savronsolutions.com

June 28, 2024

Kirsten Rügge
Civ. Ing., K., PhD
COWI A/S
Parallelvej 2
2800 Kongens Lyngby
Via email: kiru@cowi.com

Subject: **Ex Situ Smoldering (STARx) Bench Treatability Study Report to Treat PFAS-Impacted Soil from Danish Defense Site in Oksbøl, Denmark**

Dear Kirsten:

Savron Solutions (Savron), a Geosyntec Consultants (Geosyntec) company, conducted a treatability study to evaluate performance of ex-situ smoldering (STARx) for treating per- and polyfluoroalkyl Substances (PFAS) present in soil from a firefighter training area (FFTA) at a Danish Defense site in Oksbøl, Denmark (the "Site"). This report presents the scope of work, the results of treatability testing, and recommendations for future phases for work.

SCOPE OF WORK

The treatability study was conducted in accordance with the Work Plan dated 16 April 2024. A bulk soil sample was collected from two locations around the Site in February 2024. Soil was collected by COWI A/S from topsoil both inside and outside of the FFTA, mixed equally to form one composite sample, and shipped to Savron in Cambridge, Ontario. Results of preliminary analysis on the two sample locations are presented in **Appendix A**.

Background on PFAS Treatment by Smoldering

In many STARx applications, the contaminant acts as the fuel source for the self-sustaining smoldering reaction. As PFAS are not contaminants that can support smoldering combustion in and of themselves, a surrogate fuel is required. Numerous fuel surrogates are viable; however, granular activated carbon (GAC) is often selected as low concentrations of GAC can be used to generate the high temperatures required to maximize PFAS destruction. The Site soil was mixed with 50 grams (g) GAC per 1 kilogram (kg) of pre-treatment material (i.e., 5% GAC). This ratio was selected based on the work of Duchesne et al. (2020)[1] which showed this concentration of

---

GAC will produce temperatures greater than 900 degrees Celsius (^oC), the temperature threshold required to thermally decompose PFAS.

Thermal treatment of PFAS will generate a variety of breakdown products, including hydrogen fluoride (HF), short-chain volatile PFAS species, and other products of incomplete destruction (PIDs). PFAS-impacted soils are often impacted with PFAS precursors or may possess some natural inorganic fluoride content. These fluorine sources are not captured with conventional PFAS analysis. Due to these analytical limitations, a mass balance was not an objective of this study.

While the generation of HF and PIDs in process emissions is a good indicator of PFAS destruction, HF is a reactive gas, and PIDs may require further emissions treatment. Controlling the release of HF and PIDs improves site safety and the lifespan of equipment in full-scale operations. Research has found that the implementation of calcium-bearing minerals such as calcium oxide (CaO) limit the generation of HF and PIDs during thermal treatment, instead forming calcium fluoride (CaF_2).[2-4] In this study, CaO amendments were added to the pre-treatment soil.

Experimental Apparatus

The experimental apparatus is illustrated in **Figure 1**. The column used in this study employed a conductive heating coil at the column base, with an integrated air distribution plenum. Injection air was supplied to the base of the plenum using an air compressor and flow controller. At the column top, a hood was placed that directed emissions through two separate emissions trains, meant to capture volatile PFAS and HF.

The HF emissions train consisted of four impingers placed in series. The middle two impingers were filled with 0.1 N sulfuric acid solution (H_2SO_4) meant to capture HF and solubilize as fluoride (F^-) (modified EPA Method 26). The 1st and 4th impingers were empty to serve as moisture knockouts. Following the test, samples were collected and analyzed using a Fluoride Ion Selective Electrode (ISE) Probe.

The PFAS emissions train consisted of two vessels filled with GAC, meant to capture volatile PFAS. Following treatment, samples were collected from each tube and sent to the Royal Military College (RMC) for analysis.

Ignition Protocol

A smoldering combustion test was completed on the as-received soils sample taken from the Site (**Figure 2**). Contaminated soil was mechanically mixed with GAC and CaO at concentrations of 50 grams per kilogram (g/kg) and 10 g/kg, respectively, and loaded into the column. Two mixed batches of soil, weighing approximately 3.18 kilograms (kg) (3 kg soil, 150g GAC and 30 g CaO) were loaded into the column to a height of approximately 33 centimeters (cm) above the heating coil, with a 5 cm clean sand cap placed on top to act as a heat sink. Both mixed soil batches were of an equal mass, and collected from various locations within the bulk soil sample. A conductive heating source was used to preheat the soil adjacent to the heater and initiate the smoldering combustion process. Once a target ignition temperature of 400°C was reached, injection air was turned on and maintained at a fixed air flux of 5 centimeters per second (cm/s) (or 24 liters per minute [LPM]).

RESULTS

Smoldering Characteristics

As indicated in Figure 3a, combustion was successfully initiated in the column test after a preheating period of approximately 74 minutes (mins) (**Figure 3a**). Evidence of successful smoldering was confirmed at the base of the contaminant pack, as well as the generation of combustion gases (i.e., carbon dioxide [CO_2] and carbon monoxide [CO]) and the decrease in oxygen (O_2) in emissions, as shown in **Figure 3b**. The combustion test demonstrated strong self-sustaining smoldering behavior; that is, temperatures at each thermocouple location within the column demonstrated consistent, progressing peak temperatures following the termination of the heating source at t=85 mins (**Figure 3a**). As the smoldering front moves upwards through the reactor, thermocouples in the soil sequentially show a rapid increase in temperature following a temperature increase from the preceding thermocouple below. This is evidence of a thin combustion front propagating upwards from the column base through the Soil and GAC mixture.

The average peak temperature recorded for this smoldering test was 955 ± 53°C, with temperatures ranging from 851 to 1159°C. The smoldering front propagation speed was estimated to be 1.04 centimeters per minute (cm/min) (or 0.62 meters per hour [m/hr]). Smoldering front velocity is correlated to soil properties and the mass of fuel (in this case, GAC) present in the soil pore space and will vary during field implementation as a function of soil heterogeneity, soil moisture content, and other soil and process characteristics.

Figure 3a appears to show multiple thermocouple (TC) locations smoldering simultaneously during the combustion test (i.e. TCs 5, 6 and 7). This can be attributed to the presence of heterogeneities in the soil that form preferential airflow pathways through the soil matrix. This can be managed during field implementation by thorough mixing of soil and amendments. It may also be due to the presence of organics in the pre-treatment soil. As these organics are consumed in the smoldering front, void spaces are formed that create preferential airflow pathways.

A summary of all soil and emissions samples collected and submitted for analysis are presented in **Tables 1-5**.

Soil Analytical Results

Following the smoldering combustion test, the column was allowed to cool to ambient temperatures. The clean cap was removed, and the column excavated in short lifts. The soil in the column settled approximately 6 cm during the smoldering test, likely due to the loss of organics and soil moisture during treatment.

Analytical results for the ‘pre-treatment’ and ‘post-treatment’ soils are presented in **Tables 1-3**. Pre-treatment soils were collected from the as-received soil stockpile. A post-treatment soil sample was collected from the middle of the soil pack, approximately 14 cm above the heater, and submitted for analysis. Data presented includes concentrations of PFAS, as well as physical characteristics such as soil moisture, total organic carbon (TOC) and total solids (TS).

Pre- and post-treatment soils were analyzed for PFAS using EPA Method 1633 (**Table 1**). The principal PFAS in the pretreatment soil included perfluorooctanesulfonic acid (PFOS), perfluorodecanoic acid (PFDA), perfluorododecanoic acid (PFDoA), and 8:2 fluorotelomer sulfonic acid (8:2 FTS) at concentrations of 32, 24, 20 and 16 nanograms per gram (ng/g) (or micrograms per kilogram [µg/kg]). The mass of all PFAS present in the column prior to treatment, when converted to an equivalent organic fluorine mass, was approximately 0.25 mg. All PFAS species were reduced to below the method detection limit (MDL) following smoldering treatment, with detection limits ranging from 0.013 to 0.59 ng/g. Based on the analysis, the total mass of PFAS in the pre-treatment soil, converted to equivalent organic fluorine was reduced by >99%.

An additional set of pre- and post-treatment samples were submitted by COWI to ALS Denmark A/S, for analysis of PFAS via DIN 38414-14:2011. Results of this analysis are presented in **Table 2**. Similar treatment effectiveness was achieved for these samples, where all analytes in the post-treatment soil were found to be below the MDL.

Physical characteristics found 18% soil moisture in the pre-treatment soil, where post-treatment soil had <1.0% moisture content (**Table 3**). A high pre-treatment TOC concentration was also observed, whereas post-treatment soil TOC was found to be below the MDL. This TOC and moisture content supports the observations above, where high organics and moisture can form voids during smoldering, generating preferential flow pathways through the soil matrix.

Photographs of the as-received soil and post-treatment soil are presented in **Figures 2** and **4**, respectively, and provide visual evidence of treatment.

Principal Components of Smoldering Emissions

Concentrations of CO, CO_2 and O_2 as measured in real time via a continuous emissions monitoring system (CEMs) are presented in **Figure 3b**. HF and volatile PFAS were captured on emissions traps outlined in **Figure 1**.

Combustion gases generated from smoldering had peak CO_2 and CO concentrations of 23.3% and 4,875 parts per million (ppm), respectively. Minimum O_2 concentrations were also measured at 0.8%. High concentrations of combustion gases, coupled with high temperatures suggest a strong smoldering reaction that is characteristic of high-energy fuels such as GAC. Due to a leak in the CEMs sample line, the first few minutes of the smoldering test were not captured, however, temperature data from this time suggests effective smoldering. The leak was identified and repaired at approximately t=95 mins, whereupon CO and CO_2 data spiked.

HF emissions were captured using a series of four impingers arranged in series. As soon as injection air was introduced to the column to ignite smoldering, the HF emissions train was turned on to begin sampling emissions. Emissions were collected for approximately 48 minutes. The HF emissions train was switched off when condensate broke through the 4th knockout impinger and began to flow into the desiccant impinger (t=124 mins, **Figure 3a**). After the test was complete, the impinger solution was collected and analyzed using a fluoride ISE probe (**Table 4**). The total mass of fluoride (F^-) detected in the impinger solution, corrected for flow splitting and blank subtraction, was approximately 0.559 mg F^-. Compared to the mass of organic fluorine in the pre-treatment soil, this represents a 227% recovery of fluorine as HF in emissions. This high relative recovery may be due to several factors including 1) sources of organic F, such as PFAS precursors present in the soil that are not detected by a conventional PFAS analysis and/or 2) naturally occurring inorganic fluoride often present in natural soils. Fluoride is often found naturally in soils at concentrations ranging from 200-1000 mg/kg and may be a factor in the larger than expected HF recovery.[5,6] Duchesne et al. (2020) saw similar results, where tests with natural site soils recovered between 577-2438% F as HF in emissions. Assuming a constant release of F during smoldering, the estimate concentration of HF in the column emissions was approximately 0.98 ppm.

Volatile PFAS emissions were captured using two GAC tubes arranged in series (**Figure 1**). Emissions sampling began when injection air was turned on to ignite the column and continued for 32 mins (t = 108 mins). The emissions train was turned off when condensate was observed to be breaking through the second GAC trap into the desiccant trap. After the test was complete, samples were collected from both tubes and analyzed for PFAS (**Table 5**). The total mass of PFAS detected in the emissions, converted to equivalent organic fluorine and corrected for flow splitting was approximately 0.38 mg F. Compared to the mass of organic fluorine in the pre-treatment soil this represents a 153% recovery of volatile PFAS in emissions. This high relative recovery may be due to the presence of fluorinated precursors outside of a typical analytical scope. It should be noted that previous projects demonstrated effective removal of PFAS from soils following smoldering treatment according to total organic precursor (TOP) assay and total organic fluorine (TOF) analysis.[7] Low concentrations of PFAS in the pre-treatment soil may also contribute to this phenomenon. The presence of unquantified organic or inorganic fluorine in the pre-treatment soil challenges the mass balance of the smoldering system. For this reason, a mass balance of fluorine within the smoldering system was not an objective of this study.

RECOMMENDATIONS & CONSIDERATIONS FOR FULL-SCALE IMPLEMENTATION

Self-sustaining smoldering combustion was observed for the Site soil when mixed with low concentrations of GAC and CaO. The remediation efficiency and the calculated smoldering propagation velocity suggest that STARx can be successfully applied to treat PFAS-impacted soils from the Site.

The treatability study used commercially available fresh GAC as the fuel source for self-sustaining smoldering combustion. Smoldering column tests and past pilot studies have demonstrated that spent GAC from water treatment is a viable surrogate fuel for PFAS treatment. Future work could consider the use of spent GAC from water treatment or other systems at the Site or nearby locations as an opportunity to co-treat contaminants by STARx.

Several important parameters will need to be considered in the scale-up of smoldering as a PFAS-treatment technology. Typical of similar STARx applications, the quantity of condensate generated during treatment will need to be managed at larger scales. This will be an important parameter for this site if the soils collected for this study are representative of Site soil moisture conditions. It should be noted that condensate generated during full-scale smoldering operations is influenced by several factors, including overall soil moisture, recent precipitation and ambient temperatures. These factors will range widely based on site location and local climate. Previous full-scale operations have employed large moisture knockouts and mist filters to remove condensate from process emissions. This condensate will need to be characterized and managed as a separate waste stream.

The use of CaO as a soil amendment in smoldering has been shown to greatly reduce the quantity of HF and PIDs released in emissions during smoldering. The addition of CaO or any other alkaline minerals as amendments will alter the pH of the soil and have implications for the reuse of treated soil stockpiles. Soil pH of treated soil stockpiles can be adjusted with amendments or allowed to attenuate via CO_2 exchange with air.

The high temperatures required for treatment of PFAS-impacted soils will create significant stress on the structural elements of full-scale treatment plants. Careful materials selection during the design phase will mitigate these potential issues. During operations, careful inspection of structural elements exposed to high temperatures and thermal cycling must be done to ensure safe and continuous operations.

The mass recovery of HF and PFAS in the smoldering process emissions suggests that there are sources of inorganic fluoride or PFAS precursors present in the Site soils. Sources of inorganic fluoride such as cryolite (Na_3AlF_6) or fluorapatite [$Ca_5(PO_4)_3F$] may be naturally present in the pre-treatment material and could increase the overall F^- recovery in the HF emissions train.[5] Organic fluorine, such as PFAS and PFAS precursors outside of the scope of conventional PFAS analytical methods could also contribute to this high recovery of PFAS. Future work could attempt to characterize this fluorine content and improve reaction mass balance. TOF or TOP assay could be used to measure and convert precursors into measurable PFAS. Total fluorine methods, such as Particle Induced Gamma Emissions (PIGE) spectroscopy or X-ray Fluorescence (XRF) spectrometry, could be used on site soils to confirm the presence of inorganic fluoride in the pre-treatment soils. Previous projects (publication in progress) where site soils were found to contain PFAS precursors demonstrated >99% removal of all organic fluorine according to TOP assay and TOF following smoldering.[7]

Similarly, future work could investigate the potential presence of residual PFAS and PFAS precursors in post-treatment soil below existing soil detection limits. A synthetic precipitate leaching procedure (SPLP) could be used to determine the presence and mobility of any residual PFAS following treatment.

The presence of precursors and uncharacterized fluorine in Site soil challenge the mass balance of STARx in this application. It is likely that other breakdown products and PIDs were generated during smoldering and not captured via HF and PFAS analysis. Recent laboratory studies (publication in progress) have demonstrated that the addition of CaO as a soil amendment significantly reduces the quantity of these emissions, but trace amounts are still present. The mass of HF and total fluorine emitted during smoldering was reduced by approximately 90% where CaO amendments were used, compared to tests of the same material where CaO was not used. Future work could seek to characterize PIDs generated during smoldering that are not captured using these analytical methods.

We recommend that a field pilot be conducted to collect additional data for full scale design, costing, and operation of a STARx system. This would include an assessment of processing/treatment rates and optimization of operational parameters (e.g., determination of injection air flow rates appropriate for full-scale treatment to maximize throughput, or the addition of soil amendments such as CaO to limit emissions byproducts). New analytical methods to investigate PFAS precursors and inorganic fluoride in the pre- and post-treatment soil could also be implemented in this field pilot.

Sincerely,
SAVRON SOLUTIONS

Warren Ferguson, M.A.Sc., P.Eng. (ON)
Principal

Brian Harrison, M. E. Sc.
Staff Professional

REFERENCES

[1] Alexandra L. Duchesne, Joshua K. Brown, David J. Patch, David Major, Kela P. Weber, and Jason I. Gerhard. Remediation of PFAS-Contaminated Soil and Granular Activated Carbon by Smoldering Combustion. *Environmental Science & Technology.* 2020 54 (19), 12631-12640. DOI: 10.1021/acs.est.0c03058

[2] Fei Wang, Xingwen Lu, Xiao-yan Li and Kaimin Shih. Effectiveness and Mechanisms of Defluorination of Perfluorinated Alkyl Substances by Calcium Compounds during Waste Thermal Treatment. *Environmental Science & Technology.* 2015 49 (9), 5672-5680. doi: 10.1021/es506234b

[3] Fei Wang, Kaimin Shih, Xingwen Lu, and Chengshuai Liu. Mineralization behavior of fluorine in perfluorooctanesulfonate (PFOS) during thermal treatment of lime-conditioned sludge. *Environmental Science & Technology*. 2013 47(6), 2621-2627. doi: 10.1021/es305352p

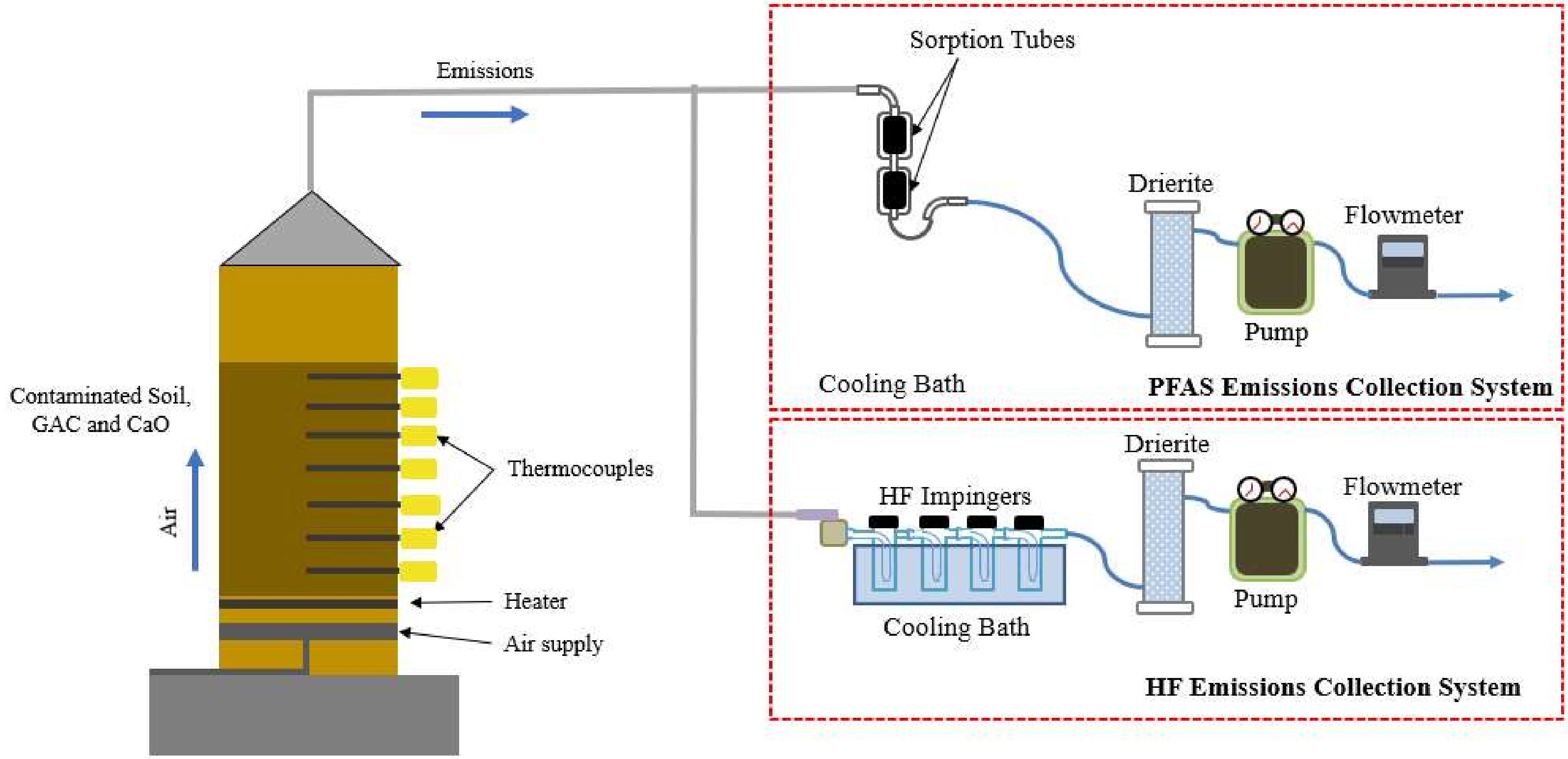
[4] Theran P. Riedel, M. Ariel Geer Wallace, Erin P. Shields, Jeffrey V. Ryan, Chun Wai Lee and William P. Linak. Low Temperature Thermal Treatment of Gas-Phase Fluorotelomer Alcohols by Calcium Oxide. *Chemosphere* 2021 272. doi: 10.1016/j.chemosphere.2021.129859

[5] Subbaiah Muthu Prabhu, Mohammed Yusuf, Yongtae Ahn, Ho Bum Park, Jaeyoung Choi, Mohammed A. Amin, Krishna Kumar Yadav & Byong-Hun Jeon. Fluoride occurrence in environment, regulations, and remediation methods for soil: A comprehensive review. *Chemosphere* 2023, 324. Doi: 10.1016/j.chemosphere.2023.13833

[6] International Programme on Chemical Safety, Environmental Health Criteria 227: Fluorides. *World Health Organization,* 2002.

[7] Savron (Publication Pending). ER18-1593: Demonstration of Smoldering Combustion Treatment of PFAS-Impacted Investigation-Derived Waste, Phase II. To be retrieved from https://serdp-estcp.mil/projects

FIGURES



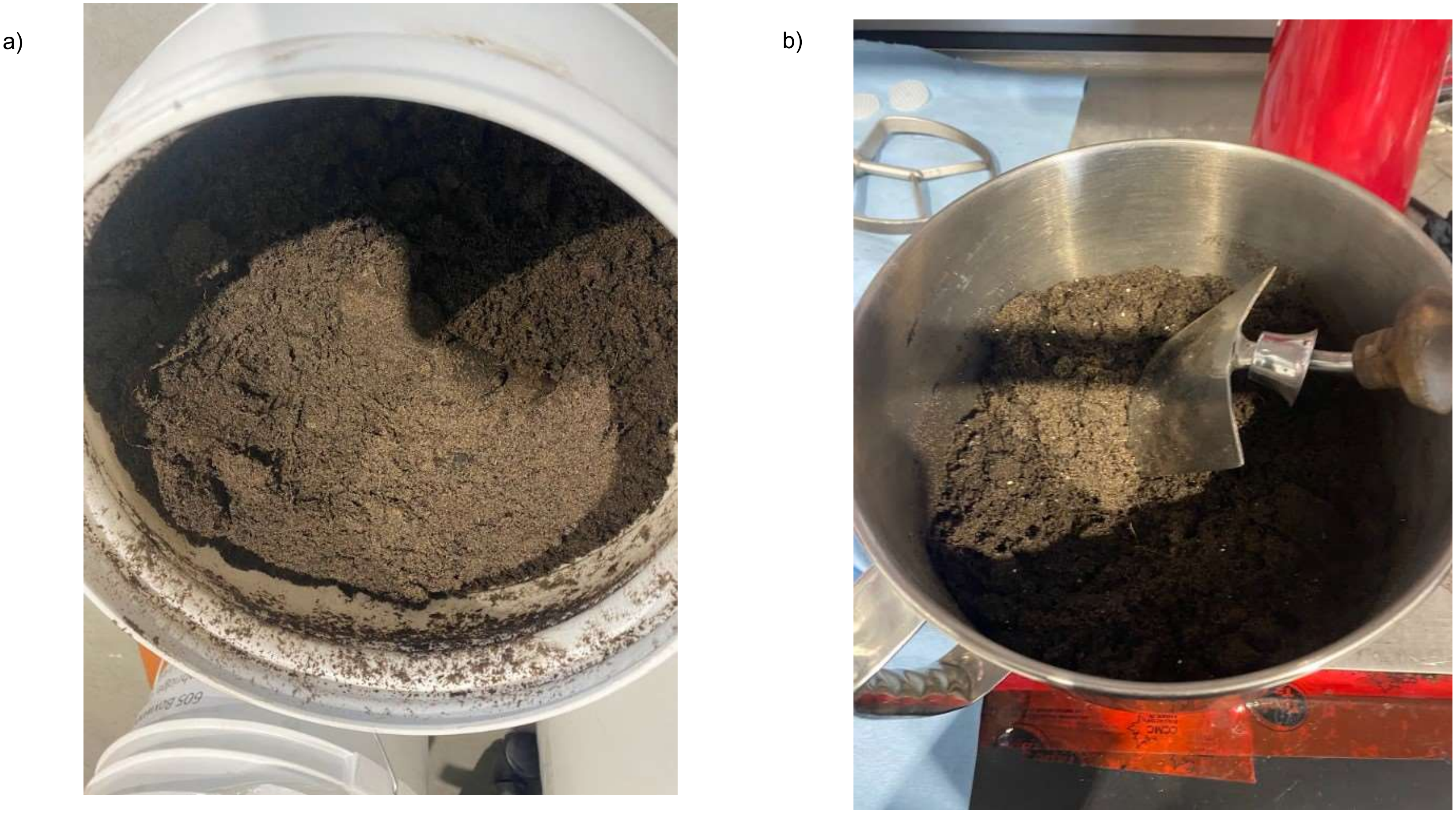
Notes:

Experimental Setup - Schematic
Danish EPA
MEM2530

savron

May 2024

Figure 1



Notes:

a) Photo of as-received soil samples, taken May 1, 2024

b) Photo of soil following mixing with GAC and CaO, taken May 1, 2024

Column Loading - Pre-treatment Soils

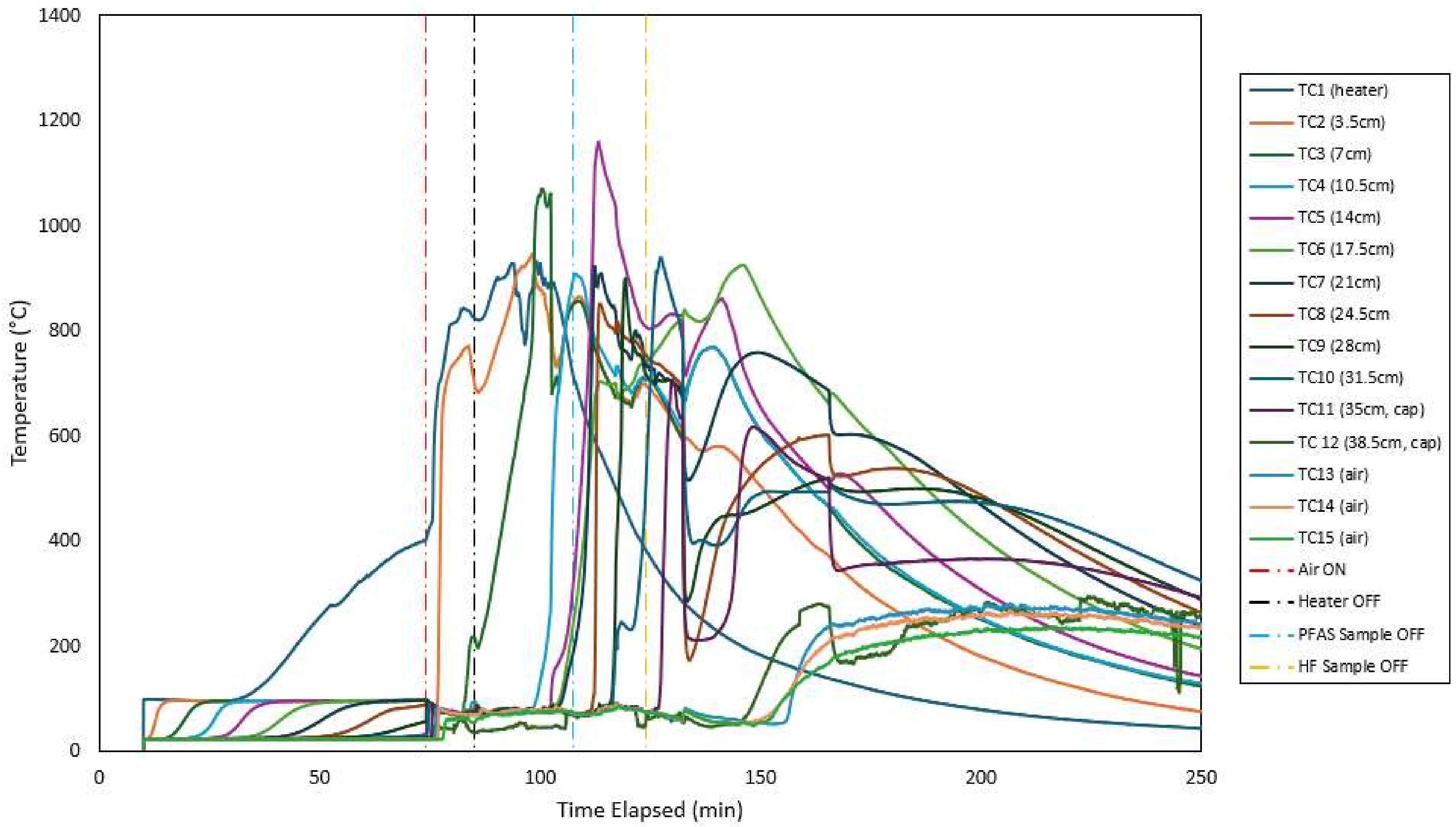
Danish EPA

MEM2530

savron

May 2024

Figure 2



Notes:

1) TC - Thermocouple. Distance measurement refers to the height of the thermocouple above the conductive heater.

2) CEMS - Continuous Emissions Monitoring System

STARx Thermocouple Temperature & CEMS Histories

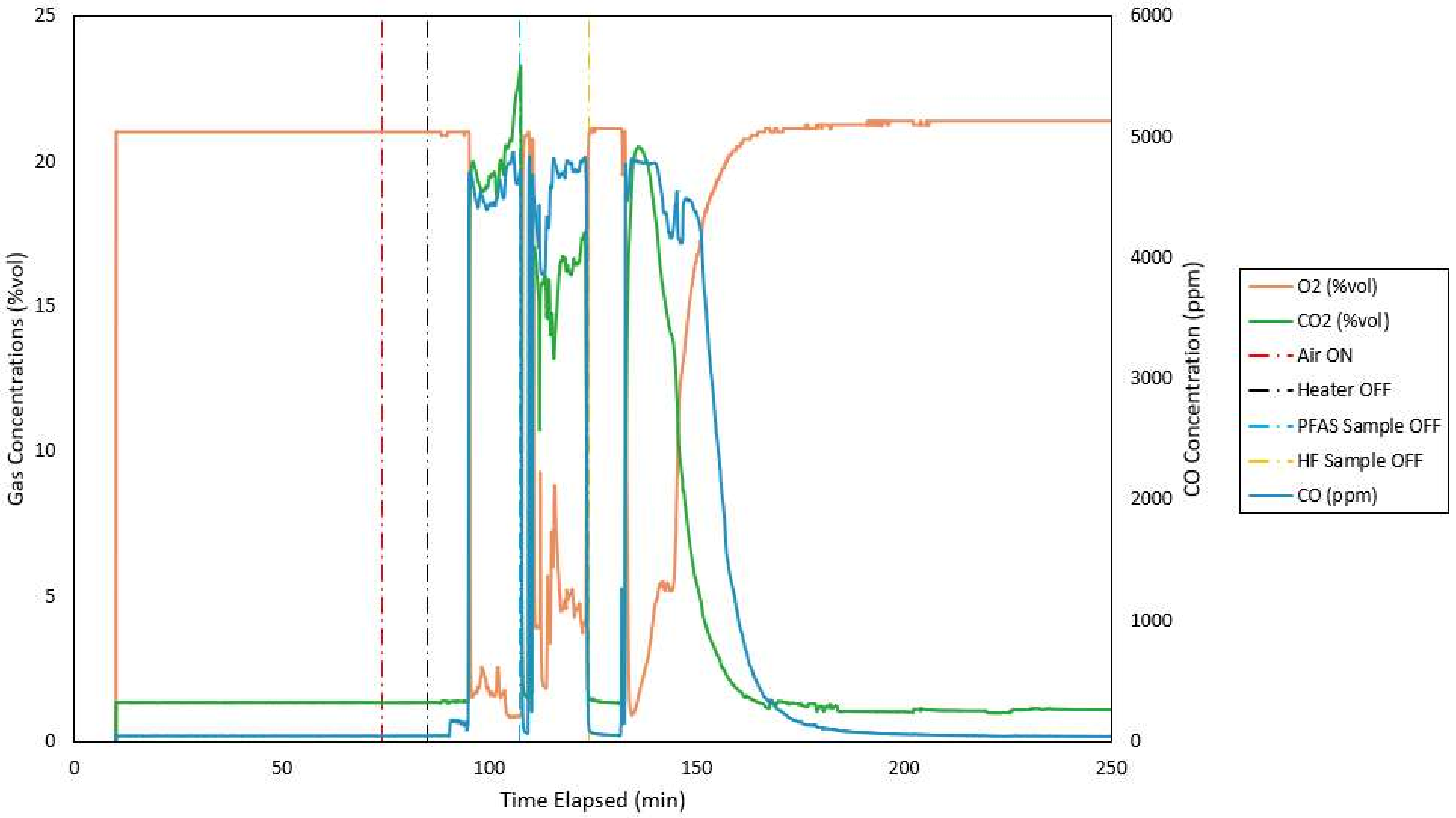
Danish EPA

MEM2530

savron

May 2024

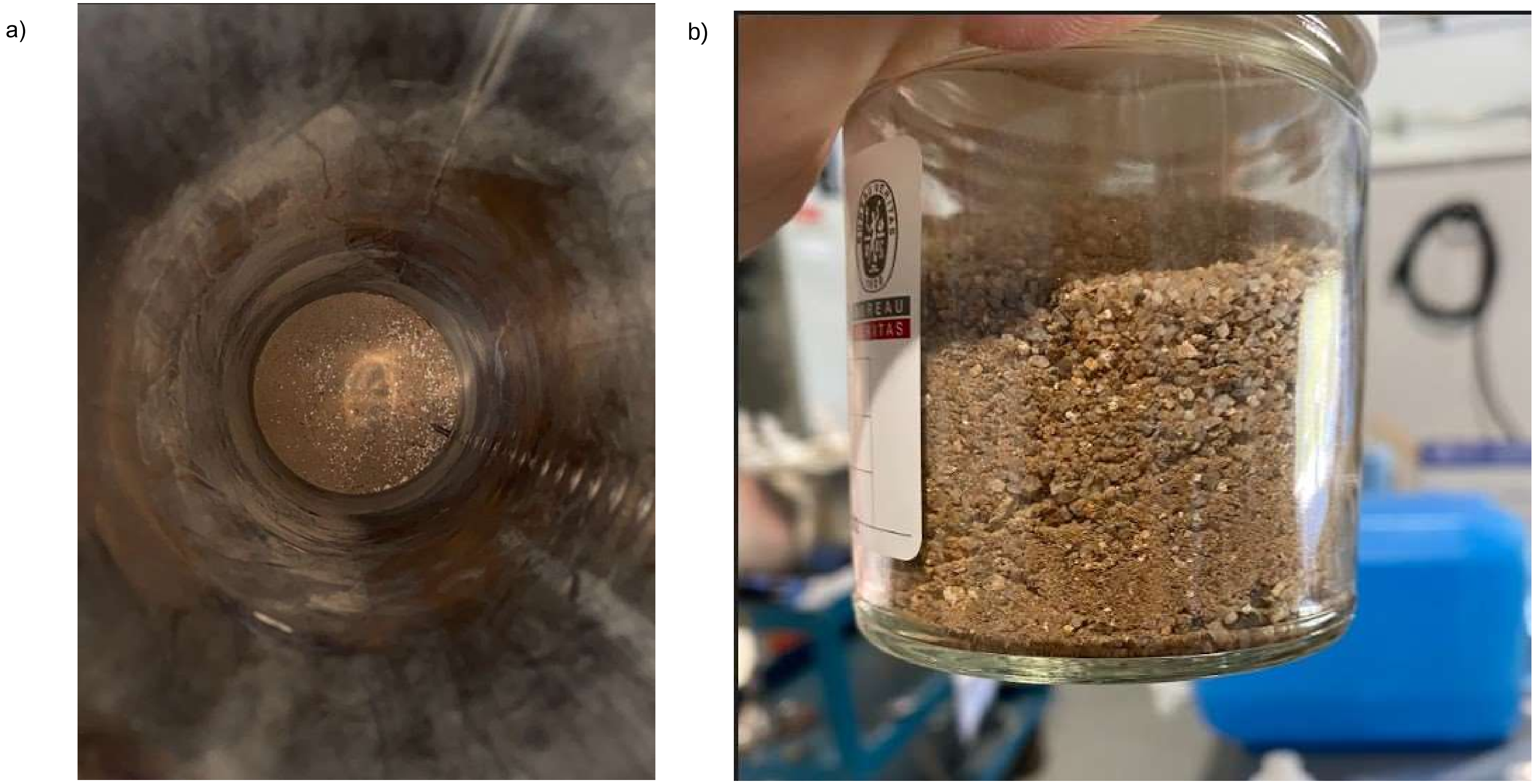
Figure 3a



Notes:

1) CEMS - Continuous Emissions Monitoring System
2) CO - Carbon Monoxide
3) CO2 - Carbon Dioxide
4) O2 - Oxygen

STARx Thermocouple Temperature & CEMS Histories **Danish EPA** MEM2530	
savron	**May 2024**
	Figure 3b



Notes:

a) Photo taken during column excavation at TC3-4 height (approximately 7-10.5cm above heater coil).

b) Post-treatment soil

Soil Excavation - Post-treatment Soils

Danish EPA

MEM2530

May 2024

Figure 4

TABLES

Table 1
Concentrations of PFAS Compounds in Pre-and Post-treatment Soil, Burea Veritas
Danish EPA
MEM2530

Analyte	Units	Pre-treatment Soil		Post-treatment Soil	
		RDL	COWI-PF-PRE	RDL	COWI-PF-POST-TC15
PFAS in Soil (EPA Draft Method 1633)					
Perfluorobutanoic acid (PFBA)	ng/g	0.80	0.78	0.80	<0.066
Perfluoropentanoic acid (PFPeA)	ng/g	0.40	1.1	0.40	<0.031
Perfluorohexanoic acid (PFHxA)	ng/g	0.20	1.8	0.20	<0.022
Perfluoroheptanoic acid (PFHpA)	ng/g	0.20	1.3	0.20	<0.027
Perfluorooctanoic acid (PFOA)	ng/g	0.20	5.0	0.20	<0.036
Perfluorononanoic acid (PFNA)	ng/g	0.20	2.2	0.20	<0.049
Perfluorodecanoic acid (PFDA)	ng/g	0.20	24	0.20	<0.030
Perfluoroundecanoic acid (PFUnA)	ng/g	0.20	6.3	0.20	<0.031
Perfluorododecanoic acid (PFDoA)	ng/g	0.20	20	0.20	<0.030
Perfluorotridecanoic acid (PFTRDA)	ng/g	0.20	0.73	0.20	<0.040
Perfluorotetradecanoic acid(PFTEDA)	ng/g	0.20	1.0	0.20	<0.025
Perfluorobutanesulfonic acid (PFBS)	ng/g	0.18	0.098	0.18	<0.018
Perfluoropentanesulfonic acid PFPes	ng/g	0.19	0.14	0.19	<0.026
Perfluorohexanesulfonic acid(PFHxS)	ng/g	0.18	1.2	0.18	<0.022
Perfluoroheptanesulfonic acid PFHpS	ng/g	0.19	0.12	0.19	<0.045
Perfluorooctanesulfonic acid (PFOS)	ng/g	1.9	32 (1)	0.19	<0.028
Perfluorononanesulfonic acid (PFNS)	ng/g	0.19	0.67	0.19	<0.040
Perfluorodecanesulfonic acid (PFDS)	ng/g	0.19	0.60	0.19	<0.020
Perfluorododecanesulfonic acid	ng/g	0.19	0.16	0.19	<0.063
4:2 Fluorotelomer sulfonic acid	ng/g	0.75	<0.094	0.75	<0.094
6:2 Fluorotelomer sulfonic acid	ng/g	0.76	1.0	0.76	<0.12
8:2 Fluorotelomer sulfonic acid	ng/g	0.77	16	0.77	<0.15
Perfluorooctane Sulfonamide (PFOSA)	ng/g	0.20	2.5	0.20	<0.018
MeFOSA	ng/g	0.20	<0.030	0.20	<0.030
EtFOSA	ng/g	0.20	<0.013	0.20	<0.013
MeFOSAA	ng/g	0.20	<0.046	0.20	<0.046
EtFOSAA	ng/g	0.20	2.6	0.20	<0.059
MeFOSE	ng/g	2.0	<0.26	2.0	<0.26
EtFOSE	ng/g	2.0	2.0	2.0	<0.59
Hexafluoropropyleneoxide dimer acid	ng/g	0.80	<0.077	0.80	<0.077
4,8-Dioxa-3H-perfluorononanoic acid	ng/g	0.76	<0.077	0.76	<0.077
Perfluoro-3-methoxypropanoic acid	ng/g	0.40	<0.024	0.40	<0.024
Perfluoro-4-methoxybutanoic acid	ng/g	0.40	<0.050	0.40	<0.050
Nonafluoro-3,6-dioxaheptanoic acid	ng/g	4.0	<0.54	4.0	<0.54
9Cl-PF3ONS (F-53B Major)	ng/g	0.75	<0.082	0.75	<0.082
11Cl-PF3OUdS (F-53B Minor)	ng/g	0.76	<0.096	0.76	<0.096
Perfluoro2ethoxyethanesulfonic acid	ng/g	0.36	<0.063	0.36	<0.063
3-Perfluoropropylpropanoic acid	ng/g	1.0	<0.26	1.0	<0.26
2H2H3H3H-Perfluorooctanoic acid	ng/g	5.0	<0.89	5.0	<0.89
3-Perfluoroheptylpropanoic acid	ng/g	5.0	<0.85	5.0	<0.85

Table 1
Concentrations of PFAS Compounds in Pre-and Post-treatment Soil, Burea Veritas
Danish EPA
MEM2530

Analyte	Units	Pre-treatment Soil		Post-treatment Soil	
		RDL	COWI-PF-PRE	RDL	COWI-PF-POST-TC15
Extraction Surrogate Recovery (%)					
13C2-Perfluorodecanoic acid	%	-	92	-	78
13C2-Perfluorohexanoic acid	%	-	77	-	82
13C2-Perfluorotetradecanoic acid	%	-	22	-	115
13C3-Perfluorobutanesulfonic acid	%	-	58	-	111
13C3-Perfluorobutanoic Acid	%	-	69	-	80
13C3-Perfluorohexanesulfonic Acid	%	-	63	-	110
13C4-Perfluorooctanesulfonic acid	%	-	87	-	83
13C4-Perfluorooctanoic acid	%	-	85	-	74
13C5-Perfluorohexanoic Acid	%	-	59	-	112
13C5-Perfluorononanoic acid	%	-	90	-	79
13C6-Perfluorodecanoic Acid	%	-	52	-	112
13C7-Perfluoroundecanoic Acid	%	-	54	-	119
13C8-Perfluorooctanesulfonic Acid	%	-	48	-	113
13C8-Perfluorooctanoic Acid	%	-	59	-	119
13C9-Perfluorononanoic Acid	%	-	58	-	119
18O2-Perfluorohexanesulfonic acid	%	-	75	-	81
D3-MeFOSAA	%	-	47	-	107
D5-EtFOSAA	%	-	41	-	107
D7-MeFOSE	%	-	14 (2)	-	86
D9-EtFOSE	%	-	10 (2)	-	85
Instrument Surrogate Recovery (%)					
13C2-4:2-Fluorotelomersulfonic Acid	%	-	139	-	128
13C2-6:2-Fluorotelomersulfonic Acid	%	-	138	-	123
13C2-8:2-Fluorotelomersulfonic Acid	%	-	131	-	111
13C2-Perfluorododecanoic acid	%	-	44	-	112
13C3-HFPO-DA	%	-	56	-	115
13C4-Perfluorobutanoic acid	%	-	58	-	112
13C4-Perfluoroheptanoic acid	%	-	60	-	107
13C5-Perfluoropentanoic acid	%	-	57	-	110
13C8-Perfluorooctane Sulfonamide	%	-	34	-	99
D3-MeFOSA	%	-	10 (2)	-	79
D5-EtFOSA	%	-	6.8 (2)	-	74

Notes:
ng/g - nanograms per gram
RDL - reportable detection limit
PFAS - per- and polyfluoroalkyl substances
1. Due to high concentration of the target analyte, sample required dilution. Detection limit was adjusted accordingly (10x).
2. Extracted internal standard analyte recovery was below the defined lower control limit. Laboratory control sample (spike) resulted in satisfactory recovery of the associated native analyte result. When considered together, QC data suggests matrix interferences may be increasing the variability of the associated native analyte result.

Table 2
Concentrations of PFAS Compounds in Pre-and Post-treatment Soil, ALS Denmark A/S
Danish EPA
MEM2530

Analyte	Units	Pre-treatment	Post-treatment
PFAS in Soil (DIN 38414-14:2011)			
Perfluoroheptanoic acid (PFHpA)	µg/kg	0.73	<0.20
Perfluorooctane Sulfonamide (PFOSA)	µg/kg	1.98	<0.050
$PFAS_{22}$ in Soil Low DL	-		:
Perfluorohexanoic acid (PFHxA)	µg/kg	0.92	<0.20
Perfluorooctanoic acid (PFOA)	µg/kg	3.89	<0.050
Perfluorononanoic acid (PFNA)	µg/kg	1.6	<0.050
Perfluorobutanesulfonic acid (PFBS)	µg/kg	<0,10	<0.10
Perfluorohexanesulfonic acid (PFHxS)	µg/kg	0.76	<0.10
Perfluorooctanesulfonic acid (PFOS)	µg/kg	17.8	<0.050
Perfluorodecanesulfonic acid (PFDS)	µg/kg	0.48	<0.050
Perfluorobutanoic acid (PFBA)	µg/kg	0.617	<0.050
Perfluoropentanoic acid (PFPeA)	µg/kg	0.63	<0.20
Perfluoroundecanoic acid (PFUnA)	µg/kg	4.58	<0.050
Perfluorododecanoic acid (PFDoA)	µg/kg	14.5	<0.050
Perfluorodecanoic acid (PFDA)	µg/kg	15.4	<0.050
6:2 Fluorotelomer sulfonic acid (6:2 FTS)	µg/kg	0.296	<0.050
Perfluorododecanesulfonic aicd (PFDoDS)	µg/kg	0.284	<0.050
Perfluoroheptanesulfonic acid (PFHpS)	µg/kg	<0,10	<0.10
Perfluorononanesulfonic acid (PFNS)	µg/kg	0.493	<0.050
Perfluoropentanesulfonic acid (PFPeS)	µg/kg	<0,050	<0.050
Perfluorotridecanoic acid (PFTRDA)	µg/kg	0.26	<0.050
Perfluorotridecanesulfonic acid (PFTrDS)	µg/kg	<0,20	<0.20
Perfluorooundecanesulfonic acid (PFUnDS)	µg/kg	0.3	<0.20
Sum of $PFAS_{22}$	µg/kg	65.5	<1.00
Sum of PFOA, PFOS, PFNA, PFHxS	µg/kg	24.0	<0.125

Notes:

ug/kg - micrograms per kilogram

PFAS - per- and polyfluoroalkyl substances

Table 3
Physical Properties of Pre- and Post-treatment Soils
Danish EPA
MEM2530

Compound	Units	Pre-treatment Soil			Post-treatment Soil		
		RDL	COWI-PF-PRE	COWI-TOC-PRE	MDL	COWI-PF-POST-TC15	COWI-TOC-POST-TC15
Physical Properties, Total Organic Carbon (TOC)							
Moisture Content	%	1	18	-	1	<1	-
Total Organic Carbon	mg/kg	500	-	16,000	500	-	<500
Solids	%	1	82	-	1	100	-

Notes:
mg/kg - milligrams per kilogram
RDL - reportable detection limit

Table 4
Hydrogen Fluoride (HF) Mass Recoveries
Danish EPA
MEM2530

Sample Name	Sample Volume (L)	Measured F^- Concentration (mg/L)	Dilution Factor	Corrected F^- Concentration (mg/L)	Raw F^- Mass (mg)[1]	F^- Mass (mg)[2]	HF[3] mass (mg)
HF Impinger 1/2	0.171	0.369	2	0.738	0.108	0.169	0.178
HF Impinger 3/4	0.187	0.720	2	1.440	0.249	0.390	0.411
Blank	-	0.053	2	0.106	-	-	
				Total F- Mass Recovered (mg)	0.358	0.559	0.589

Notes
1 - Mass of fluoride recovered, corrected for blank sample measurement
2 - total mass of fluoride recovered, corrected for flow splitting of PFAS and HF emissions trains
3 - total mass of HF recovered
HF - Hydogen fluoride
L - liters
mg/L - milligrams per liter
mg - milligrams
F^- - Fluoride
ISE - ion selective electrode

Table 5
Concentrations of PFAS in Emissions, RMC
Danish EPA
MEM2530

Analyte	**Unit**	**Detection Limits**		**Emissions GAC**	
		MDL	**LOQ**	**GAC 1 ('Lead')**	**GAC 2 ('Lag')**
PFBA	µg/kg	1.62	2.66	125.3	85.3
PFPeA	µg/kg	0.22	0.65	38.0	7.0
PFHxA	µg/kg	0.11	0.29	475.9	8.9
PFHpA	µg/kg	0.32	0.42	42.0	2.2
PFOA	µg/kg	0.18	0.41	881.1	9.8
PFNA	µg/kg	0.17	0.36	22.47	0.48
PFDA	µg/kg	0.10	0.34	384.61	7.25
PFUdA	µg/kg	0.17	0.28	6.94	0.28 J
PFDoDA	µg/kg	0.14	1.85	100.31	1.65 J
PFTrDa	µg/kg	1.48	2.40	<DL	<DL
PFTeDA	µg/kg	0.69	1.06	3.62	<DL
PFBS	µg/kg	0.29	0.53	<DL	0.86
PFPeS	µg/kg	0.18	0.40	<DL	<DL
PFHxS	µg/kg	0.18	0.40	1.50	<DL
PFHpS	µg/kg	0.17	0.36	<DL	<DL
PFOS	µg/kg	0.39	2.42	1.47	<DL
PFNS	µg/kg	0.18	0.35	<DL	<DL
PFDS	µg/kg	0.15	0.29	<DL	<DL
PFDoS	µg/kg	1.46	2.38	<DL	<DL
PFOSA	µg/kg	0.16	0.31	3.09	<DL
Me-FOSAA	µg/kg	0.03	0.24	<DL	<DL
Et-FOSAA	µg/kg	1.96	3.98	<DL	<DL
4:2 FTS	µg/kg	0.89	1.98	<DL	<DL
6:2 FTS	µg/kg	0.76	1.82	12.74	<DL
8:2 FTS	µg/kg	0.68	1.41	16.70	<DL
6:2 FTSAB	µg/kg	0.18	0.38	<DL	<DL
5:3 FTB	µg/kg	0.23	0.50	<DL	<DL
5:1:2 FTB	µg/kg	0.25	0.55	<DL	<DL

Notes
Concentrations of PFAS detected in emissions treatment train.
µg/kg - micrograms per kilogram
GAC - granular activated carbon
PFAS - per- and polyfluoroalkyl substances
MDL - method detection limit
LOQ - limit of quantitation

APPENDIX A: PRELIMINARY SITE CHARACTERIZATION RESULTS

Ordrenr: 841480
Sagsnavn: A272516
Udtaget: 23-02-2024

ALS Denmark A/S
Bakkegårdsvej 406 A
DK-3050 Humlebæk
Telefon: +45 4925 0770
www.alsglobal.dk

ANALYSERAPPORT

COWI
Parallelvej 2
2800 Lyngby
Att.: COWI

Udskrevet:	21-03-2024
Version:	1
Modtaget:	26-02-2024
Analyseperiode:	26-02-2024 - 21-03-2024
Ordrenr.:	841480

Sagsnavn:	A272516
Lokalitet:	Oksbøl
Udtaget:	23-02-2024
Prøvetype:	Jord
Prøvetager:	Rekv./MNFO
Kunde:	COWI, Parallelvej 2, 2800 Lyngby, Att. Kirsten Rügge

Prøvenr.:		42918/24	42919/24		
Prøve ID:		Indenfor område	Udenfor område		
Dybde:		- m u.t	- m u.t		
Kommentar		*1	*1		
Parameter				**Enhed**	**Metode**
Tørstof	*3	64.5	88.2	%	CSN ISO 11465
PFHpA, Perfluorheptansyre	*3	0.81	0.47	µg/kg TS	DIN 38414-14:2011
PFOSA, Perfluoroctansulfonamid	*3	53.4	0.124	µg/kg TS	DIN 38414-14:2011
PFAS 22 i jord lav DL				-	DIN 38414-14:2011
PFHxA, Perfluorhexansyre	*3	<6.60	0.92	µg/kg TS	DIN 38414-14:2011
PFOA, Perfluoroctansyre	*3	6.03	4.52	µg/kg TS	DIN 38414-14:2011
PFNA, Perfluornonansyre	*3	3.92	2.08	µg/kg TS	DIN 38414-14:2011
PFBS, Perfluorbutansulfonsyre	*3	0.72	<0.10	µg/kg TS	DIN 38414-14:2011
PFHxS, Perfluorhexansulfonsyre	*3	5.42	0.12	µg/kg TS	DIN 38414-14:2011
PFOS, Perfluoroctansulfonsyre	*3	165	15.5	µg/kg TS	DIN 38414-14:2011
PFDS, Perfluordecansulfonsyre	*3	6.80	<0.050	µg/kg TS	DIN 38414-14:2011
PFBA, Perfluorbutansyre	*3	<0.650	0.307	µg/kg TS	DIN 38414-14:2011
PFPeA, Perfluorpentansyre	*3	<4.40	0.42	µg/kg TS	DIN 38414-14:2011
PFUnDA, Perfluorundecansyre	*3	11.6	4.69	µg/kg TS	DIN 38414-14:2011
PFDoDA, Perfluordodecansyre	*3	20.6	9.00	µg/kg TS	DIN 38414-14:2011
PFDA, Perfluordecansyre	*3	30.2	25.4	µg/kg TS	DIN 38414-14:2011
6:2 FTS, 1H,1H,2H,2H-Perfluoroctansulfonsyre	*3	1.47	<0.050	µg/kg TS	DIN 38414-14:2011
PFDoDS, Perfluordodecansulfonsyre	*3	4.84	<0.050	µg/kg TS	DIN 38414-14:2011
PFHpS, Perfluorheptansulfonsyre	*3	0.79	<0.10	µg/kg TS	DIN 38414-14:2011
PFNS, Perfluornonansulfonsyre	*3	15.1	0.073	µg/kg TS	DIN 38414-14:2011
PFPeS, Perfluorpentansulfonsyre	*3	0.559	<0.050	µg/kg TS	DIN 38414-14:2011
PFTrDA, Perfluortridecansyre	*3	0.576	0.174	µg/kg TS	DIN 38414-14:2011
PFTrDS, Perfluortridecansulfonsyre	*3	4.07	<0.20	µg/kg TS	DIN 38414-14:2011
PFUnDS, Perfluorundecansulfonsyre	*3	4.88	<0.20	µg/kg TS	DIN 38414-14:2011
Sum af PFAS, 22 stoffer	*2	337	63.8	µg/kg TS	DIN 38414-14:2011
Sum af PFOA, PFOS, PFNA, PFHxS	*2	180	22.2	µg/kg TS	DIN 38414-14:2011

Tegnforklaring, Resultat:
i.p.: Ikke påvist, -: analysen er ikke udført
i rapporten betyder ikke akkrediteret

ANALYSERAPPORT

Kommentar

*1	Ingen kommentar
*2	# Underleverandør: ALS Czech Republic s.r.o, CAI L1163
*3	Underleverandør: ALS Czech Republic s.r.o, CAI L1163

Katrin Potthoff

Katrin Potthoff