAA	N-ethyl perfluorooctane sulfonamido		
N-MEFOSE	N-methyl perfluorooctane sulfonamidoethanol		
N-ETFOSE	N-ethyl perfluorooctane sulfonamidoethanol		

DoD investigations at sites with a history of AFFF usage indicate many sites will likely require further investigation and possibly remedial action in the coming years (AFCEC, 2015). Investigation alone will generate substantial volumes of PFAS-impacted investigation-derived waste (IDW) from subsurface investigations. In response to the Strategic Environmental Research and Development Program (SERDP) fiscal year 2018 Statement of Need Number ERSON-18-L1, Innovative Approaches for Treatment of Waste Derived from PFAS Subsurface Investigations, co-principal investigators from CH2M HILL, Inc. (CH2M) and Battelle Memorial Institute (Battelle), and our co-performer organizations (Iron Creek Group Holdings, Inc. [Iron Creek], SGS AXYS, and the Colorado School of Mines [CSM]), propose to demonstrate the infrared thermal treatment of IDW soil contaminated with PFAS.

The team identified the former Naval Air Station (NAS) Willow Grove, near Horsham, Pennsylvania, as a source of PFAS-impacted soil. The team created two synthetic IDW batches by blending the contaminated soil removed as part of remediation activities with local soil and treating two batches (up to 10 tons). The testing focused on perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA), because there are regulatory limits for these compounds in soil. The testing also looked at other detectable PFAS compounds and precursor compounds that might degrade to form detectable compounds to evaluate the fate of PFAS in the treatment system.

The treatment system is a transportable unit that can be loaded on a flat-bed truck using a forklift. The system is purposefully designed to be transportable and energy efficient because Iron Creek specializes in treating contaminated soil at remote, hard-to-reach sites, in places like the Yukon Territory, Canada, and the Antarctic. The treatment system was setup in the parking lot at Iron Creek's Estacada, Oregon, fabrication shop, and the synthetic IDW was treated in Estacada in two batches, each during a 1-week pilot test period. Because it is already established that the system is transportable, treatment and handling was conducted in a parking lot to simulate site conditions. Samples of soil batches, wipe samples from the treatment chamber, vapor cartridges, condensate, and vapor-phase granular activated carbon (VGAC) were collected and analyzed to demonstrate the effectiveness of the treatment process (thermal desorption) and fate of PFAS in the treatment system. The results from the study will be used to optimize design and operation of a mobile treatment system to efficiently and cost-effectively treat PFAS-contaminated soil IDW.

1.1 HISTORY

1.2 REGULATORY ENVIRONMENT

Regulation of PFAS is a recent development and the regulatory environment is rapidly evolving. In 2009, the U.S. Environmental Protection Agency (USEPA) published a provisional health advisory for PFOA and PFOS in tap water (USEPA, 2009A) and risk-based concentrations for these two compounds in soil (USEPA, 2009B). Since then, USEPA has published tap water advisories for PFOS and PFOA of 70 nanograms per liter (ng/L) (USEPA, 2016A) (USEPA, 2016B). The advisories are based on potential adverse health effects, including developmental effects to fetuses or breastfed infants, cancer, liver effects, immune effects, and thyroid effects. Regional screening levels (RSLs), based on the available health data, are also published for perfluorobutane sulfonate (PFBS) (USEPA, 2016C). Several states have also moved to regulate PFAS in groundwater, including Alaska, New Hampshire, North Carolina, Michigan, and Vermont, where standards are promulgated, as well as Michigan and Texas, where advisory levels are published. Alaska regulates a migration to groundwater (MTGW) pathway for PFOS and PFOA (Division of Spill Prevention and Response- Contaminated Sites Program, August 25, 2018), which provides a much more restrictive set of limits (0.0030 milligrams per kilograms [mg/kg] PFOS and 0.0017 mg/kg PFOA) than the published soil guidance based on human health exposure (6 mg/kg PFOS and 16 mg/kg PFOA) (USEPA, 2009B). In general, regulation of these compounds is evolving, but the trend is toward concentrations in the low ng/L range in groundwater and corresponding concentrations in soil that are protective of groundwater.

DoD is currently proceeding with investigation of numerous PFAS-related locations; the U.S. Navy, U.S. Army, and U.S. Air Force are all actively identifying and investigating potential PFAS use locations and identifying sources of PFAS contamination in groundwater, while responding to cases where PFAS pose a threat to human health. Early results indicate PFAS contamination has been detected at virtually all locations where AFFF has been used (AFCEC, 2015). It is likely that delineation will eventually be required for all these locations, driving significant site investigation efforts, which will drive the production of PFAS-related IDW. This waste could pose a significant cost to DoD for disposal, and disposal of the contaminated material may present a liability. The ability to cost-effectively treat small quantities of contaminated solid waste onsite would help lower this cost and mitigate liability.

1.3 THERMAL DESORPTION TREATMENT

The infrared treatment module indirectly heats contaminated materials that are stored in a reducedoxygen or oxygen-free environment to minimize volatilization of the contaminated vapor. By indirectly heating the contaminated material, the control of the flow of heat applied to the contaminated material is improved and loss of heating air or other heating gas is reduced. These embodiments can operate at lower temperatures than conventional remediation or desorption systems as the efficiency savings and control of the flow of the heating agent enables longer durations of remediation without increasing the energy expended. A benefit of lower operating temperatures is easier permit or license acquisition to establish a remediation site, thereby greatly increasing the available locations for conducting remediation of contaminated material.

The infrared treatment module uses dual heating to indirectly heat the contaminated material by providing inside-out heating from the core heating conduit and outside-in heating from the space within the bin housing insulator. Energy efficiency is improved by preserving heat from the heated gas to efficiency ranges between 70 and 90 percent, as compared to 30 percent of currently

available systems. Additionally, injecting heated gas directly into contaminated material quickly dissipates the heat of the gas and precludes secondary use of the gas as a heating agent, requiring a constant stream of heated gas through a contaminated material to maintain appropriate temperatures. Avoiding this dissipation of heat correspondingly improves the efficiency of the system.

2.0 OBJECTIVES

2.1 Performance Objectives

The objectives of this pilot study are as follows:

- 1) Evaluate whether the infrared thermal treatment technology (desorption) can effectively treat PFAS-contaminated soil IDW such that it allows soil re-use.
- 2) Provide documentation of the fate of PFAS in the treatment process.
- 3) Demonstrate that the treated PFAS in the vapors can be captured.

2.1.1 Objective 1: Treat PFAS-Contaminated Soil to Allow for Re-Use

The first objective is to evaluate whether the infrared thermal desorption treatment technology can effectively treat PFAS-contaminated soil IDW such that it allows soil re-use. Although USEPA health-based RSLs for PFOA and PFOS are 16 mg/kg and 6 mg/kg, respectively, these criteria do not consider the potential for the soil to act as a source of groundwater contamination. The Alaska Department of Environmental Control (ADEC) has calculated, published, and promulgated MTGW standards for PFOA and PFOS of 0.0017 and 0.0030 mg/kg (1.7 and 3.0 micrograms per kilogram [μ g/kg]), respectively. Therefore, the ADEC criteria are used to define "suitable for reuse onsite." In reality, re-use is a complicated issue and would have to be addressed on a site-by-site basis.

2.1.1.1 Data Requirements

The technology effectiveness was evaluated based on post-treatment contaminant concentration reductions in soil relative to starting concentrations. Results from samples collected during testing were used to understand how remediation progressed. Data requirements include pre- and post-treatment contaminant concentrations in soil and concentrations collected during the test.

Post-treatment soil concentrations will be compared to initial concentrations and the ADEC MTGW to calculate the amount of mass removed and determine whether re-use criteria are met, respectively.

2.1.1.2 Success Criteria

The objective will be considered met if post-treatment concentrations in both soil batches meet the ADEC MTGW for PFOA and PFOS.

2.1.2 Objective 2: Improve Understanding of the Fate of PFAS in the Treatment Process

The second objective is to construct an effective mass balance of the treatment process. The study calculates a mass balance of PFAS throughout the treatment process using soil, carbon-18 (C-18) vapor sorption cartridges, VGAC, condensate, and wipe sample results. These data will evaluate

where the mass of PFAS removed from the soil batches is captured in the treatment process. Success of the study is dependent on how much of the starting mass of total PFAS is tracked during the study. The study's goal is 75 percent efficiency of the mass balance in tracking total PFAS.

PFAS treatment has proven difficult to demonstrate because of the tendency of PFAS to partition to unexpected compartments and for transformation to add and subtract PFAS from the detectable quantities. Precursors can degrade to form detectable compounds, which tends to increase concentrations of PFCAs and PFSAs. Changing physical and chemical conditions in the treatment technology can cause PFAS to shift compartments in unexpected ways. A 75-percent closure on a mass balance is adequate to demonstrate treatment while acknowledging uncertainties in the experiment.

2.1.2.1 <u>Data Requirements</u>

Data requirements include analysis for individual PFAS in this study. Analysis of the soil, pre- and post-treatment, allow an evaluation of the input of PFAS into the treatment system, including the presence of specific compounds and potential precursor compounds. Analysis of PFAS in condensate, vapor, treatment unit surfaces (wipe samples), and VGAC allow an evaluation of how PFAS is removed in the treatment system and retained in residuals. Wipe samples collected from the interior of the treatment system before and after each test help to understand how much PFAS is retained on the treatment equipment. Total Oxidizable Precursor Assay (TOPA) analysis provides a gross estimate of the total quantity of all PFAS in the system. The detailed analysis of PFAS will help understand the composition of the TOPA, and how the PFAS compounds in the system degrade and transform.

To calculate the various masses, the mass of soil, the surface area of the thermal desorption unit (TDU), the mass of VGAC and the amount of condensate produced were measured. These data and concentrations of PFAS as determined by analysis of samples were used to estimate total mass. Samples collected from each component of the treatment process included pre- and post-treatment soil, wipe samples from the walls of the TDU, condensate samples, and VGAC samples. The C-18 cartridges were used to collect PFAS samples to evaluate PFAS in the vapor stream.

2.1.2.2 Success Criteria

The objective is considered met if 75 percent of the initial mass is accounted for in the mass balance [(mass of total PFAS post treatment)/(mass of total PFAS in initial soil) = E > = 75 percent). A better understanding of the fate of PFAS during the treatment process benefits future treatment system designs and informs changes to temperature and duration of the tests to eventually determine optimal conditions that result in total mass removal or that maximize the amount of mass removed versus transformed. A better mass balance also allows a greater degree of certainty in the testing procedures.

Mass balance of PFAS throughout the treatment process is used to evaluate effectiveness of the treatment module and better understand the fate of PFAS in the treatment process.

2.1.3 Objective 3: Evaluate Vapor-Phase Treatment

The third objective is to document effective transfer of the PFAS to the VGAC. The effectiveness of the study is a function of the degree to which the treatment module removes PFAS from post-

condensate vapor. Success depends on the mass of PFAS in the soil and the mass of PFAS transferred to the VGAC.

2.1.3.1 <u>Data Requirements</u>

The technology effectiveness evaluates the amount of PFAS entering the VGAC system, and the efficiency of capture in the VGAC system. Data required to evaluate the effectiveness of the module are the C-18 cartridge vapor samples collected through side-streams and the VGAC samples.

2.1.3.2 Success Criteria

The objective is considered met if 90 percent of the mass of PFOA and PFOS detected in the soil is transferred to the VGAC during treatment.

3.0 MATERIALS AND METHODS

3.1 TEST SOIL

Test soil was obtained from Former NAS Joint Reserve Base Willow Grove, historically a private airfield that began operation in 1926. The area of Willow Grove where PFAS was identified in soil is Building 608, the former Base fire station. Constructed in 1989, the fire station housed firefighting equipment, supplies, and personnel, including AFFF, which is likely the source of much of the contamination found at the location. The building is still standing and has been considered part of the redevelopment plan for the facility (RKG Associates, March 2012).

For this demonstration, a baseline soil sample was collected from a four-drum bulk soil sample collected at NAS Willow Grove. The sample was analyzed for PFAS, volatile organic compounds (VOCs), polynuclear aromatic hydrocarbons (PAHs), and total petroleum hydrocarbons gasoline-range organics (TPH-GRO). Low-levels of PAHs were detected (Table 3.1). TPH-GRO and VOCs were not detected above the limit of detection. PFAS results are presented in Table 3.2. Laboratory analytical reports are presented in the Demonstration Plan (Appendix A).

Table 3.1. VOCs, PAHs and TPH-GRO Detected in Willow Grove Bulk Soil Sample

Analyte	Result	Qual	LOD	Units
VOCs by EPA Method SW846 8260B				
Methylene Chloride	12.9	J	17	μg/kg
PAHs by EPA Method SW846 8270D BY SIM				
Acenaphthylene	51.8	J	38	μg/kg
Anthracene	52.5	J	38	μg/kg
Benzo(a)anthracene	247		7.7	μg/kg
Benzo(a)pyrene	197		7.7	μg/kg
Benzo(b)fluoranthene	193		7.7	μg/kg
Benzo(g,h,i)perylene	81.8		7.7	μg/kg
Benzo(k)fluoranthene	188		7.7	μg/kg
Chrysene	235		7.7	μg/kg
Dibenzo(a,h)anthracene	24.3		7.7	μg/kg

Analyte	Result	Qual	LOD	Units
Fluoranthene	467		38	μg/kg
Indeno (1,2,3-cd) pyrene	115		7.7	μg/kg
Phenanthrene	196		38	μg/kg
Pyrene	336		38	μg/kg
TPH-GRO by EPA Method SW846 8015C				
TPH (C10-C28)	5.25	J	5.8	mg/kg

Notes:

EPA = U.S. Environmental Protection Agency

J = estimated

μg/kg = micrograms per kilogram

mg/kg = milligrams per kilogram

LOD = limit of detection

PAH = polycyclic aromatic hydrocarbon

TPH-GRO = total petroleum hydrocarbons – gasoline-range organics

VOC = volatile organic compound

Table 3.2. Pre- and Post- TOPA PFAS in Willow Grove Bulk Soil Sample

Compound	Soil Concentration (µg/kg)	LOD/DL (µg/kg)	Post-Oxidation Concentration (μg/kg)	Reporting Limit (µg/kg)
PFBA	U	3.23 (LOD)	13.8	6.99
PFPeA	4.13	0.784 (DL)	33.5	3.5
PFHxA	17.1	0.402 (DL)	187	1.75
PFHpA	3.77	0.402 (DL)	8.29	1.75
PFOA	22.8	0.402 (DL)	21.1	1.75
PFNA	2.24	0.392 (DL)	3.46	1.49
PFDA	0.537	0.0.392 (DL)	U	1.4
PFUnA	U	0.784 (LOD)	U	1.4
PFDoA	U	0.784 (LOD)	U	1.4
PFTrDA	U	1.96 (LOD)	U	1.4
PFTeDA	U	1.96 (LOD)	U	1.4
PFBS	1.98	0.397 (DL)	U	1.75
PFPeS	8.52	0.367 (DL)	9.19	1.75
PFHxS	198	0.402 (DL)	200	1.75
PFHpS	32.4	0.372 (DL)	15.5	1.75
PFOS	1,230	0.402 (DL)	960	1.75
PFNS	1.84	0.377 (DL)	U	1.75
PFDS	U	0.784 (LOD)	U	1.75
PFDoS	U	1.86 (LOD)	U	1.75
Total PFAS	1,523		1,452	
4:2 FTS	U	3.13 (LOD)	NA	-

Compound	Soil Concentration (µg/kg)	LOD/DL (µg/kg)	Post-Oxidation Concentration (μg/kg)	Reporting Limit (µg/kg)
6:2 FTS	59.6	1.57 (DL)	NA	-
8:2 FTS	5.33	1.57 (DL)	NA	-
PFOSA	0.719	0.402 (DL)	NA	-
N-MEFOSA	U	2.25 (LOD)	NA	-
N-ETFOSA	U	4.90 (LOD)	NA	-
MEFOSAA	U	0.784 (LOD)	NA	-
ETFOSAA	U	0.784 (LOD)	NA	-
N-MEFOSE	U	20.0 (LOD)	NA	-
N-ETFOSE	U	15.0 (LOD)	NA	-

Notes:

DL = detection limit

FTS = Fluorotelomer sulfates

LOD = limit of detection

μg/kg = micrograms per kilogram

NA = not applicable

U = undetected

PFAS results indicate predominantly PFOS (1,227 μg/kg) in the composite soil sample, with significant concentrations of PFHxS (198.5 μg/kg) 6:2 fluorotelomer sulfates (FtS) (59.58 μg/kg), and PFOA (22.82 μg/kg). Other compounds were detected at less than 20 μg/kg. The total PFAS concentration was 1,585 μg/kg. In the post-oxidation soil (TOPA results), the total PFAS was reduced slightly to 1,452 μg/kg, with PFOS reduced to 960 μg/kg (21 percent reduction in concentration) and minor increases in PFCAs. These results suggest some minor losses of PFAS in the test and generation of small amounts of PFCAs. Other than the loss of PFOS, the changes were minor and suggests the sample does not contain much precursor material. The low-levels of precursors suggests a weathered product that contained substantial concentrations of detectable compounds. Given that PFOS and PFHxS were major constituents, it is likely the source was an electrochemical process product.

The plan for implementation shifted during the test. Ultimately, it was decided to shift a smaller sample of soil from Willow Grove to Iron Creek's facility for treatment. Two batches of soil were created for treatment, a batch of Willow Grove soil diluted with clean local soil (the Low-Concentration test soil) and a batch containing Willow Grove soil, clean local soil and AFFF (the High-Concentration Test soil), based on the calculations presented in the Demonstration Plan (Appendix A). A material safety data sheet for the AFFF used to spike the High-Concentration Test is also included in the calculation sheet in the Demonstration Plan (Appendix A). The AFFF was 150 milliliters (mL) of an electrochemical process AFFF collected at a DoD facility. It was thought that this might be a good match for the Willow Grove contamination.

3.2 TEST EQUIPMENT AND OPERATION

The system design as used in the tests is shown on Figure 3.1. Once the system was started, components shown on Figure 3.1 were checked and soil temperature was recorded from four

sensors daily. All system components were monitored and checked frequently throughout the day to ensure proper operation.

3.2.1 Thermal Desorption Unit

The TDU was operated by heating the air in the treatment bin using propane as the source of heat. The TDU is a closed-loop system where gases are captured and recirculated back the treatment bin.

As the vapors exit the treatment bin, they are recirculated through a condenser and both liquidand vapor-phase effluent were treated using granular activated carbon (GAC). Additional details about each component of the system are provided in the following subsections.

3.2.2 Condensate Handling and Treatment

A blower was used to draw vapors from the TDU through a condenser (Figure 3.1). Condensate then accumulated in high-density polyethylene drums. During the Low-Concentration Test, 45 gallons of condensate was recovered. During the High-Concentration Test, 48 gallons of condensate was recovered. Separate drums were used to collect the condensate from each test run. Condensate was treated to remove PFAS using an LSU-30 liquid-phase GAC unit from Cameron Great Lakes, Inc., Molecular Filtration Specialists (Appendix B). The used GAC was returned to the vender for regeneration.

3.2.3 Vapor Handling and Treatment

After the vapors passed through the condenser, they were piped from the condenser to a VGAC vessel; C-18 sample ports were located before and after the VGAC vessel, as shown on Figure 3.1. Treated vapors were returned to the treatment chamber via a fan. The sample ports were used to allow vapor to filter through the cartridges to provide samples for analysis as discussed in Section 3.4. Two VGAC treatment units (carbon vessels) were used to treat the vapors during recirculation, one for each test run. The carbon vessels were VSU-30 vapor phase units from Cameron Great Lakes, Inc., Molecular Filtration Specialists (Appendix B). The GAC medium was sampled as described in Section 3.4. Used VGAC was returned to the vender for regeneration.

3.3 EXPERIMENTAL DESIGN

The test design for this demonstration project consisted of obtaining contaminated soil from an active remediation site, blending the soil with cleaner soil and adding AFFF to create two synthetic soil IDWs (a lower concentration and a higher concentration IDW, respectively). The higher concentration IDW had AFFF added as described in Section 3.1.2. These synthetic IDWs were treated in the portable treatment unit.

In addition to documenting the treatment process, mass balance was used to track the total PFAS through the treatment process. This includes using TOPA to estimate the total mass of PFAS in the system, and detailed analysis of the PFAS compounds by the CSM to identify the main constituents of the TOPA. Standard compound list PFAS analysis was used to track PFAS through the treatment process, including in residuals, to understand its fate in the process.

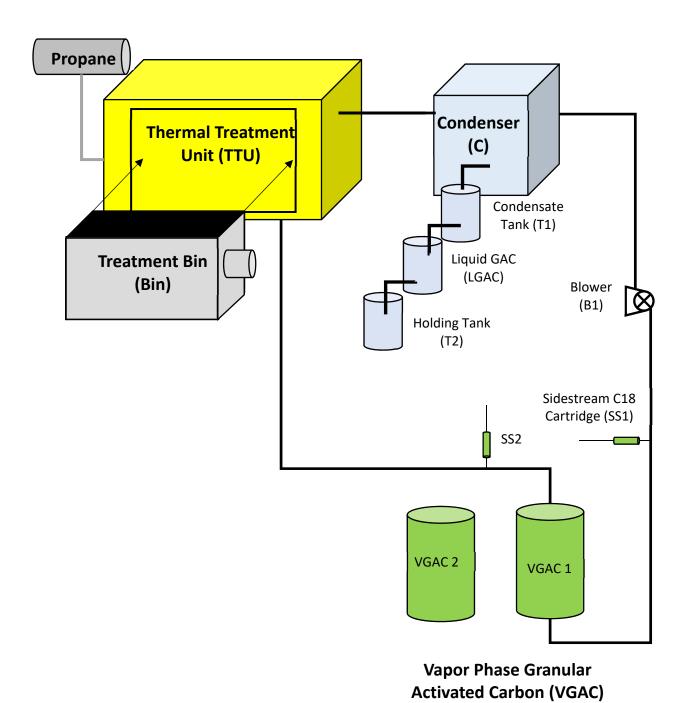


Figure 3.1. Thermal Treatment Unit Design Schematic

3.3.1 Bench-Scale Testing Results

Two infrared thermal treatment bench-scale tests that were conducted in 2017 at multiple temperatures and multiple testing times. Results of the two bench-scale tests are included in the Demonstration Plan (Appendix A).

Results of bench scale testing indicated all PFAS tested (PFCAs, PFSAs, FtS, and PFOSA) were removed at 350 degrees Celsius (°C), while removal was less consistent at 300°C. Most removal occurred over 1 day at 350°C; although, some compounds (PFOS and PFHxS) remained until the end of the test at very low concentrations; one compound (PFHpS) appeared to increase at 4 days.

3.3.2 Test Design

Based on the bench-scale test results (Section 3.3.1), the pilot test was proposed to be conducted at 350°C for 8 days. Because the bench-scale test results showed most removal occurring in 1 day, an 8-day span was selected for the design to allow sufficient time for the compounds to desorb.

3.4 FIELD TESTING METHODS

3.4.1 Soil Batch Development

Two soil batches were created to evaluate thermal desorption of PFAS in soil with both low and high concentrations of PFAS. To create the low-concentration batch, 8,500 pounds of clean fill, generally silty or clayey soil, was added to 1.5 drums of Willow Grove soil. No AFFF was added to this batch. To create the high-concentration batch, 7,800 pounds of clean fill, generally silty or clayey soil, was added to 1.5 drums of Willow Grove soil and supplemented with 150 mL of AFFF.

Test soil was mixed in a dumpster in three roughly equal lifts (one-third of the total in each lift). Each lift was created using a layer of clean soil overlain by a layer of Willow Grove soil. For the high-concentration soil, 50 mL of AFFF was added with 5 gallons of water to the surface of each lift. The soil in the dumpster was then mixed until thoroughly blended.

3.4.2 Sample Collection

Table 3.3 summarizes the samples collected during the tests.

Table 3.3. Sample Matrix

Laboratory:	AXSYS SGS CANADA		AXSYS SGS ORLANDO	CSM	Batelle	Geo Testing
Analysis:	PFAS	TOPA PFAS	PAHs	PFAS	PFAS	Grain Size
Low Concentration Test	ITAS	ITAS	1 Alls	ITAS	ITAS	Size
SOIL-DAY0-LOW-0719	X	X	X	X		
SOIL-DAY1-LOW-0719	X	X		X		
SOIL-DAY2-LOW-0719	X	X		X		
SOIL-DAY3-LOW-0719	X	X		X		
SOIL-DAY4-LOW-0719	X	X		X		
SOIL-DAY5-LOW-0719	X	X		X		
SOIL-DAY6-LOW-0719	X	X		X		
SOIL-DAY7-LOW-0719	X	X		X		

Laboratory:	AXSYS SGS CANADA		AXSYS SGS ORLANDO	CSM	Batelle	Geo Testing
	2216	TOPA		2216	22.0	Grain
Analysis:	PFAS	PFAS	PAHs	PFAS	PFAS	Size
SOIL-DAY8-LOW-0719	X	X	X	X	37	X
C18-DAY1-LOW-0719					X	
C18-DAY2-LOW-0719		1			X	
C18-DAY3-LOW-0719					X	
C18-DAY4-LOW-0719		1			X	
C18-DAY5-LOW-0719					X	
C18-DAY6-LOW-0719					X	
C18-DAY7-LOW-0719					X	
C18-DAY8-LOW-0719					X	
WIPE-DAY0-LOW-0719					X	
WIPE-DAY8-LOW-0719					X	
COND-DAY8-LOW-0719					X	
VGAC-DAY <mark>8</mark> -LOW-0719					X	
VIRGIN-CARBON-0719					X	
Quality Assurance/Quality						
Control Samples						
SOIL-DAY8-LOW-0719-QA	X	X		X		
SOIL-DAY8-HIGH-0719-QA	X	X	X	X		
'C18-HIGH-0719-FIELD-					v	
BLANK					X	
WIPE-HIGH-0719-FIELD-					V	
BLANK					X	
COND-HIGH-0719-FIELD-					V	
BLANK					X	
High Concentration Test						
SOIL-DAY0-HIGH-0719	X	X	X	X		
SOIL-DAY1-HIGH-0719	X	X		X		
SOIL-DAY2-HIGH-0719	X	X		X		
SOIL-DAY3-HIGH-0719	X	X		X		
SOIL-DAY4-HIGH-0719	X	X		X		
SOIL-DAY5-HIGH-0719	X	X		X		
SOIL-DAY6-HIGH-0719	X	X		X		
SOIL-DAY7-HIGH-0719	X	X		X		
SOIL-DAY8-HIGH-0719	X	X	X	X		X
C18-DAY1-HIGH-0719		1.		- 11	X	
C18-DAY2-HIGH-0719		†			X	
C18-DAY3-HIGH-0719		†			X	
C18-DAY4-HIGH-0719		+			X	
C18-DAY5-HIGH-0719		+			X	
C18-DAY6-HIGH-0719		1			X	
C18-DAY7-HIGH-0719		1			X	
C18-DAY8-HIGH-0719		+			X	
		+			X	
WIPE-DAY9-HIGH-0719		1		1	X	
WIPE-DAY8-HIGH-0719		1		1		
COND-DAY8-HIGH-0719		1			X	
VGAC-DAY0-HIGH-0719		1			X	
VGAC-DAY8-HIGH-0720		1			X	

Notes:

PFAS - per- and polyfluoro alkyl substances

3.4.2.1 Soil Sample Collection

During the Low-Concentration Test and High-Concentration Test, pre-, in-, and post-treatment soil samples (five grabs per sample) were collected from the treatment bin, composited, and split into two composite samples. CSM and SGS AXYS (Sidney, British Columbia) analyzed these samples for PFAS. Also, SGS AXSYS (Vineland, Florida) analyzed the Day 0 and Day 8 samples from each test for the presence of PAHs (four total samples). One PAH QC sample was also collected from the Day-8, High-Concentration Test (Table 3.3).

Soil samples were composite samples constructed of five roughly 10-ounce (oz.) grabs of soil. Individual grab samples for the composite sample were collected and homogenized. Once mixed, the sample containers were filled. Pre- samples (Day 0) were collected directly from various locations in the bin. During the run, Days 1 through 8, samples were collected through the sampling ports installed in the TDU at approximately the same time each day.

3.4.2.2 Wipe Sample Collection

Wipe samples were collected from the inside of the TDU, outside the bin, before it was loaded with soil and at the completion of each test. Two sub-samples were collected at two locations (from sections of two walls or the top and bottom of the inside of the chamber) and sent to Battelle for PFAS analyses. The same locations were used for all wipe samples.

Wipe samples were collected by taping a cardboard template, with a hole measuring 2.5 inches by 2.5 inches (10 centimeters [cm] by 10 cm) square (roughly 6 square inches [100 square cm]), to the surface to be sampled and wiping the area with a methanol-moistened cotton wipe. As the gauze became soiled, the soiled surface was folded inward, and a fresh surface was used until the entire area had been wiped clean. The wipe samples were then placed in the laboratory-provided container. The process was repeated in the second area and all wipes used to collect the sample were placed in the same container.

3.4.2.3 C-18 Cartridges

To assess the mass of PFAS in vapor, C-18 cartridges were attached to a side-stream of the exhaust vapor (Figure 3.1). For early test samples where higher concentrations were expected, two cartridges were hooked together using the Luer slip fitting (syringe fitting) and attached to the side stream tubing before the VGAC unit using adaptors with hose barbs. During the latter days of the test, single C-18 cartridges were used to collect samples after the VGAC treatment unit because it was expected that concentrations would be lower. The C-18 cartridges were swapped out daily during treatment to evaluate PFAS desorption over time. Cartridges were collected, placed in a polyethylene centrifuge tube and shipped to Battelle. Where two cartridges were used, both cartridges were collected together as a single sample.

3.4.2.4 VGAC Samples

The test used two VGAC units. The units were swapped between tests to estimate what was adsorbed during each test.

At the beginning of the Low-Concentration Test, the first VGAC unit (VGAC1) was used. After the first test was completed, a composite sample was collected from VGAC1 using the same

method as described for soil samples (collect five grab samples from the surface, bottom and middle of the unit, mix and collect the composite sample, return remaining VGAC to the unit). After sampling the VGAC1, it was removed and replaced with VGAC2 for the High-Concentration Test. After the second test was completed, the second composite VGAC sample was collected from the VGAC2 unit. VGAC samples were shipped to Battelle for analysis.

3.4.2.5 Residuals

Condensate was collected from the condensate tank using a spigot installed in the tank. Condensate samples were collected at the end of each run, on Day 8, and shipped to Battelle. The total volume of condensate was estimated by measuring the diameter of the tank and the height of the condensate in the tank, 45 gallons for the Low-Concentration Test and 48 gallons for the High-Concentration Test. After the Low-Concentration Test, the condensate was emptied into the holding tank (Figure 3.1) before the High-Concentration Test was completed.

3.4.3 Sample Analysis

The following procedures were used by SGS AXYS, CSM, and Battelle laboratories to prepare and analyze the samples collected as described in Section 3.4.2. Laboratory reports are provided in Appendix C. Target PFAS analytes are listed in Table 1.1.

3.4.3.1 SGS AXYS

Soil samples were analyzed for PFAS and TOPA by SGS AXYS Analytical Services using two analytical approaches. SGS AXYS method MLA-110 was used to determine the concentrations of 29 targeted PFAS and SGS AXYS Method MLA-111 was used measure perfluorinated carboxylates and sulfonates after a TOPA reaction (Houtz and Sedlak 2012) to estimate presence of oxidizable perfluoroalkyl acid (PFAA) precursors in the samples.

Samples were homogenized and two separate subsamples were taken for each analysis. Prior to extraction, MLA-110 samples were fortified with isotopically labeled quantification standards for quantitation and MLA-111 samples were fortified with a subset of isotopically labeled standards to monitor losses through extraction. Each subsample was extracted in methanolic ammonium hydroxide and exchanged to an aqueous solution. For the oxidizable precursors, the extract pH was adjusted to 13 and allowed to react at 85°C for 6.5 hours. For both MLA-110 and MLA-111, the resulting aqueous extracts are then cleaned using Waters Oasis 150 milligrams (mg) WAX solid-phase extraction (SPE) cartridge. Sample extracts are analyzed by liquid chromatograph coupled to triple quadrupole mass spectrometer (UPLC-MS/MS) with the MS run with unit mass resolution in the multiple reaction monitoring (MRM) mode. Analyte concentrations are determined using MassLynx v.4.1 software.

3.4.3.2 Colorado School of Mines

All PFAS analytical standards were purchased from Wellington Laboratories Inc. TCEP-d12 from Sigma Aldrich and atrazine-d5, diuron-d6, triphenyl phosphate-d15, metolachlor-d6, and carbamazepine-d10 from CDN Isotopes were used as positive mode electrospray (electrospray ionization+ [ESI+]) injection standards. Unless otherwise noted, all solvents were Optima HPLC-grade and purchased from Fisher Scientific.

All soil samples were air dried and homogenized with a mortar and pestle and sieved to 2 millimeters (mm). The dry weight of soil was determined after drying overnight at 105°C.

Approximately 0.5 gram (g) of soil was weighed into a 50 mL polypropylene centrifuge tube, then 4 mL of 0.1 moles (M) of ammonium hydroxide (NH4OH) in methanol ("basic methanol") was added to the tube, which was vortexed for 30 seconds, sonicated for 15 minutes at 30°C, and centrifuged at 2,470 relative centrifugal force (rcf) for 20 minutes. Finally, the supernatant was decanted and a second extraction round was begun. After the second extraction round, two more rounds were performed with 4 mL of 0.5 M hydrochloric acid (Fisher Scientific TraceMetal grade) in methanol ("acidic methanol") instead of the basic methanol. After the four extraction rounds (two basic, two acidic), the basic and acidic extracts were passed through separate Envicarb (Sigma Aldrich, 250 mg) SPE cartridges, then the acidic extracts were cooled at -20°C for at least an hour and neutralized with 1:1 NH4OH:methanol. Basic and acidic extracts were then evaporated to dryness under nitrogen in a 30°C water bath. The extracts were reconstituted in 1.5 mL of 1 percent acetic acid in methanol and recombined with a glass pipet. Finally, the combined extract was transferred to a microcentrifuge tube while avoiding the transfer of precipitants formed during the acidic extract neutralization and evaporation. Extracts were then stored at -20°C overnight and centrifuged for 10 minutes at 17,000 rcf, before the dilution of a 100 microliter (µL) aliquot to 400 μL total volume (20 percent aqueous/80 percent methanol composition) in an autosampler vial. Quality control samples prepared each extraction batch included a method blank consisting of clean sand containing PFAS internal standards, a matrix spike sample consisting of site soil with PFAS internal standards spiked known quantity of injection standards, and laboratory control samples consisting of clean sand with PFAS internal standards spiked known quantity of injection standards.

All PFAS analysis was performed by liquid chromatography Quadrupole Time of Flight Mass Spectrometry (SCIEX X500R). Each extract was analyzed twice—once for anionic and zwitterionic PFAS with negative mode electrospray (ESI-) analysis and once for cationic and zwitterionic PFAS with ESI+ analysis. Both analyses (100 µL injections) were made with identical chromatographic conditions except for the guard columns used. A Gemini C-18 analytical column (3 mm × 100 mm × 5 micrometer (µm); Phenomenex, Torrance, California), one SecurityGuard C-18 Guard Cartridge (4 mm × 2 mm I.D.; Phenomenex), and either two Zorbax DIOL guard columns (4.6 mm × 12.5 mm × 6 µm; Agilent, Santa Clara, California) for ESI- mode or one Zorbax SIL guard column (4.6 mm × 12.5 mm × 5 μm; Agilent) for ESI+ mode were used for all injections. All data were collected via SWATH Data-Independent Acquisition. A calibration curve of anionic target PFAS ranging from 0.02 picograms (pg) to 2000 pg (mass injected) was run with all ESI- analytical runs. For ESI- analysis, 13C2-PFOA was used as an injection standard, added directly to the autosampler vials to check for consistency of injections and matrix effects. Because no isotopically labeled PFAS standards are currently available that ionize by ESI+, the following compounds were used as injection standards in ESI+: atrazine-d5, diuron-d6, TCEP-d12, triphenyl phosphate-d15, metolachlor-d6, and carbamazepine-d10. Data were only used when the recorded peak area of injection standards in samples were within 50 percent of the average peak areas recorded during the calibration curve. In addition, thee ESI+ standards: n-dimethyl ammonio propyl perfluorohexane sulfonamide, n-trimethylammoniopropyl perfluorohexane sulfonamide, and 6:2 fluorotelomer sulfonamido propyl betaine were used in a calibration curve for ESI+ analysis. Instrument blanks, double blanks, and continuing calibration verification standards were run after every 10 samples. A mixture of AFFFs was injected alongside samples as a chromatographic reference point for suspect screening hits.

3.4.3.3 <u>Battelle La</u>boratory

3.4.3.3.1 Sample Preparation for different Matrices

3.4.3.3.1.1 Solid Samples and Wipe Samples

Solid samples were aliquoted into extraction tubes, while the wipe samples were extracted as received in the 50 mL polypropylene vials. Both the solid and the wipe samples were spiked with surrogates prior to the addition of solvent for extraction. The solid sample was serially extracted twice using 0.4 percent ammonia (NH₃) in methanol and cleaned up using ENVI-Carb SPE cartridges. Extracts were concentrated to dryness under nitrogen and reconstituted with 80:20 methanol/water (V/V) and fortified with internal standard for liquid chromatography–mass spectrometry/mass spectrometry (LC/MS/MS) analysis.

3.4.3.3.1.2 C-18 Cartridges

At each sampling point, two 900 mg C-18 cartridges connected in series were collected (Figure 3.1). The samples were prepared for extraction by spiking with surrogates prior to elution with 0.4 percent NH₃ in methanol. Extracts were concentrated to dryness under nitrogen with a water bath set between 35°C and 45°C, reconstituted with 80:20 methanol/water (V/V) and fortified with internal standard. Extracts were transferred for LC/MS/MS analysis.

3.4.3.3.1.3 Water Samples

The water samples were spiked with surrogates in the original sample container from the field. The water was extracted using a weak ion exchange SPE cartridge and eluted from the SPE with 0.4 percent NH₃ in methanol. Extracts were concentrated to dryness under nitrogen and reconstituted with 80:20 methanol/water (V/V) and fortified with internal standard. Extracts were transferred for LC/MS/MS analysis.

3.4.3.3.2 Analysis

Analysis of PFAS was performed using liquid chromatography tandem mass spectrometry in the MRM. An initial calibration consisting of representative target analytes, labelled extracted internal standards (EIS), and labeled internal standards was analyzed prior to analysis to demonstrate the linear range of analysis. Calibration verification was performed at the beginning and end of 10 injections and at the end of each sequence. The concentration of each analyte is determined by using the isotope dilution quantitation technique following the QSM 5.1.1 Table B-15 quality criteria. The isotopically labeled analog of an analyte (surrogate) is used for quantitation if commercially available. If a labeled analog is not commercially available, internal standard quantitation is performed using the surrogate analyte with closest retention time to the analyte. Except for two analytes (PFBA and PFPeA), two transitions are monitored, one for quantitation and the other for confirmation.

3.4.3.3.3 Quality Assurance and Quality Control

The analytical laboratory provided pre-tested PFAS free water for field blanks. The field blank analysis provides information on any potential contamination during sampling. A procedural blank (or method blank) is included for each analytical batch to ensure the sample extraction and analysis methods are free of contamination. A laboratory control sample is included for every analytical batch. The percent recoveries of target analytes are calculated to measure accuracy. The EIS masslabelled analytes are added prior to extraction in all samples and QC samples. The EIS recoveries are calculated to measure extraction efficiency. For each analytical batch, at a minimum, a 10 percent review of all data was performed by a quality assurance (QA) auditor, or designee. The

review includes tracing samples from receipt through extraction, analysis, and final reporting. All QA reviews was approved by management and stored as part of QA records.

3.5 DATA ANALYSES

Due to the number of PFAS in the environment and different analytical methods used in this study to quantify concentrations and evaluate precursors, the data are generally discussed as total PFAS defined as the sum of detected PFAS. In addition, due to their different properties, the PFAS were split into two main groups, PCFAs and PFSAs. Although, FtS are also discussed, where applicable. In particular, select PFCAs and PFSAs were used to evaluate overall composition of different sample matrices using radar plots. For comparison, PFBA, PFPeA, PFHxA, PFHpA, and PFOA are the PFCAs evaluated to look at trends in matrix composition. The PFSAs evaluated include PFBS, PFPeS, PFHxS, PFHpS, and PFOS.

3.5.1 **Contaminant Removal and Retention**

Contaminant removal rates were calculated using the concentrations of total PFAS in the soil batches before and after treatment. .

3.5.1.1 Soil

Removal rates were calculated for each treatment batch. Removal from soil was calculated for the test endpoint, as well as for the samples collected during treatment to allow an evaluation of removal over time.

percent Removal = $1 - Con_t/Con_b$

(Equation 3-1)

- Untreated concentration (Conb)
- Treated concentration (Con_t)

The contaminant mass in the soil (untreated and treated) (Mc) is:

$$TCMass = C * SMass$$

(Equation 3-2)

- Soil concentration (C), either treated or untreated
- Soil mass (SMass)

3.5.1.2 Vapor Phase (C-18 Cartridges)

The vapor phase cartridges were not used in the mass balance and the amount of PFAS removed by treatment was estimated in the VGAC instead. Because gasses were recirculated, PFAS passing the VGAC (if there was any) was returned to the treatment unit.

3.5.1.3 Surfaces

The contaminant mass on the equipment surface (TCMass_s) is:

$$TCMass_s = MW/AW*Ae$$

(Equation 3-3)

- Mass of contaminant on wipe (MW)
- Area wiped (AW)
- Area of equipment (Ae)

3.5.1.4 <u>VGAC</u>

To contaminant mass adsorbed to the VGAC (TCMass_v) is:

 $TCMass_v = Conc_v * Vol_v$

(Equation 3-4)

- VGAC sample concentration (Con_v)
- Volume of VGAC (Vol_v)

3.5.1.5 Condensate

The contaminant mass in the condensate (TCMasscc) is:

TCMass_{cc} = Conc * Volc

(Equation 3-5)

- Condensate concentration (Conc)
- Volume of condensate (Volc)

3.5.1.6 Mass Balance

The mass balance includes the following elements:

 $TCMassb = TCMass_t + TCMass_{vgac} + TCMass_{cc} + TCMass_e + TCMass_{tr}$ (Equation 3-6)

- Total Contaminant Mass in the untreated soil batch (TCMass_b)
- Total Contaminant Mass in the treated soil batch (TCMass_t)
- Total Contaminant Mass captured on VGAC (TCMass_{vgac})
- Total Contaminant Mass capture in condensate (TCMass_{cc})
- Total Contaminant Mass on the equipment surface (TCMass_e)
- Total Contaminant Mass change from transformation (TCMass_{tr})

The mass of contamination lost to transformation is estimated by converting the mass of contaminants into moles, calculating an average molecular weight, and using the change in molecular weight and totals moles of contaminants to estimate the change in total mass. This was done for total PFAS and is anticipated to capture the loss of functional group mass from the PFAS, as well as any unzipping (which is not expected to be significant). However, because the mass of functional groups on some PFAS compounds can be greater than the perfluorinated portion, this loss of mass can be significant. Comparing the total detected PFAS to the TOPA results on a molar basis give a reasonable estimate of how much of the total PFAS can be accounted for by this method.

The moles of contaminant (CMole) is calculated by dividing the contaminant mass (CMass) by its molecular weight (CMwt)

(Equation 3-7)

The sum of the moles of contaminants is the total moles of contaminants (TCMole).

The average molecular weight (AvgMwt) of the contaminants is:

(Equation 3-8)

If contaminants degrade during treatment, the average molecular weight will decrease. This loss of mass is calculated by:

 $TCMass_t = (AvgMwt_1 - AvgMwt_2) * TCMole$

(Equation 3-9)

- Mass lost to transformation (TCMass_t)
- AvgMwt₁ is the average molecular weight of the untreated sample
- AvgMwt₂ is the average molecular weight of the treated sample
- TCMole is the initial moles of PFAS in the sample.

Any PFAS converted into compounds not detected would appear to be a loss of mass. Any compound not detected in the initial testing could transform into detectable compounds and appear to be an increase in mass. The calculation assumes that the perfluorinated ends of the PFAS are stable and the PFAS compounds degrade by simplifying, making it likely that degradation products will remain detectable and therefore, moles of PFAS will not be changed significantly during treatment. Therefore, a loss of moles in the calculation indicates unaccounted mass.

Once all elements of the mass balance are assembled, Equation 3-6 can be solved for the efficiency (E) of the mass balance in capturing contaminants.

3.5.1.7 Water Balance

To better understand how well the recirculation and vapor treatment was able to contain and manage the vapors, moisture was tracked in the system. A water balance was added to the mass balance, with the moisture content and mas of soil in the test batches used as the initial water content, and water recovery in the condenser and other process locations used to estimate recovery.

4.0 RESULTS AND EVALUATION

The following objectives are taken from the revised demonstration plan.

4.1 **OBJECTIVE 1: TREAT PEAS-CONTAMINATED SOIL**

Results for the Low-Concentration and High-Concentration tests are as follows. **Low-Concentration Soil Test Results**

Table 4.1 summarizes the temperatures recorded at each sensor and the average daily temperature for each Test. Results indicate that the average target temperature of 350°C was met by Day 4 of the Low-Concentration Test.

Table 4.2 presents the concentrations of the PAHs in soil before and after treatment in the Low-Concentration Test. No PAHs were detected in any of the post-treatment samples indicating that thermal desorption effectively treats PAHs where present as co-contaminants with PFAS.

Table 4.1. Daily Temperature Readings

Test Day	Date/Time	Sensor 0	Sensor 1	Sensor 2	Sensor 3	Average
Low Concents	ration Test (Celsius)			•		
Day 1	7/7/19 10:00 AM	201	100	197	135	158
Day 2	7/8/19 8:00 AM	315	223	287	272	274
Day 3	7/8/19 12:01 PM	334	309	310	308	315
Day 4	7/9/19 7:00 AM	379	341	349	368	359
Day 5	7/9/19 12:01 PM	388	353	353	378	368
Day 6	7/10/19 6:00 AM	414	389	376	409	397
Day 7	7/11/19 7:00 AM	406	394	361	361	380
Day 8	7/12/19 9:00 AM	386	375	344	389	373
High Concent	ration Test (Celsius)					
Day 1	7/17/19 7:00 AM	100	100	101	101	100
Day 2	7/18/19 7:00 AM	100	183	149	133	141
Day 3	7/19/19 7:00 AM	140	261	228	266	224
Day 4	7/20/19 7:00 AM	264	362	312	289	307
Day 5	7/21/19 7:00 AM	312	353	301	272	309
Day 6	7/22/19 7:00 AM	313	378	333	307	333
Day 7	7/23/19 7:00 AM	318	393	352	319	346
Day 8	7/24/19 7:00 AM	312	369	313	316	328

Bold font indicates target average temperature of 350 degrees Celsius was achieved.

Table 4.2. PAH Analytical Results from SGS AXYS

	Low Conc	entration Test				High Concentra	tion Test		
Pre-Treatment		Post-Treatn	ent	PRE-TREATMENT	Γ - HIGH-C	POST-TREATMEN	T - HIGH-C	POST-TREATMENT -	HIGH-C (QA)
		Sample Collected: 14-Jul-		Sample Collected: 16-Jul-		Sample Collected: 25-Jul-		Sample Collected: 25-Jul-	
Sample Collected: 7-Jul-2019		2019		2019		2019		2019	
	Result								
Compound	(µg/kg)	Compound	Result (µg/kg)	Compound	Result (µg/kg)	Compound	Result (µg/kg)	Compound	Result (µg/kg)
Acenaphthene	35 U	Acenaphthene	33 U	Acenaphthene	37 U	Acenaphthene	32 U	Acenaphthene	32 U
Acenaphthylene	35 U	Acenaphthylene	33 U	Acenaphthylene	37 U	Acenaphthylene	32 U	Acenaphthylene	32 U
Anthracene	35 U	Anthracene	33 U	Anthracene	37 U	Anthracene	32 U	Anthracene	32 U
Benzo(a)anthracene	11.9 J	Benzo(a)anthracene	6.6 U	Benzo(a)anthracene	15.7	Benzo(a)anthracene	6.5 U	Benzo(a)anthracene	6.5 U
Benzo(a)pyrene	11.7 J	Benzo(a)pyrene	6.6 U	Benzo(a)pyrene	17.1	Benzo(a)pyrene	6.5 U	Benzo(a)pyrene	6.5 U
Benzo(b)fluoranthene	24.4	Benzo(b)fluoranthene	6.6 U	Benzo(b)fluoranthene	24.4	Benzo(b)fluoranthene	6.5 U	Benzo(b)fluoranthene	6.5 U
Benzo(g,h,i)perylene	9.4 J	Benzo(g,h,i)perylene	6.6 U	Benzo(g,h,i)perylene	12.5 J	Benzo(g,h,i)perylene	6.5 U	Benzo(g,h,i)perylene	6.5 U
Benzo(k)fluoranthene	7.4 J	Benzo(k)fluoranthene	6.6 U	Benzo(k)fluoranthene	8 J	Benzo(k)fluoranthene	6.5 U	Benzo(k)fluoranthene	6.5 U
Chrysene	18.7	Chrysene	6.6 U	Chrysene	19.1	Chrysene	6.5 U	Chrysene	6.5 U
Dibenzo(a,h)anthracene	7 U	Dibenzo(a,h)anthracene	6.6 U	Dibenzo(a,h)anthracene	7.4 U	Dibenzo(a,h)anthracene	6.5 U	Dibenzo(a,h)anthracene	6.5 U
Fluoranthene	27.5 J	Fluoranthene	33 U	Fluoranthene	36.3 J	Fluoranthene	32 U	Fluoranthene	32 U
Fluorene	35 U	Fluorene	33 U	Fluorene	37 U	Fluorene	32 U	Fluorene	32 U
Indeno(1,2,3-cd)pyrene	10.7 J	Indeno(1,2,3-cd)pyrene	6.6 U	Indeno(1,2,3-cd)pyrene	14.2 J	Indeno(1,2,3-cd)pyrene	6.5 U	Indeno(1,2,3-cd)pyrene	6.5 U
2-Methylnaphthalene	35 U	2-Methylnaphthalene	33 U	2-Methylnaphthalene	37 U	2-Methylnaphthalene	32 U	2-Methylnaphthalene	32 U
Naphthalene	35 U	Naphthalene	33 U	Naphthalene	37 U	Naphthalene	32 U	Naphthalene	32 U
Phenanthrene	35 U	Phenanthrene	33 U	Phenanthrene	19.5 J	Phenanthrene	32 U	Phenanthrene	32 U
Pyrene	22.2 J	Pyrene	33 U	Pyrene	29 J	Pyrene	32 U	Pyrene	32 U
Sum of detected PAHs	143.9	Sum of detected PAHs	ND	Sum of detected PAHs	195.8	Sum of detected PAHs	ND	Sum of detected PAHs	ND

J - Value is estimated

µg/kg - micrograms per kilogram

QA - quality assurance sample

U - Undetected

ND - none detected

Total PFAS concentrations reported by SGS AXYS, post-oxidation concentrations reported by SGS AXYS, and total PFAS concentrations reported by CSM during the Low-Concentration Test are presented in Tables 4.3, 4.4, and 4.5, respectively. Removal rates for PFOA, PFOS, and Total PFAS are summarized in Table 4.6.

Table 4.3 results indicate all compounds were removed to less than the detections limits except PFHxS (0.126 μ g/kg) and PFOS (0.703 μ g/kg). Removal of PFOA was to below the detection limit and exceeded 97.4 percent. Removal of PFOS was 99.6 percent and removal of total PFAS was also 99.6 percent (Table 4.6).

Table 4.4 results indicates all compounds were removed to less than the detections limits except for PFBA (2.68 μ g/kg) and PFOS (1.59 μ g/kg). Removal of PFOA was to below the detection limit and exceeded 74.3 percent (the detection limit was elevated at 1.59 μ g/kg). Removal of PFOS was 99.6 percent and removal of total PFAS was also 99.6 percent (Table 4.6).

Table 4.5 results also indicated all compounds were removed to less than the detection limits, although the detection limits were elevated at 0.6 μ g/kg to 18.47 μ g/kg. Removal rates were estimated from greater than 19.5 to 99.9 percent (Table 4.6).

Figure 4.1 presents pre- and post- oxidation PFAS concentrations over the Low-Concentration Test based on daily composite sample results. Depletion of PFAS compounds over time are also shown on radar plots included as Figure 4.2. The data shows that most of the compounds and post-oxidation compounds were removed from soil in 4 days when the average temperature of the test soil was heated to the target temperature of 350°C. The re-use criteria were met for both PFOA and PFOS.

4.1.2 High-Concentration Soil Test Results

Table 4.1 summarizes the temperatures recorded at each sensor and the average daily temperature for each test. Results indicate that the average target temperature (350°C) was not met during the High-Concentration Test; however, the average temperature on Day 7 was 346°C. Sensors 1 and 2 reached the target temperature on Days 3 through 8 and Day 7, respectively.

Table 4.2 presents the concentrations of the PAHs in soil before and after treatment in the High-Concentration Test. No PAHs were detected in any of the post-treatment sampled indicating that thermal desorption effectively treats PAHs where present as co-contaminants with PFAS even though the target temperature was not met.

Total PFAS concentrations reported by SGS AXYS, post-oxidation concentrations reported by SGS AXYS, and total PFAS concentrations reported by CSM during the High-Concentration Test are presented in Tables 4.7, 4.8, and 4.9, respectively. Removal rates for PFOA, PFOS, and Total PFAS are summarized in Table 4.6.

Table 4.7 results indicate 14 PFAS (PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFBS, PFPeS, PFHxS, PFHpS, PFOS, PFNS, PFDS, 6:2 FtS and 8:2 FtS) were detected in the post-treatment sample at concentrations from $0.17 \,\mu\text{g/kg}$ to $326 \,\mu\text{g/kg}$. Removal of PFOA exceeded 97.3 percent. Removal of PFOS was 74.0 percent and removal of total PFAS was 73.0 percent (Table 4.6).

Table 4.3. Soil Results - Low Concentration Test - SGS AXYS (Total)

Compound	Re-use Criteria (µg/kg)	Day 0 (μg/kg)	Day 1 (μg/kg)	Day 2 (μg/kg)	Day 3 (μg/kg)	Day 4 (μg/kg)	Day 5 (μg/kg)	Day 6 (μg/kg)	Day 7 (μg/kg)	Day 8	(QA) /kg)
PFBA		0.606	0.837	0.465	0.603 U	0.604 U	0.601 U	0.59 U	0.6 U	0.596 U	0.597 U
PFPeA		2.6	3.3	1.09	0.279 K	0.302 U	0.301 U	0.295 U	0.3 U	0.298 U	0.298 U
PFHxA		11.8	15.6	4.11	0.459	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
PFHpA		1.17	1.38	0.371	0.066	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
PFOA	1.7ª	5.55	5.86	1.5	0.124	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
PFNA		0.876	0.873	0.24	0.146 U	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
PFDA		0.114	0.145	0.147 U	0.146 U	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
PFUnA		0.154 U	0.096	0.147 U	0.146 U	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
PFDoA		0.154 U	0.155 U	0.147 U	0.146 U	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
PFTrDA		0.154 U	0.155 U	0.147 U	0.146 U	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
PFTeDA		0.154 U	0.155 U	0.147 U	0.146 U	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
PFBS		0.52	0.751	0.614	0.439	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
PFPeS		2.05	2.45	1.72	1.08	0.107	0.137 U	0.134 U	0.136 U	0.03	0.136 U
PFHxS		44.6	47.4	28.2	15.8	1.7	0.191	0.075	0.059	0.482	0.126
PFHpS		7.38	7.39	3.16	1.16	0.126	0.137 U	0.134 U	0.136 U	0.057	0.136 U
PFOS	3.0^{a}	429 D	536 D	203 D	89.3	7.15	0.798	0.343	0.157	2.96	0.703
PFNS		0.136	0.223	0.054	0.137 U	0.137 U	0.137 U	0.134 U	0.136 U	0.135 U	0.136 U
PFDS		0.254	0.202	0.098 K	0.146 U	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
PFDoS		0.145 U	0.186	0.137 U	0.137 U	0.137 U	0.137 U	0.134 U	0.136 U	0.135 U	0.136 U
4:2 FTS		0.617 U	0.621 U	0.587 U	0.585 U	0.601 U	0.583 U	0.573 U	0.582 U	0.578 U	0.579 U
6:2 FTS		6.86	7.16	2.58	0.53 U	0.301 U	0.528 U	0.519 U	0.527 U	0.524 U	0.524 U
8:2 FTS		1.76	1.87	0.682	0.585 U	0.146 U	0.583 U	0.573 U	0.582 U	0.578 U	0.579 U
PFOSA		0.901	0.851 K	0.276	0.146 U	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
N-MeFOSA		NQ	NQ	NQ							
N-EtFOSA		NQ	NQ	NQ							
MeFOSAA		0.154 U	0.155 U	0.147 U	0.146 U	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
EtFOSAA		0.134	0.101	0.147 U	0.146 U	0.146 U	0.146 U	0.143 U	0.145 U	0.144 U	0.145 U
N-MeFOSE		1.57 U	1.58 U	1.49 U	1.49 U	1.49 U	1.48 U	1.46 U	1.48 U	1.47 U	1.47 U
N-EtFOSE		1.19 U	1.19 U	1.13 U	1.12 U	1.12 U	1.12 U	1.1 U	1.12 U	1.11 U	1.11 U
SUM of Detected PFAS		516	632	248	109	9.08	0.989	0.418	0.216	3.53	0.83

^a ADEC 18 AAC 75 Table B1 Soil Cleanup Level Migrations to Groundwater (ADEC, 2018)

Soil concentrations in micrograms per kilogram (µg/kg).

QA - quality assurance

ADEC = Alaska Department of Environmental Conservation

NQ = not quantified

U = undetected

D - dilution data

K - peak detected but did not meet quantification criteria, result reported represents the estimated maximum possible concentration.

-- = no criteria

Shaded indicates value exceeds re-use criteria

Table 4.4. Soil Results - Low Concentration Test - SGS AXYS (Post-oxidation)

Compound	Re-use Criteria (μg/kg)	Day 0 (μg/kg)	Day 1 (μg/kg)	Day 2 (μg/kg)	Day 3 (μg/kg)	Day 4 (μg/kg)	Day 5 (μg/kg)	Day 6 (μg/kg)	Day 7 (μg/kg)		y 8 ^b /kg)
PFBA		0.1 U	6.85	6.07 U	6.42 U	6.39 U	6.36 U	6.26 U	6.31 U	6.36 U	6.36 U
PFPeA		22.5	21.3	9.25	3.91	3.19 U	3.18 U	3.13 U	3.15 U	3.18 U	3.18 U
PFHxA		110	119	42.5	10.5	2.94	2.54	1.95	2.17	1.97	2.68
PFHpA		7.16	4.98	2.01	1.6 U	6.39 U	1.59 U	1.57 U	1.58 U	1.59 U	1.59 U
PFOA	1.7ª	10.4	8.48	2.67	1.6 U	3.19 U	1.59 U	1.57 U	1.58 U	1.59 U	1.59 U
PFNA		1.79	1.33 U	1.29 U	1.6 U	6.39 U	1.35 U	1.33 U	1.34 U	1.35 U	1.35 U
PFDA		1.29 U	1.26 U	1.21 U	1.36 U	3.19 U	1.27 U	1.25 U	1.26 U	1.27 U	1.27 U
PFUnA		1.29 U	1.26 U	1.21 U	1.28 U	6.39 U	1.27 U	1.25 U	1.26 U	1.27 U	1.27 U
PFDoA		1.29 U	1.26 U	1.21 U	1.28 U	3.19 U	1.27 U	1.25 U	1.26 U	1.27 U	1.27 U
PFTrDA		1.29 U	1.26 U	1.21 U	1.28 U	6.39 U	1.27 U	1.25 U	1.26 U	1.27 U	1.27 U
PFTeDA		1.29 U	1.26 U	1.21 U	1.28 U	3.19 U	1.27 U	1.25 U	1.26 U	1.27 U	1.27 U
PFBS		1.29 U	1.57 U	1.52 U	1.6 U	6.39 U	1.59 U	1.57 U	1.58 U	1.59 U	1.59 U
PFPeS		2.1	1.57 U	2.58	1.6 U	3.19 U	1.59 U	1.57 U	1.58 U	1.59 U	1.59 U
PFHxS		58.6	60.2	39.1	23.4	6.39 U	1.59 U	1.57 U	1.58 U	1.59 U	1.59 U
PFHpS		6.10	7.89	4.23	2.19	3.19 U	1.59 U	1.57 U	1.58 U	1.59 U	1.59 U
PFOS	3.0^{a}	490	512	244	114	6	1.59 U	1.57 U	1.58 U	2.72	1.59 U
PFNS		1.61 U	1.57 U	1.52 U	1.6 U	1.6 U	1.59 U	1.57 U	1.58 U	1.59 U	1.59 U
PFDS		1.61 U	1.57 U	1.52 U	1.6 U	1.6 U	1.59 U	1.57 U	1.58 U	1.59 U	1.59 U
PFDoS		1.61 U	1.57 U	1.52 U	1.6 U	1.6 U	1.59 U	1.57 U	1.58 U	1.59 U	1.59 U
SUM of Detected I	PFAS	709	741	346	154	8.94	2.54	1.95	2.17	4.69	2.68

Soil concentrations in micrograms per kilogram (µg/kg).

U - undetected

-- = no criteria

Shaded indicates value exceeds re-use criteria

ADEC = Alaska Department of Environmental Conservation

^a ADEC 18 AAC 75 Table B1 Soil Cleanup Level Migrations to Groundwater (ADEC, 2018)

^b Two samples were collected on Day 8 and sent the laboratory in separate containers. One samples represents the normal samples and one sample represents a duplicate of the normal sample.

Table 4.5. Low Concentration Test Results - CSM (Total)

Compound	Re-use Criteria (μg/kg)	Day 0 (μg/kg)	Day 1 (μg/kg)	Day 2 (μg/kg)	Day 3 (μg/kg)	Day 4 (μg/kg)	Day 5 (μg/kg)	Day 6 (μg/kg)	Day 7 (μg/kg)		y 8 ^b /kg)
PFBA		< 6	6.18	< 6	< 6	< 6	< 6	< 6	< 6	< 6	< 6
PFPeA		< 141.3	< 141.3	< 141.3	< 141.3	< 141.3	< 141.3	< 141.3	< 141.3	< 141.3	< 141.3
PFHxA		22.2	21.0	8.86	< 1.8	< 1.8	< 1.8	< 1.8	< 1.8	< 1.8	< 1.8
PFHpA		< 6.08	< 6.08	< 6.08	< 6.08	< 6.08	< 6.08	< 6.08	< 6.08	< 6.08	< 6.08
PFOA	1.7ª	7.57	7.25	< 6.09	< 6.09	< 6.09	< 6.09	< 6.09	< 6.09	< 6.09	< 6.09
PFNA		< 22.44	< 22.44	< 22.44	< 22.44	< 22.44	< 22.44	< 22.44	< 22.44	< 22.44	< 22.44
PFDA		< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6
PFUnA		< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6
PFDoA		< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6
PFTrDA		< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6
PFTeDA		< 1.2	< 1.2	< 1.2	< 1.2	< 1.2	< 1.2	< 1.2	< 1.2	< 1.2	< 1.2
PFBS		0.958	1.17	1.18	1.06	< 0.13	< 0.13	< 0.13	< 0.13	< 0.13	< 0.13
PFPeS		3.97	4.16	3.62	3.88	< 1.2	< 1.2	< 1.2	< 1.2	< 1.2	< 1.2
PFHxS		95.9	84.5	63.2	54.6	3.04	1.01	0.517	< 0.47	0.609	0.977
PFHpS		9.80	8.97	4.82	3.27	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6
PFOS	3.0^{a}	612	498	282	187	< 18.47	< 18.47	< 18.47	< 18.47	< 18.47	< 18.47
PFNS		1.80	1.26	0.68	0.206	< 0.15	< 0.15	< 0.15	< 0.15	< 0.15	< 0.15
PFDS		0.816	0.625	1.12	< 0.12	< 0.12	< 0.12	< 0.12	< 0.12	< 0.12	< 0.12
PFDoS		0.648	0.468	0.210	< 0.17	< 0.17	< 0.17	< 0.17	< 0.17	< 0.17	< 0.17
4:2 FTS		< 1.2	< 1.2	< 1.2	< 1.2	< 1.2	< 1.2	< 1.2	< 1.2	< 1.2	< 1.2
6:2 FTS		16.9	23.7	2.78	< 2.4	< 2.4	< 2.4	< 2.4	< 2.4	< 2.4	< 2.4
8:2 FTS		< 12	< 12	< 12	< 12	< 12	< 12	< 12	< 12	< 12	< 12
PFOSA		1.35	1.33	< 1.27	< 1.27	< 1.27	< 1.27	< 1.27	< 1.27	< 1.27	< 1.27
N-MEFOSAA		< 0.12	< 0.12	< 0.12	< 0.12	< 0.12	< 0.12	< 0.12	< 0.12	< 0.12	< 0.12
N-ETFOSAA		< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6	< 0.6
Me-FOSA		< 1.77	< 1.77	< 1.77	< 1.77	< 1.77	< 1.77	< 1.77	< 1.77	< 1.77	< 1.77
Et-FOSA		< 1.98	< 1.98	< 1.98	< 1.98	< 1.98	< 1.98	< 1.98	< 1.98	< 1.98	< 1.98
FOSAA		< 5.66	< 5.66	< 5.66	< 5.66	< 5.66	< 5.66	< 5.66	< 5.66	< 5.66	< 5.66
SUM of Detected PFAS		774	652	368	365	3.04	1.01	0.517	ND	0.609	0.977

^a ADEC 18 AAC 75 Table B1 Soil Cleanup Level Migrations to Groundwater (ADEC, 2018)

^b Two samples were collected on Day 8 and sent the laboratory in separate containers. One samples represents the normal samples and one sample represents a duplicate of the normal sample.

Soil concentrations in micrograms per kilogram (µg/kg).

< - undetected (below limit of quantitation)

CSM - Colorado School of Mines

ND - none detected

ADEC = Alaska Department of Environmental Conservation

Table 4.6. Summary of Removal Rates

Test	Compound	Source	Day 0 (μg/kg)	Day 1 (μg/kg)	Day 2 (μg/kg)	Day 3 (μg/kg)	Day 4 (μg/kg)	Day 5 (μg/kg)	Day 6 (μg/kg)	Day 7 (μg/kg)	Day 8 (μg/kg)
	PFOA		5.55	5.86	1.5	0.124	0.146	ND	ND	ND	ND
	PFOS	Native Result	429	536	203	89.3	7.15	0.798	0.343	0.157	1.8315
	Total PFAS		516	632	248	109	9.08	0.989	0.418	0.216	2.18
	PFOA	3.T. /*		-5.6%	73.0%	97.8%	>97%	>97%	>97%	>97%	>97%
	PFOS	Native Removal		-24.9%	52.7%	79.2%	98.3%	99.8%	99.9%	100.0%	99.6%
	Total PFAS	Kemovai		-22.4%	51.9%	78.9%	98.2%	99.8%	99.9%	100.0%	99.6%
	PFOA		10.4	8.48	2.67	ND	ND	ND	ND	ND	ND
	PFOS	TOPA Result	490	512	244	114	6	1.59	ND	ND	1.758
I C	Total PFAS		709	741	346	154	8.94	1.95	1.95	4.69	2.68
Low Concentration	PFOA	TODA		18.5%	>74%	>74%	>74%	>74%	>74%	>74%	>74%
	PFOS	TOPA Removal		-4.5%	50.2%	76.7%	98.8%	99.7%	>99%	>99%	99.6%
	Total PFAS	Keliloval		-4.5%	51.1%	78.3%	98.7%	99.7%	99.7%	99.3%	99.6%
	PFOA		7.57	7.25	6	ND	ND	ND	ND	ND	ND
	PFOS	CSM Result	612	498	282	187	ND	ND	ND	ND	ND
	Total PFAS		774	652	368	365	3.04	1.01	0.52	ND	0.79
	PFOA	GG) f		4.3%	>19%	>19%	>19%	>19%	>19%	>19%	>19%
	PFOS	CSM Removal		18.6%	54.0%	69.4%	>97%	>97%	>97%	>97%	>97%
	Total PFAS	Keliloval		15.7%	52.4%	52.8%	99.6%	99.9%	99.9%	>99.9%	99.9%
	PFOA		22.6	12.5	4.04	8.43	3.13	1.78	1.22	1.32	0.6125
	PFOS	Native Result	1,800	831	794	918	611	705	668	664	468
	Total PFAS		2,244	1,115	1,074	1,309	864.14	955	895	891	606.29
	PFOA	37		44.7%	82.1%	62.7%	86.2%	92.1%	94.6%	94.2%	97.3%
	PFOS	Native Removal		53.8%	55.9%	49.0%	66.1%	60.8%	62.9%	63.1%	74.0%
High	Total PFAS	Removai		50.3%	52.1%	41.7%	61.5%	57.4%	60.1%	60.3%	73.0%
Concentration	PFOA	Post-	81.3	39.9	16.6	27.5	9.50	5.2	3.52	3.95	1.55
	PFOS	1 050	1,410	732	555	732	795	638	705	563	443
	Total PFAS R		2,582	1,464	1,013	1,375	1,155	879	957	828	563
	PFOA	Post-		50.9%	79.6%	66.2%	88.3%	93.6%	95.7%	95.1%	>98.1
	PFOS	oxidation		48.1%	60.6%	48.1%	43.6%	54.8%	50.0%	60.1%	68.6%
	Total PFAS	Removal		43.3%	60.8%	46.8%	55.3%	66.0%	63.0%	68.0%	78.2%

Test	Compound	Source	Day 0 (μg/kg)	Day 1 (μg/kg)	Day 2 (μg/kg)	Day 3 (µg/kg)	Day 4 (μg/kg)	Day 5 (μg/kg)	Day 6 (μg/kg)	Day 7 (μg/kg)	Day 8 (μg/kg)
	PFOA		30.04	18.7	8.89	10.4	7.22	ND	ND	ND	ND
	PFOS	CSM Result	3,227	1,406	1,095	1,216	1,386	1,086	803	1,157	820
	Total PFAS		4,351	2,054	1,649	1,962	2,072	1,573	1,140	1,686	1,145
	PFOA			38%	70%	65%	76%	>76%	>76%	>76%	>76%
	PFOS	CSM Removal		56.4%	66.1%	62.3%	57.1%	66.4%	75.1%	64.2%	74.6%
	Total PFAS	Keliloval		52.8%	62.1%	54.9%	52.4%	63.9%	73.8%	61.2%	73.7%

Results measured in micrograms per kilogram (µg/kg).

Day 8 is the average of the result and the QC sample

PFOA - perfluorooctanoic acid

PFOS - perfluorooctane sulfonate

PFAS - Per- and Polyfluoroalkyl Substances

CSM - Colorado School of Mines

ND - compound was not detected

 Table 4.7. Soil Results - High Concentration Test - SGS AXYS (Total)

Compound	Re-use Criteria	Day 0	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Da	y 8 ^b
P 33	(µg/kg)	(μg/kg)		g/kg)							
PFBA		3.83	5.36	5.1	7.15	5.1	3.88	3.45	3.64	2.43	0.781
PFPeA		8.09	6.93	4.51	7.15	4.49	3.40	3.12	3.17	2.18	0.739
PFHxA		35.7	30.8	14.1	28.2	13.3	7.42	6.06	6.66	4.18	0.999
PFHpA		5.34	3.17	1.33	2.52	1.40	0.906	0.610	0.741	0.465	0.170
PFOA	1.7ª	22.6	12.5	4.04	8.43	3.13	1.78	1.22	1.32	0.851	0.374
PFNA		1.45	0.795	0.284	0.546	0.223	0.128	0.147 U	0.146 U	0.146 U	0.145 U
PFDA		0.184	0.14 K	0.147 U	0.108 K	0.146 U	0.149 U	0.147 U	0.146 U	0.146 U	0.145 U
PFUnA		0.076	0.147 U	0.147 U	0.146 U	0.146 U	0.149 U	0.147 U	0.146 U	0.146 U	0.145 U
PFDoA		0.145 U	0.147 U	0.147 U	0.146 U	0.146 U	0.149 U	0.147 U	0.146 U	0.146 U	0.145 U
PFTrDA		0.145 U	0.147 U	0.147 U	0.146 U	0.146 U	0.149 U	0.147 U	0.146 U	0.146 U	0.145 U
PFTeDA		0.145 U	0.147 U	0.147 U	0.146 U	0.146 U	0.149 U	0.147 U	0.146 U	0.146 U	0.145 U
PFBS		15.9	11.4	8.77	15.4	10.0	11.0	10.5	10.6	9.19	4.69
PFPeS		22.7	13.2	10.7	16.8	12.0	13.7	13.3	11.7	11.3	5.30
PFHxS		221 D	143 D	115	162 D	114	130	128	117	114	56.4
PFHpS		57.9	23.6	15.7	26.6	15.7	17.6	20.3	18.5	16.0	7.39
PFOS	3.0^{a}	1800 D	831 D	794 D	918 D	611 D	705 D	668 D	664 D	610 D	326 D
PFNS		2.07	0.852	0.522	0.999	0.595	0.604	1.06	0.622	0.669	0.262
PFDS		1.23	0.537	0.277	0.618	0.382	0.377	0.376	0.44	0.377	0.204 K
PFDoS		0.421	0.137	0.138 U	0.208	0.142	0.145	0.129	0.187	0.147	0.136 U
4:2 FTS		1.45	2.16	1.85	2.67	1.44	1.07	0.658	0.989	0.451	0.579 U
6:2 FTS		39.7 D	1.94 D	73 D	80.8 D	50.8 D	41.2 D	25.7 D	36.3 D	20.2	4.37
8:2 FTS		3.53	26.3	24.9	30.5 D	20.2	17.1	12.6	14.9	10.4	2.07
PFOSA		0.985 K	0.78 K	0.399 K	0.435 K	0.233 K	0.149 U	0.147 U	0.146 U	0.146 U	0.145 U
N-MeFOSA		0.163 U	0.165 U	0.166 U	0.164 U	0.164 U	0.167 U	0.166 U	0.164 U	0.164 U	0.163 U
N-EtFOSA		0.362 U	0.367 U	0.369 U	0.364 U	0.364 U	0.372 U	0.369 U	0.365 U	0.364 U	0.362 U
MeFOSAA		0.145 U	0.147 U	0.147 U	0.146 U	0.146 U	0.149 U	0.147 U	0.146 U	0.146 U	0.145 U
EtFOSAA		0.097 K	0.101 K	0.147 U	0.146 U	0.146 U	0.149 U	0.147 U	0.146 U	0.146 U	0.145 U
N-MeFOSE		1.47 U	1.49 U	1.50 U	1.48 U	1.48 U	1.52 U	1.50 U	1.49 U	1.48 U	1.47 U
N-EtFOSE		1.11 U	1.13 U	1.13 U	1.12 U	1.12 U	1.14 U	1.13 U	1.12 U	1.12 U	1.11 U
SUM of											
Detected PFAS		2244	1115	1074	1309	864	955	895	891	803	410

^a ADEC 18 AAC 75 Table B1 Soil Cleanup Level Migrations to Groundwater (ADEC, 2018)

^b Two samples were collected on Day 8 and sent the laboratory in separate containers. One samples represents the normal samples and one sample represents a duplicate of the normal sample.

Soil concentrations in micrograms per kilogram (µg/kg).

NQ - not quantified

U - undetected

D - dilution data

K - peak detected but did not meet quantification criteria, result reported represents the estimated maximum possible concentration.

K - peak detected but did not meet quantification criteria, result reported represents the estimated maximum possible concentration.

Table 4.8. Soil Results - High Concentration Test - SGS AXYS (Post-oxidation)

Compound	Re-use Criteria	Day 0	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Day 8	3 (QA)
Compound	(μg/kg)	(μg/kg)	(μg/kg)	(μg/kg)	(μg/kg)	(μg/kg)	(μg/kg)	(μg/kg)	(μg/kg)	_	/kg)
PFBA		93.6	63.7	27.2	36.8	21.9	10.6	8.18	9.2	6.03 U	6.36 U
PFPeA		164	112	51.4	69.7	34.4	17.0	14.0	17.4	7.78	3.29
PFHxA		464	302	141	211	95.3	42.5	38.7	41.0	22.1	8.19
PFHpA		74.3	42.7	17.9	26.6	10.5	5.58	4.98	6.20	3.07	1.59 U
PFOA	1.7 ^a	81.3	39.9	16.6	27.5	9.50	5.2	3.52	3.95	1.51 U	1.59 U
PFNA		6.24	3.75	1.32	2.59	1.38 U	1.31 U	1.3 U	1.27 U	1.28 U	1.35 U
PFDA		1.23 U	1.25 U	1.16 U	1.24 U	1.3 U	1.23 U	1.22 U	1.2 U	1.21 U	1.27 U
PFUnA		1.23 U	1.25 U	1.16 U	1.24 U	1.3 U	1.23 U	1.22 U	1.2 U	1.21 U	1.27 U
PFDoA		1.23 U	1.25 U	1.16 U	1.24 U	1.3 U	1.23 U	1.22 U	1.2 U	1.21 U	1.27 U
PFTrDA		1.23 U	1.25 U	1.16 U	1.24 U	1.3 U	1.23 U	1.22 U	1.2 U	1.21 U	1.27 U
PFTeDA		1.23 U	1.25 U	1.16 U	1.24 U	1.3 U	1.23 U	1.22 U	1.2 U	1.21 U	1.27 U
PFBS		12.7	8.29	10.6	18.5	9.6	9.76	9.35	11.1	6.55	2.57
PFPeS		20.3	13.2	15.3	21.2	12.6	12.5	13.1	15.4	8.81	4.67
PFHxS		233	137	167	216	153	126	147	150	104	57.7
PFHpS		23.0	9.52	9.71	12.7	12.8	11.5	12.8	10.3	7.8	4.6
PFOS	3.0^{a}	1410	732	555	732	795	638	705	563	553	332
PFNS		1.53 U	1.56 U	1.45 U	1.55 U	1.62 U	1.54 U	1.53 U	1.49 U	1.51 U	1.59 U
PFDS		1.53 U	1.56 U	1.45 U	1.55 U	1.62 U	1.54 U	1.53 U	1.49 U	1.51 U	1.59 U
PFDoS		1.53 U	1.56 U	1.45 U	1.55 U	1.62 U	1.54 U	1.53 U	1.49 U	1.51 U	1.59 U
SUM of Detected PFAS		2582	1464	1013	1375	1155	879	957	828	713	413

Soil concentrations in micrograms per kilogram (µg/kg).

U - undetected

QA - quality assurance sample

^a ADEC 18 AAC 75 Table B1 Soil Cleanup Level Migrations to Groundwater (ADEC, 2018)

Table 4.9. Soil Results - High Concentration Test - CSM (Total)

	Re-use										
Compound	Criteria	Day 0	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Da	y 8
	(µg/kg)	(µg/kg)	(µg/kg)	(µg/kg)	(µg/kg)	(µg/kg)	(µg/kg)	(µg/kg)	(µg/kg)	(μg	/kg)
PFBA		14.5	6.38	9.09	24.9	7.55	11.8	8.42	< 2400	< 2400	12.6
PFPeA		< 1200	< 1200	< 1200	< 1200	< 1200	< 1200	< 1200	< 1200	< 1200	< 1200
PFHxA		70.1	53.4	33.1	39.9	38.3	12.5	7.11	17.0	6.80	3.12
PFHpA		12.3	7.56	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400
PFOA	1.7ª	30.0	18.7	8.89	10.4	7.22	< 600	< 600	< 600	< 600	< 600
PFNA		< 2400	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400
PFDA		< 2400	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400
PFUnA		< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60
PFDoA		< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120
PFTrDA		< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120
PFTeDA		< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120
PFBS		56.7	23.9	18.2	23.0	23.3	20.1	16.1	18.0	14.2	6.95
PFPeS		107	29.9	26.1	34.1	34.8	31.6	22.0	23.4	18.5	9.68
PFHxS		689	360	319	347	369	305	235	308	243	112
PFHpS		68.7	32.8	27.2	30.6	30.6	27.3	18.2	23.7	21.3	8.05
PFOS	3.0^{a}	3227	1406	1095	1216	1386	1086	803	1157	820	415
PFNS		27.0	8.27	5.73	6.84	7.78	6.72	4.76	6.51	4.05	1.88
PFDS		9.59	3.16	2.13	2.56	2.87	2.14	1.73	2.48	1.49	0.557
PFDoS		3.19	1.19	0.706	0.775	1.12	0.703	0.676	0.832	0.649	0.209
4:2 FTS		4.18	1.74	4.30	3.71	2.42	2.90	< 60	< 60	< 60	< 60
6:2 FTS		30.2	73.1	65.2	198	93.1	66.8	22.6	113	14.8	8.94
8:2 FTS		< 240	26.5	35.4	23.4	68.5	< 240	< 240	16.4	< 240	< 240
PFOSA		1.78	1.49	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400	< 2400
Me-FOSA		< 1200	< 1200	< 1200	< 1200	< 1200	< 1200	< 1200	< 1200	< 1200	< 1200
Et-FOSA		< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60
FOSAA		< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120
MeFOSAA		< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120
EtFOSAA		< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120	< 120
SUM of Detected PFAS		4351	2054	1649	1962	2072	1573	1140	1686	1145	579

^a ADEC 18 AAC 75 Table B1 Soil Cleanup Level Migrations to Groundwater (ADEC, 2018)

Soil concentrations in micrograms per kilogram (µg/kg).

< - undetected (below limit of quantitation)

CSM - Colorado School of Mines

QA - quality assurance sample

ND - none detected

ADEC = Alaska Department of Environmental Conservation

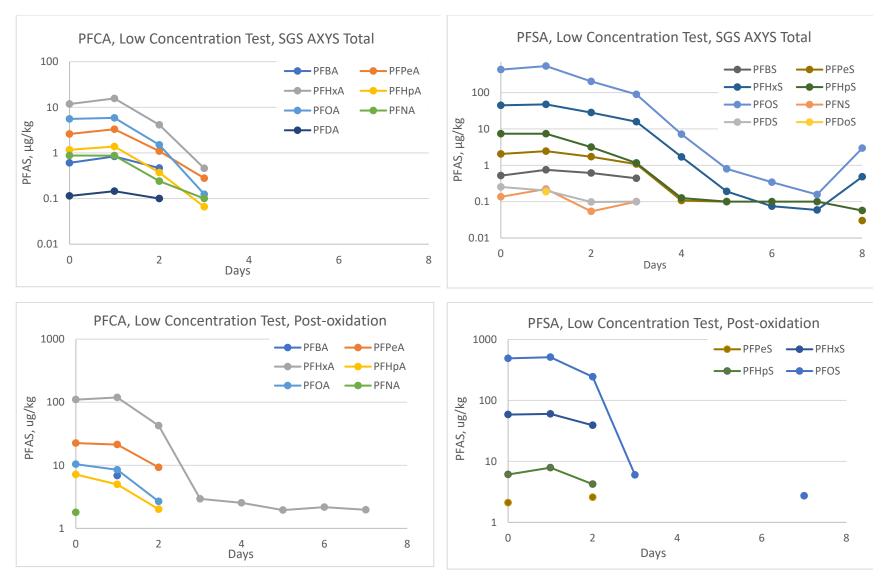


Figure 4.1. PFCA and PFSA Concentration Trends - Low Concentration Test

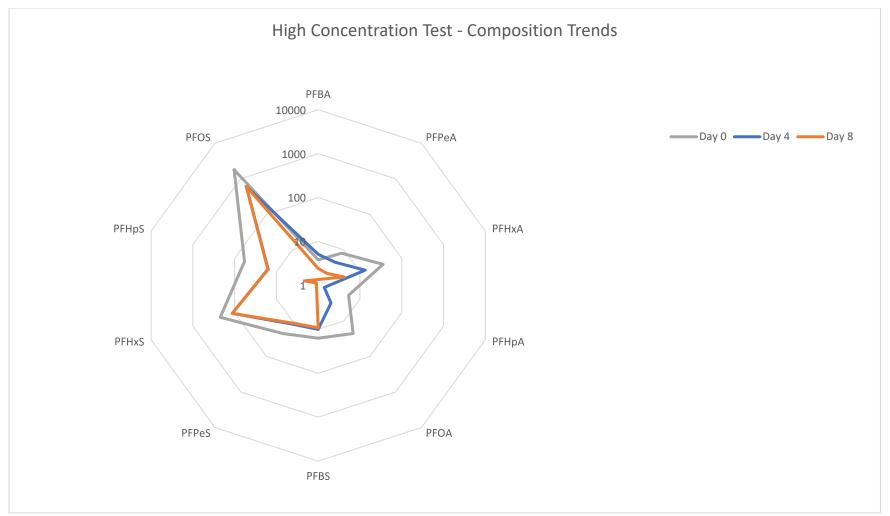


Figure 4.2. Changes in PFAS Composition Over Time

PFAS - per- and poly-fluoroalkyl substances

Table 4.8 results indicate that seven PFAS (PFPeA, PFHxA, PFBS, PFPeS, PFHxS, PFHpS and PFOS) were detected in the post-treatment sample at concentrations from 2.57 μg/kg to 332 μg/kg. Removal of PFOA was to below the detection limit and exceeded 98.1 percent. Removal of PFOS was 68.6 percent and removal of total PFAS was 78.2 percent (Table 4.6).

Table 4.9 results indicate that 11 PFAS (PFBA, PFHxA, PFBS, PFPeS, PFHxS, PFHpS, PFOS, PFNS, PFDS, PFDoS, and 6:2 FtS) were detected in the post-treatment sample at concentrations ranging from 0.209 μ g/kg to 415 μ g/kg. Removal of PFOA was to below the detection limit and exceeded 98.1 percent. Removal of PFOS was 74.6 percent and removal of total PFAS was 73.7 percent (Table 4.6).

Figure 4.3 presents pre- and post-TOPA oxidation PFAS concentrations over the High-Concentration Test based on daily composite samples. Depletion of PFAS compounds over time are also shown on radar plots included as Figure 4.2. The data shows that the PFCA were removed better than the PFSA and that the PFOA concentration was reduced to below the ADEC criteria. PFOS concentrations initially decreased, then decreased and stabilized for the last 3 days.

4.1.3 Conclusions

PFAS removal from soil met ADEC soil to groundwater criteria and the treatment goal for the Low-Concentration Test. In the Low-Concentration test, the temperature of the treatment unit was maintained at or above the target temperature of 350°C and treatment was achieved in 4 days. Overall removal exceeded 99 percent total PFAS and residual PFAS was limited to very low concentrations of PFHxS and PFOS. PFOA and all other compounds tested in the Environmental Laboratory Approval Program-certified laboratory were non-detectable at less than 1 μ g/kg.

PFAS removal in the High-Concentration Test did not meet treatment goals. In this test, the temperature of the treatment unit did not meet the planned temperature, with the highest average temperature slightly less than 350°C, and most other temperatures well below the 350°C objective. As observed in the bench-scale test (included in the Demonstration Plan [Appendix A]), although PFCAs were treated at a lower temperature, PFSA removal is minimal below 350°C. Not attaining and maintaining the proper temperature limited treatment. As in the bench-scale test, PFCAs were mostly removed with lessor removal of PFSAs. The result was 73 percent removal of PFAS, a similar removal of PFOS, and reduction of only PFOA to less than the ADEC soil to groundwater criteria.

4.2 OBJECTIVE 2: IMPROVE UNDERSTANDING OF PFAS IN THE TREATMENT PROCESS

In this section, data collected during the tests is used to evaluate the thermal desorption and vapor treatment processes. This includes presenting the mass balance and the water balance and results.

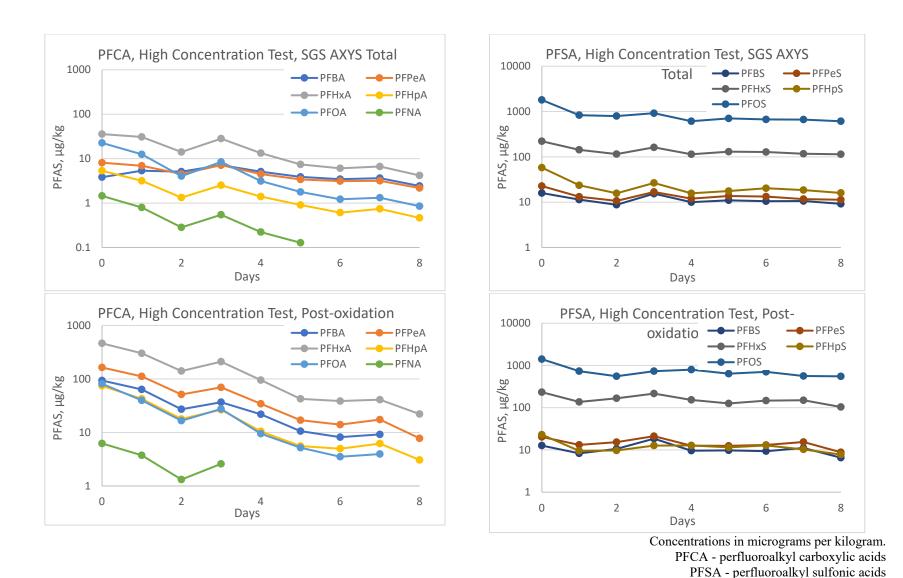


Figure 4.3. PFCA and PFSA Concentration Trends - High Concentration Test

4.2.1 PFAS Mass Balance

To conduct the mass balance evaluation, samples were collected from the interior bin surface (wipe samples), VGAC, C-18 cartridges, and condensate. These data in conjunction with the soil results were used to estimate mass captured in the system using the equations presented in Section 3.5.1. In addition, a water balance was conducted to evaluate the amount of the initial soil moisture recovered in the system.

4.2.1.1 Low-Concentration Test

Sample results from the Pre- and Post-Test wipes, virgin and post-test VGAC, and post-test condensate are presented in Tables 4.10 through 4.12, respectively. Daily C-18 cartridge sampling results for the Low-Concentration Test are presented in Table 4.13.

- Table 4.10 shows that only PFBA remained on the interior walls of the treatment unit after treatment (Figure 3.1). Two additional PFAS compounds were present in the system before treatment, suggesting the unit was cleaner after use than before use.
- \bullet Table 4.11 shows that 424 $\mu g/kg$ total PFAS was adsorbed to the VGAC after treatment.
- Table 4.12 shows that 20,201 ng/L total PFAS was retained in the condensate.
- Only PFBA was measurable in the C-18 cartridge samples (Table 4.13) and at relatively low concentrations.

The various weighted average molecular weights were calculated and are tabulated in Tables B.1 through B.7 (Appendix B). The moles of each compound per unit was calculated by dividing the result for each compound by the molecular weight of the compound; the moles of detections were summed, and the total detected was divided by the total moles to estimate the weight average molecular weight.

To conduct the water balance, the initial amount of water was estimated using the moisture content of the soil and estimated mass of soil (Table 4.14). The amount of water recovered in the condensate, VGAC, and residual soil were then calculated. An estimated 233 L of water was present in the soil before treatment. At the end of treatment, 193 L had been recovered indicating that 83 percent of the moisture in the system was captured.

The mass balance of the system was calculated as shown on Table 4.14. Initially, 2.11 grams of soil was in the test batch. After testing, only 2 percent of the total mass present in the soil before the test (0.037 g) was recovered. Most of the recovered mass was in the VGAC (0.019 g) and residual soil (0.014 g) with lesser amount in the condensate (0.003 g). The amount on the interior surface of the TDU was insignificant.

While the treated soil in the Low-Concentration Test met its treatment goal, we were not able to quantify recover sufficiently for a successful mass balance. Our goal of accounting for 75 percent of mass in the for the mass balance was not achieved.

Table 4.10. Pre- and Post- Wipe Concentrations - Battelle

		Low Concentrati	ion	Wipe Results		Hig	gh Concentr	atio	on Wipe Results	
	Pre-T	est Results		Post-Test R	Results	Pre-Test R	esults		Post-Test R	esults
	07/	05/2019		07/15/20	19	7/16/20	19		7/25/201	19
	Result			Result		Result			Result	
Analyte	(ng/Wipe)	ng/m2		(ng/Wipe)	ng/m2	(ng/Wipe)	ng/m2		(ng/Wipe)	ng/m2
PFBA	4.72 J	472		1.33 J	133	0.2 UT	U		2 U	U
PFPeA	2 U	U		2 U	U	0.2 UT	U		2 U	U
PFHxA	0.69 J	69		2 U	U	0.2 UT	U		2 U	U
PFHpA	2 U	U		2 U	U	0.2 UT	U		2 U	U
PFOA	2 U	U		2 U	U	0.2 UT	U		2 U	U
PFNA	2 U	U		2 U	U	0.2 UT	U		2 U	U
PFDA	2 U	U		2 U	U	0.2 UT	U		2 U	U
PFUnA	2 U	U		2 U	U	0.2 UT	U		2 U	U
PFDoA	1 U	U		1 U	U	0.1 UT	U		1 U	U
PFTrDA	2 U	U		2 U	U	0.2 UT	U		2 U	U
PFTeDA	4 U	U		4 U	U	0.4 UT	U		4 U	U
PFBS	2 U	U		2 U	U	0.2 UT	U		2 U	U
PFPeS	4 U	U		4 U	U	0.4 UT	U		4 U	U
PFHxS	1 U	U		1 U	U	0.1 UT	U		1 U	U
PFHpS	2 U	U		2 U	U	0.2 UT	U		2 U	U
PFOS	0.79 J	79		2 U	U	0.2 UT	U		0.72 J	72
PFNS	4 U	U		4 U	U	0.4 UT	U		4 U	U
PFDS	1 U	U		1 U	U	0.1 UT	U		1 U	U
4:2FTS	2 U	U		2 U	U	0.2 UT	U		2 U	U
6:2FTS	4 U	U		4 U	U	0.4 UT	U		4 U	U
8:2FTS	4 U	U		4 U	U	0.4 UT	U		4 U	U
PFOSA	2 U	U		2 U	U	0.2 UT	U		2 U	U
MeFOSAA	5 U	U		5 U	U	0.5 UT	U		4 U	U
EtFOSAA	4 U	U		4 U	U	0.4 UT	U		5 U	U
SUM of Detected PFAS	6.20	620		1.33	133	U	0		0.72	72

ng - nanogram U - Undetected

m2 - square meter

J - Value is estimated

Table 4.11. V-GAC Test Results - Battelle

Compound	Day 0 (Low) (μg/kg)	Day 8 - Low (μg/kg)	Day 0 (High) (μg/kg)	Day 8 - High (μg/kg)
PFBA	1.47 U	7.4	1.14 UT	10.32
PFPeA	1.47 U	13.1	0.46 JT	3.55 J
PFHxA	1.47 U	28.7	1.14 UT	12.55
PFHpA	1.47 U	22.3	1.14 UT	1.31 U
PFOA	1.47 U	69.9	1.14 UT	1.31 U
PFNA	1.47 U	12.0	1.14 UT	1.31 U
PFDA	1.47 U	12.1	1.14 UT	1.31 U
PFUnA	1.47 U	1.14 U	1.14 UT	1.31 U
PFDoA	0.74 U	0.57 U	0.57 UT	0.65 U
PFTrDA	1.47 U	1.14 U	1.14 UT	1.31 U
PFTeDA	2.94 U	2.29 U	2.27 UT	2.61 U
PFBS	1.47 U	69.6	1.14 UT	1.31 U
PFPeS	2.94 U	53.8	2.27 UT	2.61 U
PFHxS	0.74 U	131.8 D	0.57 UT	0.65 U
PFHpS	1.5 U	1.1 U	1.1 UT	1.3 U
PFOS	1.47 U	363 D	1.14 UT	1.31 U
PFNS	2.94 U	2.29 U	2.27 UT	2.61 U
PFDS	0.74 U	0.57 U	0.57 UT	0.65 U
4:2 FTS	1.47 U	1.14 U	1.14 UT	1.31 U
6:2 FTS	2.94 U	2.21 J	2.27 UT	2.61 U
8:2 FTS	2.94 U	2.29 U	2.27 UT	2.61 U
PFOSA	1.47 U	0.89 J	1.14 UT	1.31 U
NMeFOSAA	3.68 U	2.86 U	2.84 UT	3.27 U
NEtFOSAA	2.94 U	2.29 U	2.27 UT	2.61 U
SUM of Detected PFAS	U	424	0.46	26

Concentrations in micrograms per kilogram ($\mu g/kg$).

- U undetected
- T holding time exceeded
- D Diluted sample

Table 4.12. Condensate Results - Battelle

	Low-Concentration Test	High-Concentration Test
Analyte	Result (ng/L)	Result (ng/L)
PFBA	0.42 U	91.1 TD
PFPeA	305.15 D	105.55 TD
PFHxA	3006.96 D	360.3 TD
PFHpA	124.66	76.42 TD
PFOA	305.66 D	237.49 TD
PFNA	47.84	2.4 JT
PFDA	7.71	7.76 T
PFUnA	1.61 J	2.05 JT
PFDoA	1.06 J	1.62 JT
PFTrDA	0.42 U	0.43 UT
PFTeDA	0.84 U	0.86 UT
PFBS	27.87	85.52 T
PFPeS	422.99	66.26 T
PFHxS	1104.36 D	807.17 TD
PFHpS	192.66 D	601.17 TD
PFOS	14575.58 D	57819.48 TD
PFNS	2.95 Ј	79.51 T
PFDS	1.04 J	58.04 T
4:2FTS	0.42 U	3.49 JT
6:2FTS	42.32 D	772.95 TD
8:2FTS	30.19 D	476.64 TD
MeFOSAA	1.68 U	1.72 UT
EtFOSAA	0.84 U	0.86 UT
SUM of Detected PFAS	20,201	61,655

ng/L - nanogram per liter

U - Undetected

J - Value is estimated

D - dilution data

T - holding time exceeded

Table 4.13. C-18 Cartridges - Low Concentration Test - Battelle

Compound	Day 1	Day 2 Day 3		Day 4	Day 5	Day 6	Day 7	Day 8
Compound	(ng)	(ng)	(ng)	(ng)	(ng)	(ng)	(ng)	(ng)
PFBA	0.11 J	0.1 J	0.08 J	0.16 J	0.11 J	0.14 J	0.21 J	0.09 J
PFPeA	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U
PFHxA	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U
PFHpA	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U
PFOA	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U
PFNA	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U
PFDA	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U
PFUnA	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U
PFDoA	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U
PFTrDA	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U
PFTeDA	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U
PFBS	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U
PFPeS	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U
PFHxS	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U
PFHpS	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U
PFOS	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U
PFNS	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U
PFDS	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U
4:2FTS	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U
6:2FTS	0.4 U	0.4 U	0.2 U	0.4 U				
8:2FTS	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U
PFOSA	0.2 U	0.2 U	0.4 U	0.2 U				
MeFOSAA	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
EtFOSAA	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U	0.4 U

Cartridge results measured in nanograms (ng) per cartridge.

U - undetected

J - Concentration is estimated

Table 4.14. Mass Balance Calculations

Item	Low Conc.	Unit	High Conc.	Unit	Source/Calculation/Comment
Starting Condition	Conc.	Unit	Conc.	Cilit	Source/Calculation/Comment
Mass of Soil	9,000	lb	9,000	lb	Approximate
Mass of Soil	4,086	kg	4,086	kg	Convert pounds to kilograms (0.454 kg/lb)
Soil Moisture, content	5.7	%	8.0	%	SGS AXYS Laboratory Report (Appendix B)
Soil Moisture, volume ⁽¹⁾	233	L	327	L	Moisture Content * Mass (1 kg = 1 L)
PFAS in Soil, concentration	516	μg/kg	2,244	μg/kg	Tables 4.3 (Low) and 4.6 (High)
PFAS in Soil, mass	2.11	g	9.17	g	Concentration (μg/kg) * Soil Mass (kg)/ 1E6 (μg/g)
PFAS Average Molecular Weight (MWt)	476	g/mole	472	g/mole	Molecular weight calculation sheet
PFAS in Condensate					
Volume (gallons)	45	gal	48	gal	Measured
Volume (liters) ⁽¹⁾	170	L	181	L	Convert gallons to liters (3.78 L/gal)
Condensate Recovery	73	%	56	%	Condensate Volume (L)/ Soil Moisture Volume (L)
Total PFAS Concentration	20,201	ng/L	61,655	ng/L	Table 4.11
Total PFAS Mass ⁽²⁾	0.003	g	0.011	g	Concentration (ng/L) * Volume (L)/1E9 (ng/g)
MWt of Total PFAS - Condensate	441	g/mole	492	g/mole	Molecular weight calculation sheet
Corrected PFAS in Condensate, mass	0.004	g	0.011	g	Total PFAS Mass/avg MWt * Baseline MWt
PFAS in VGAC					
Mass of VGAC	100	lb	100	lb	Label
Mass of VGAC	45,400	g	45,400	g	Convert lb to g (454 g/lb)
Total PFAS, concentration	424	ng/g		ng/g	Table 4.10
Total PFAS, mass ⁽³⁾	0.019	g	0.001	g	Concentration (ng/g) * Mass (g)/ 1E9 (ng/g)
Moisture in VGAC	32	%	26	%	Battelle Laboratory Report (Appendix B)
Moisture in VGAC ⁽¹⁾	14.5	L	11.8	L	Moisture (L) * Mass (g)/ 1000 g/L
MWt of Total PFAS - VGAC	363	g/mole	298	g/mole	Molecular weight calculation sheet
Corrected PFAS in VGAC, mass	0.025	g	0.002	g	Total PFAS Mass/avg MWt * Baseline MWt
PFAS on TDU Surface			•		
Surface Area of TDU	118	sf	118	sf	Bin (66-in x 47-in x 48-in) with 15-in diameter core
Total Surface Area	11	m^2	11	m^2	Convert sf to m^2 (10.76 sf/ m^2)
Total PFAS in Wipe, concentration	133	ng/m ²	72	ng/m ²	Table 4.9
Total PFAS on surface, mass ⁽⁴⁾	3.3E-06	g	1.8E-06	g	Concentration (ng/m ²) * Area (m ²)
MWt of Total PFAS - TDU Surface	213	g/mole	499	g/mole	Molecular weight calculation sheet
Corrected PFAS on TDU Surface, mass	7.4E-06	g	1.7E-06	g	Total PFAS Mass/avg MWt * Baseline MWt
PFAS in Residual Soil					
Soil Mass	4086	kg	4086	kg	Approximate (from starting conditions)

	Low		High		
Item	Conc.	Unit	Conc.	Unit	Source/Calculation/Comment
Residual Soil Moisture, content	0.21	%	0.00	%	SGS AXYS Laboratory Report (Appendix B)
Residual Soil Moisture, volume ⁽¹⁾	9	L	0	L	Moisture * Mass (1 kg = 1 L)
Total PFAS, Concentration	3.53	μg/kg	803	μg/kg	Tables 4.3 (Low) and 4.6 (High)
Total PFAS Soil, Mass ⁽⁵⁾	0.014	g	3.28	g	Concentration (μg/kg) * Soil Mass (kg)/ 1E6 (μg/g)
MWt of Total PFAS - Residual Soil	481	g/mole	472	g/mole	Molecular weight calculation sheet
Corrected PFAS in Residual Soil, mass	0.0143	g	3.2815	g	Total PFAS Mass/avg MWt * BaselineMWt
Water Balance	1		T		
Initial Moisture In Soil		L	327	L	
Condensate Volume	170			L	(1) Values used in Water Balance
VGAC Moisture Volume	14.5			L	(1) Various used in Water Daranee
Residual Soil Moisture Volume		L		L	
Recovered Soil Moisture (total)	193	L	193	L	Sum of Condensate, VGAC, & Residual Soil Volumes
Soil Moisture Recovery (percent)	83	%	59	%	Sum of Residuals / Initial Moisture in Soil
MASS BALANCE					
Initial Total PFAS in Soil:	2.11	g	9.17	g	Starting Conditions
Uncorrected					
Total PFAS mass in Condensate	0.003	g	0.011	g	(2)
Total PFAS mass in VGAC	0.019	g	0.001	g	(3)
Total PFAS mass on TDU surface	3.33E-06	g	1.80E-06	g	(4)
Total PFAS in Residual Soil	0.014	g	3.28	g	(5)
Total PFAS Removal in Soil	99%		64%		1 - (Total PFAS Residual Soil / Initial PFAS in Soil)
Total PFAS Recovered	0.037	g	3.29	g	Sum of mass Condensate, VGAC, TDU, Residual Soil
Percent of Total PFAS Recovered	2%		36%		Total PFAS Recovered / Initial Total PFAS in Soil
Corrected for Average MWt					
Corrected PFAS mass in Condensate	0.004	g	0.011	g	(2) Correction calculated above
Corrected PFAS mass in VGAC	0.025	g	0.002	g	(3) Correction calculated above
Corrected PFAS mass on TDU surface	7.42E-06	g	1.70E-06	g	(4) Correction calculated above
Corrected PFAS in Residual Soil	0.014	g	3.28	g	(5) Correction calculated above
Corrected PFAS Removal in Soil	99%		64%		1 - (Total PFAS Residual Soil / Initial PFAS in Soil)
Corrected PFAS Recovered	0.043	g	3.29	g	Sum of mass Condensate, VGAC, TDU, Residual Soil
Percent of Corrected PFAS Recovered	2%		36%		Total PFAS Recovered / Initial Total PFAS in Soil

Item	Low Conc.	Unit	High Conc.	Unit	Source/Calculation/Comment
Change in Mass due to change in Molecular Weight					
Change in Mass	0.006		0.001		PFAS Recovered (Corrected-Uncorrected)
% Change	16%		0.041%		Change in mass due to shift in molecular weight

4.2.1.2 High-Concentration Test

Daily C-18 cartridge sampling results for the High-Concentration Test are presented in Table 4.15. Results from the C-18 cartridges, Pre- and Post-Test wipes, virgin and post-test VGAC, and post-test condensate samples are summarized as follows:

- Table 4.10 shows that only PFOS at a low, estimated concentration was detected on the interior of the treatment bin (Figure 3.1).
- Table 4.11 shows that 23 μg/kg total PFAS was adsorbed to the VGAC (this is an order of magnitude less than the amount retained during the Low-Concentration Test).
- Table 4.12 shows that 61,655 ng/L total PFAS was retained in the condensate.
- As noted in the Low-Concentration Test results, only low concentrations of PFBA was measurable in the C-18 cartridge samples (Table 4.15).

Based on initial soil moisture content, 327 L of water was estimated in the High-Concentration Test batch. Of that, 193 L were recovered (Table 4.13) indicating that 59 percent of the moisture in the system was captured.

The mass balance of the system was calculated as shown on Table 4.14. Although, 36 percent of the total mass present in the soil before the test was recovered, most of the "recovered" PFAS mass (3.31 g) was retained in the partially treated soil.

As with the Low-Concentration Test, recovery of PFAS was far less than the goal of 75 percent and this objective was not met.

4.2.2 Comparison of Total to TOPA Analyses

4.2.2.1 Low-Concentration Test

Post-oxidation results (Table 4.4) were slightly higher than results from standard analyses (Tables 4.3). The total Day-0 concentration of post-oxidation compounds detected was 709 μ g/kg compared to 516 μ g/kg total PFAS. Figure 4.3 presents the composition of the initial total and post-oxidation Low-Concentration Test results using the PFCAs and PFSAs identified in Section 3.5. The figure shows that the increase was primarily due to an increase in the PFCAs post-oxidation.

4.2.2.2 High-Concentration Test

Post-oxidation results (Table 4.8) were similar to results from standard analyses (Table 4.7). The total concentration of post-oxidation compounds detected was 2,582 μ g/kg compared to 2,244 μ g/kg total PFAS. Figure 4.4 presents the composition of the initial total and post-oxidation High-Concentration Test results using the PFCAs and PFSAs identified in Section 3.5. The figure shows that there was approximately an order of magnitude increase in the PFCAs post-oxidation accounting for the concentration increase.

Table 4.15A. C-18 Cartridges - High Concentration Test Results - Battelle

Compound	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Day 8
Compound	(ng)	(ng)	(ng)	(ng)	(ng)	(ng)	(ng)	(ng)
PFBA	0.2 UT	0.2 UT	0.14 JT	0.08 JT	0.11 JT	0.1 JT	0.2 UT	0.13 JT
PFPeA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFHxA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFHpA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFOA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFNA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFDA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFUnA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFDoA	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT
PFTrDA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFTeDA	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT
PFBS	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFPeS	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT
PFHxS	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT
PFHpS	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFOS	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFNS	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT
PFDS	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT
4:2FTS	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
6:2FTS	0.4 UT	0.22 JT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT
8:2FTS	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT
PFOSA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
MeFOSAA	0.5 UT	0.5 UT	0.5 UT	0.5 UT	0.5 UT	0.5 UT	0.5 UT	0.5 UT
EtFOSAA	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT

Cartridge results measured in nanograms (ng) per cartridge.

U - undetected

T - holding time exceeded

Table 4.15B. C-18 Effluent - High Concentration Test Results - Battelle

Compound	Day 1 (µg/kg)	Day 2 (μg/kg)	Day 3 (μg/kg)	Day 4 (μg/kg)	Day 5 (μg/kg)	Day 6 (μg/kg)	Day 7 (μg/kg)	Day 8 (μg/kg)
PFBA	0.2 UT	0.2 UT	0.2 UT	0.12 JT	0.08 JT	0.18 JT	0.2 UT	0.2 UT
PFPeA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFHxA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFHpA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFOA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFNA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFDA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFUnA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFDoA	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT
PFTrDA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFTeDA	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT
PFBS	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFPeS	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT
PFHxS	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT
PFHpS	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFOS	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
PFNS	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT
PFDS	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT	0.1 UT
4:2FTS	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
6:2FTS	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT
8:2FTS	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT
PFOSA	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT	0.2 UT
MeFOSAA	0.5 UT	0.5 UT	0.5 UT	0.5 UT	0.5 UT	0.5 UT	0.5 UT	0.5 UT
EtFOSAA	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT	0.4 UT

Cartridge effluent results measured in micrograms per kilogram (µg/kg).

U - undetected

T - holding time exceeded J - concentration is estimated



TOPA - total oxidizable precursor assay

Figure 4.4. Comparison of Total and TOPA Results in Initial Soil

4.2.3 Changes in PFAS Concentrations Over Time

4.2.3.1 Low-Concentration Test

Figure 4.2 shows general changes in the composition PFAS in the test soil during three days of the test, Days 0, 4, and 8. Composition is based on the PFCAs and PFSAs identified in Section 3.5. During the test, concentrations increased by 23 percent from Day 0 to Day 1, and then rapidly declined until 99.9 percent was removed on Day 6 (Table 4.6). Concentrations then varied slightly and on Day 8, 99.3 percent of PFAS was removed. After Day 4, soil met ADEC standards; this objective was maintained over the remainder of the test. Molecular weight did not vary greatly over the test. Near the end of the test when most of the PFAS was depleted, the molecular weight varied more depending on which compounds were detected. The final molecular weight was higher than the initial molecular weight based on a relatively high detection of PFOS on Day 8.

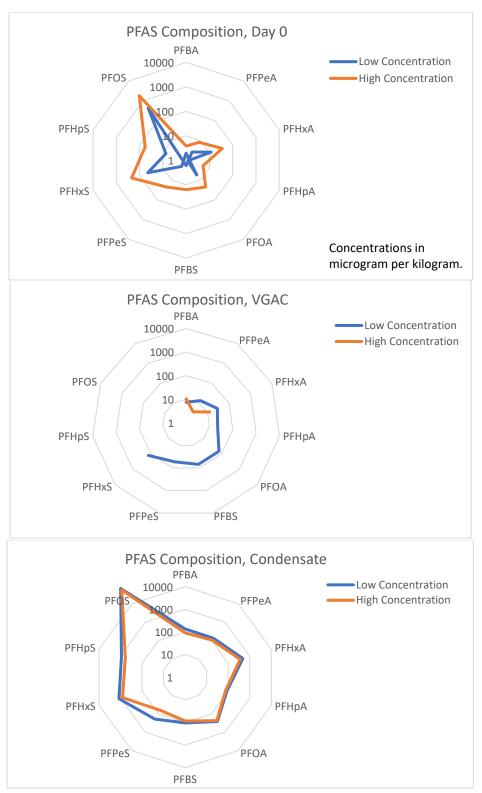
4.2.3.2 <u>High-Concentration Test</u>

Figure 4.2 shows general changes in the composition PFAS in the test soil during three days of the test, Days 0, 4, and 8. Composition is based on the PFCAs and PFSAs identified in Section 3.5. During the test, composition of the sample shifted as the PFCAs were largely depleted (86 percent removal); although, these were a small portion of the total mass. The PFSAs show a 64 percent reduction. Other compounds, including FtS and PFOSA, were also depleted, but to a lesser extent (32 percent removal [Table 4.6]) than the PFCAs and PFSA. FtS concentrations increased over the first three days of the test before declining to less than their original concentrations (Table 4.6). This likely reflects some oxidative conversion to the intermediate FtS. However, the overall FtS concentrations are small and this did not contribute significantly to the overall removal rates. Total PFAS removal reflected the dominant PFSA and was 64 percent during the test. The molecular weight of the remaining PFAS during the test was stable, suggesting little fractioning based on molecular weight or loss of mass due to degradation (Appendix B).

4.2.4 Treatment by VGAC

The amount of PFAS recovered in the VGAC, as presented in the mass balance, was very small, with 0.025 g of 2.11 g (1.1 percent) recovered in the Low-Concentration Test and 0.002 of 9.17 g (0.02 percent) recovered in the High-Concentration Test (Table 4.14). Composition of the captured PFAS is shown on Figure 4.5. One objective of the study was to capture 90 percent of the PFAS on the GAC, which did not occur. It was anticipated that the majority of the PFAS in the soil would be recovered in the VGAC and that did not happen. One explanation for the low recovery of PFAS in the VGAC is the PFAS did not made it to the VGAC.

PFAS was recovered in the condensate, with relatively high concentrations detected in both the Low- and High-Concentration Test samples. Composition of the captured PFAS is shown on Figure 4.5. The amount of PFAS measured in the condensate implies that significant PFAS made it to the condenser. Very little PFAS was detected in the C-18 cartridges, which confirms that little of the PFAS made it to the VGAC. That PFAS that was detected in the C-18 cartridges was PFBA, which is a low molecular weight carboxylic acid; it is expected that this is the most mobile of the compounds tested.



VGAC - vapor-phase granular activated carbon PFAS - per- and poly-fluoroalkyl substances

Figure 4.5. VGAC and Condensate Composition Compared to Initial Soil

On the other hand, there is pretty good evidence to suggest the PFAS made it to the condenser, including the recovery of high concentrations of PFAS in the condensate and its close proximity to the treatment unit. Because the condenser cools the air stream to remove water, it also cools the PFAS. Therefore, it can be assumed that the missing PFAS is in the condenser.

Much less PFAS was recovered on the VGAC during the High-Concentration Test, even though the mass of PFAS removed, about 6 g, was more than the total PFAS in the Low-Concentration Test (3 g). We believe this was because the High-Concentration Test did not achieve the desired temperature, which exacerbated the loss of PFAS in the condenser.

4.2.5 Fate of PFAS in Residuals

Figure 4.5 presents the composition of PFAS using the PFCAs and PFSAs identified in Section 3.5 in the initial soil, condensate, and VGAC. Because only one compound was detected on the C-18 cartridges (Tables 4.13 and 4.15), the composition of PFAS in the C-18 cartridges is not shown. Wipe samples also detected very little PFAS and are not shown. In general, the baseline contamination detected in the initial wipe sample and carry over between tests was insignificant, with test results indicating cleaner surfaces after use (Table 4.10).

The compounds captured in the two VGAC samples differ from the original soil compositions. Although a mix of PFCAs and PFSAs were detected in the Low-Concentration Test results, Figure 4.5 shows that primarily PFCAs were retained on the VGAC with three lighter PFSAs. Low concentrations of only PFCAs were detected in the High-Concentration VGAC sample. Results indicate modest capture of PFAS from the Low-Concentration Test and far less capture from the High-Concentration Test.

In the condensate, as much as 20,201 ng/L was recovered from the Low-Concentration Test and 61,655 ng/L was recovered from the High-Concentration Test (Table 4.12). A variety of compounds were recovered from both test and their compositions were similar to each other and showed similar peaks as the initial soil (PFHxA, PFOA, PFHxS, and PFOS) (Figure 4.5). The similarity of the concentrations and composition, despite the differences in the test conditions, suggests the condensate was saturated with PFAS.

Both the VGAC and GAC used to treat the condensate will be returned to the vender for regeneration. Presumably the PFAS adsorbed to the GAC will be destroyed in this process. Conclusions

The mass balance found far less PFAS than anticipated in treatment residuals. It was anticipated that most of the mobilized PFAS would be found in the VGAC, with a smaller amount in the condensate. Instead, small amounts were found in both the VGAC and condensate. The recovery of PFAS in condensate, VGAC, C-18 cartridges, and wipe samples was less than 3 percent, with 2.1 percent recovered from the Low-Concentration Test and less than 1 percent recovered from the High-Concentration Test (Table 4.14). Although the PFAS captured on VGAC during the Low-Concentration Test was representative of the compounds present in the soil sample (Figure 4.5), only lower molecular weight PFCAs were detected, likely reflecting the compounds mobilized at the lower temperatures. PFAS was also largely absent from the C-18 cartridges used to monitor vapors circulating in the system, with only low molecular weight PFBS detected (Tables 4.13 and 4.15). Lower molecular weight PFCA were expected to be more mobile in a vapor stream than

heavier compounds and PFSAs. The lack of PFAS detected in vapor confirms that PFAS typically did not make it to the VGAC or the cartridge sampling port. Instead, most of the PFAS appears to have condensed in the condenser, or perhaps in the connecting hose. PFAS does not form a classical liquid when condensed but rather a waxy coating. It appears that a portion of the condensed PFAS dissolved in the condenser liquid, most of the rest likely remains in the condenser, and small portion made it to the VGAC, where it was removed.

4.3 OBJECTIVE 3: EVALUATE VAPOR-PHASE TREATMENT

As documented in Table 4.14, very little PFAS was sorbed onto the VGAC. In the Low-Concentration Test, only 1 percent of the initial soil mass was in the VGAC. In the High-Concentration Test, PFAS in VGAC made up less than 0.001 percent of the total mass in the residuals. Only PFBA was detected on the C-18 cartridges (Tables 4.13 and 4.15). VGAC does not appear to be a good choice for PFAS treatment in the vapor phase.

5.0 CONCLUSIONS AND IMPLICATIONS FOR FUTURE RESEARCH

The objectives of this pilot study are as follows:

- 1) To evaluate whether the infrared thermal treatment technology can effectively treat PFAS-contaminated soil IDW such that it allows soil re-use,
- 2) To provide documentation of the fate of PFAS in the treatment process, and
- 3) Demonstrate that the treated PFAS in the vapors can be captured.

5.1 CONCLUSIONS

The testing confirms that when properly implemented thermal desorption is capable of fully treating PFAS contaminated soil when temperatures are maintained above 350°C. In the Low-Concentration Test, where the proper temperature was maintained, effective PFAS treatment was achieved in 4 days. Treatment time seems more a function of the ability to dry the soil and achieve treatment temperature than the amount of PFAS present.

PFAS recovery was very low, with a few percent of the PFAS mass in the sample retained in the VGAC and condensate. Because excellent treatment was achieved in the Low-Concentration Test and vapors were recycled into the treatment unit, it is unlikely that PFAS passed through the GAC without treatment. C-18 cartridge sampling indicates little PFAS was present in the vapor stream when it entered the VGAC units. Wipe samples from the interior of the TDU (outside of the bin) indicate the PFAS was not retained on the surfaces of the TDU. This suggests the PFAS that was volatilized from the soil exited the TDU but did not enter the VGAC. Analysis of condensate samples found high concentrations of PFAS, which suggest PFAS made it to the condenser. Taken together, this suggests the PFAS condensed in the condenser. It is likely the PFAS is still in the condenser, attached to the condenser surfaces.

VGAC does not appear to be a good choice for primary PFAS treatment in the vapor phase; it appears that a high temperature must be maintained to keep the PFAS mobile in an air stream. However, that temperature is unknown and VGAC is flammable and tends to spontaneously

combust at higher temperatures. Where test results indicate that some PFAS remained in the air stream, VGAC appeared to effectively remove this residual.

5.2 **RECOMMENDATIONS**

Because the High-Temperature Test soil was not fully treated since the target temperature was not obtained, the study team proposes to re-treat and test the High-Concentration soil before disposal. The proper temperature will be maintained during treatment. This cost will be borne by the project.

To evaluate whether treatment residuals were retained on the interior of the condensation tank, a wipe sample will be collected from the interior of the condenser. This sample will be tested for PFAS. This cost will be borne by the project.

Additional testing is recommended to better evaluate the fate of residuals in the treatment process. A batch similar to the High-Concentration Test could be treated. The test should ensure that the proper temperature is maintained. Because the study confirmed that PFAS can be removed from soil using thermal desorption, limited soil testing would be needed. Instead, treatment residuals should be tested to better understand the fate of the treated PFAS. A series of wipe tests are recommended at multiple locations along the treatment train to evaluate the location of residual PFAS. A mass balance would be used to quantify the recovery of PFAS.

Because the study determined that capture of PFAS from vapor is not an ideal solution for thermal desorption, the condenser system should be replaced with a wet scrubber, with GAC used to treat the scrubber water. Tests would be conducted to assess where residual PFAS remains in the treatment equipment and then remove the residual PFAS from the treatment equipment, to better manage the residuals.

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7.0 DATED SIGNATURE OF PROJECT LEAD

James W. Hatton

30 September 2019
Date

Principal Technologist, Soil and Groundwater Remediation

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