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# **Remediation of PFAS Contaminated Soil by OSE II**

# Australia 2024

CMTA International and the OSEI Corporation performed a trial to remediate PFAS contaminated soil using our technology Oil Spill Eater II (OSE II). The PFAS contaminated soil was provided by an Australian Federal Government Department for the purpose of CMTA and the OSEI Corporation to perform its own trial to demonstrate how effective OSEII is at remediating PFAS contaminated soil. The trial commenced in June 2024 and concluded in October 2024.

The OSEI Corporation developed a PFAS remediation protocol for the trial. The PFAS Contaminated soil was placed in a suitable biological container, not a chemistry test, where OSE II was mixed with natural water, and applied to the contaminated soil. Natural water was added to the system to ensure a 30% moisture level. Extraction sites were noted in the protocol and utilized, each time for consistent extraction points understanding the soil was mixed and turned three times a week to reach homogeneity.

An independent company, Hibbs & Associates Pty Ltd (Hibbs) was engaged to perform the extractions in triplicate for each of the 6 rounds of extraction intervals noted in the protocol. Hibbs transported the samples on ice to the laboratory Hibbs & Associates Pty Ltd (Hibbs) a NATA certified laboratory with experience in testing for PFAS in soil. The laboratory was directed to perform PFAS Speciation US EPA method 1633, utilized along with Total Oxidizable Precursor, Total Fluoride (Inorganic/Organic), and Total Soluble Inorganic Fluoride tests were conducted.

The mode of OSE II that allows it to remediate halogenated hydrocarbons as well as hydrocarbons, starts with the ability of the biosurfactants combined with the multitude of enzymes, as well as additional proprietary aspects of OSE II developed during the manufacturing process, to partition and penetrate the molecules of a contaminant, where in the case of halogenated hydrocarbons, causes the slight gassing off of the halogen, since it becomes in part a gas.

This also reduces the toxicity of the inorganic volume as well as the organic aspects of PFAS, hence allowing the colonization of indigenous bacteria to flourish and at some point, transition to the remaining PFAS/PFOS matrices, where it will become CO2 and water. This has been the mode of OSE II actions for numerous halogenated hydrocarbon clean ups, including PCB's (see the following links):

PCB in Farsee https://www.osei.us/wp-content/uploads/Iran-PCB-transformer-company-test.pdf

PCB Translated to English <u>https://www.osei.us/wp-content/uploads/Iran-Transformer-Research-Institute-translated-English.pdf</u>

Dichloral Benzene <u>https://www.osei.us/tech-library-pdfs/2011/16-</u> OSEI%20Manual ChlorHydroEfficacyTest.pdf

The fact that OSE II has detoxified the PFAS to levels that even single celled organisms can survive is shown by Hibbs Laboratory in their report, where they state "The aerobic microbial activity in R06 has shown a decline, with an average count of  $1.25 \times 10^7$  cfu/g, compared to  $2.3 \times 10^7$  cfu/g in R05."

Note: the reason you clean up toxic contaminants is to reduce the toxicity to the environment, so that singled celled microbes can survive, and if they can survive then there should be no adverse effects to human health, so you clean up toxic contaminants to protect human health.

The fact that there are any bacteria alive at all, shows that OSE II has detoxified the PFAS to the point singled celled micro-organisms can survive and exist. This proves the PFAS, once its molecules are partitioned, and gassed off reducing the volume of Fluorine due to OSE II, and at the same time reduced any other components of the PFAS, the bacteria can now colonize and start digesting the remaining matrices of PFAS. This also means in a short time after applying OSE II a dramatic decrease in the PFAS toxicity has been reduced quickly reducing the environmental impact of PFAS.

# There are a few items to discuss regarding the results.

CMTA International received results as they were produced during the PFAS remediation time frame as they became available.

Hibbs report number MFGO290 showed the soil test/PFAS results:

- MFGO290-01, RO4-1 showed a Fluoride level of 0.50 mg/kg.
- MFGO290-02, RO4-2 showed a fluoride level of 0.55 mg/kg.
- MFGO290-03, RO4-3 showed a Fluoride level of 0.51 mg/kg.

Hibbs stated OSE II had met the required reduction standard. The RO4 tests correlated with the 30 days after application of OSE II interval for extraction of soil samples in the protocol. The laboratory also relayed there were some calculation problems, which had been worked out as well. Keep in mind the standard to meet in PFAS level, is not the same as 100% reduction level. The OSEI Corporation and CMTA wanted to conduct a longer-term trial to show the long- term benefits regarding the use of OSE II.

The final report on PFAS, shows some anomalies on the laboratory report, and particularly the final interval, round 6, which was not required, since OSE II had long since shown its ability to remediate the PFAS. Round 6 showed the third increase in Fluorine level, where Hibbs Laboratory, considered the moisture level of 30% had decreased to 26%, may have been part of the cause. However, once you collect a sample of soil on a steel spatula, most of the water runs off, and you place the soil with some water in the glass jar. The Hibbs Laboratory also stated that "The aerobic microbial activity in R06 has shown a decline", with an average count of  $1.25 \times 10^7$  cfu/g, compared to  $2.3 \times 10^7$  cfu/g in R05.", the reduction in microbes is expected once the food source has declined, to some extent, since the surface area, to attach and digest the food source, has been reduced.

Given the above two dot points it is recommended to end this trial and disregard the original and last rounds due to system instabilities resulting in increased variability within the trending of results. This may be the result of incomplete mixing, depletion of the original soil mass, reduction in microbial activity and/or approaching the limits of detection for some PFAS analytes. Approaching the limits of detection causing anomalies is the most probable cause of the problem, from our 34 years of experience reading laboratory reports.

The fact OSE II met the standard according to the Hibbs Laboratory, is great, however if you look at the percent reduction in 45 days which is noted by the laboratory, and then extrapolate to 100% one can determine the time for virtually 100% reduction in all the PFAS analytes measured. The highlighted in yellow calculations will show the point in days for 100% reduction, for each grouping.

- "The PFOA concentrations in R06 decreased further, maintaining a 74% reduction compared to the baseline, with a slight reduction from 1.05 μg/kg in R05 to 0.96 μg/kg in R06. This consistent decline in PFOA levels continues to highlight the effectiveness of the treatment process in mitigating PFOA contamination." therefor, 74% reduction is 1.64% per day, there for if you multiply 1.64 % per day it would require approximately 61 days to reach 100% reduction.
- "PFBS levels remained constant from R05 to R06 with a result of 0.4 μg/kg, maintaining an 80 % reduction compared to the baseline." therefor, 80% reduction is 1.77% per day, there for if you multiply 1.77 % per day it would require approximately 57 days to reach 100% reduction.
- "PFDS levels were reduced marginally with a result of 41 μg/kg in RF06 from 42 μg/kg in R05, maintaining an 80 % reduction compared to the baseline "therefor, 80% reduction is 1.77% per day, there for if you multiply 1.77 % per day it would require approximately 57 days to reach 100% reduction.
- "PFHxS levels decreased from 8.8 µg/kg in RO5 to 6.2 in RO6 maintaining a 76% reduction compared to the baseline." therefor, 76% reduction is 1.68% per day, there for if you multiply 1.68% per day it would require approximately 60 days to reach 100% reduction.
- "PFHxA levels increased marginally from 0.6 μg/kg in RO5 to 0.8 in RO6 maintaining a 76% reduction compared to the baseline." therefor, 76% reduction is 1.68% per day, there for if you multiply 1.68% per day it would require approximately 60 days to reach 100% reduction.
- "PFOSA levels decreased from 5.1 μg/kg in RO5 to 4.3 μg/kg in RO6 maintaining a 57% reduction compared to the baseline." therefor, 57% reduction is 1.26% per day, there for if you multiply 1.26% per day it would require approximately 80 days to reach 100% reduction.
- "PFPeS levels marginally decreased from 0.59 μg/kg in RO5 to 0.49 μg/kg in RO6 maintaining a 79% reduction compared to the baseline." therefor, 79% reduction is 1.75% per day, there for if you multiply 1.75% per day it would require approximately 57. days to reach 100% reduction.
- "PFPeA levels increased marginally from 0.30 μg/kg in RO5 to 0.36 in RO6 maintaining a 48% reduction compared to the baseline." therefor, 48% reduction is 1.06% per day, there for if you multiply 1.06 % per day it would require approximately 95 days to reach 100% reduction.

OSE II according to Hibbs's report states, <u>"Overall, while significant progress has been made in the</u> <u>reduction of PFAS concentrations."</u> which means OSE II can remediate PFAS to not only the standard, but to a <u>100% reduction overtime</u>, and the fact there is a potential end point of 95 days, is absolutely great, when compared to the global understandings of PFAS that it can persist in the environment forever, hence the global community stating PFAS is the forever chemical.

The fact that OSE II can detoxify and render the chemical level acceptable in 30 to 45 days, virtually eliminating its effect upon human health, is a great step forward in causing the recovery of earth from this terrible persistent chemical.

OSE II has proven through this trial that it is capable of remediating PFAS.

Steven Pedigo CEO OSEI Corporation

# See test report below showing OSE II met the standard, followed by the complete report where the bacteria count or MPN tests are the last tests presented.



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## Certificate of Analysis MFG0290

Client Details								
Client	Hibbs & Associate	Hibbs & Associates Pty Ltd						
Contact	George Dervusosk	George Dervusoski						
Address	PO Box 4266, HOI	PO Box 4266, HOMEBUSH, NSW, 2140						
Sample Details								
Your Reference	S13167							
Number of Samples	2 Sludge, 3 Soil	Soil is equivalent to PFAS, which becomes obvious based on the testing of						
Date Samples Received	16/07/2024	fluoride, and analytes on pages 4-8, of MGO290 report here.						
Date Instructions Received	16/07/2024							
Analysis Details								
Please refer to the following pages for Samples were analysed as received frr Results are reported on a dry weight b	om the client. Results relate	specifically to the samples as received.						
Please refer to the following pages for Samples were analysed as received frr Results are reported on a dry weight t Report Details	om the client. Results relate	specifically to the samples as received.						
Samples were analysed as received from Results are reported on a dry weight b	om the client. Results relate	specifically to the samples as received.						
Please refer to the following pages for Samples were analysed as received for Results are reported on a dry weight the Report Details Date Results Requested by Date of I ssue NATA Accreditation Number 2901. This	om the client. Results relate basis for solids and on an a 23/07/2024 23/07/2024 s document shall not be re	e specifically to the samples as received. s received basis for other matrices.						
Please refer to the following pages for Samples were analysed as received for Results are reported on a dry weight the Report Details Date Results Requested by Date of I ssue NATA Accreditation Number 2901. This Accredited for compliance with I S	23/07/2024 23/07/2024 23/07/2024 s document shall not be re <b>SO/ I EC 17025. Tests not</b> Azrin Akram, Senic	e specifically to the samples as received. s received basis for other matrices.  produced except in full.  covered by NATA are denoted with * .  pr Chemist istant Lab Manager						

## Samples in this Report

•	•			
Envirolab I D	Sample I D	Matrix	Date Sampled	Date Received
MFG0290-01	R04-1	Soil	16/07/2024	16/07/2024
MFG0290-02	R04-2	Soil	16/07/2024	16/07/2024
MFG0290-03	R04-3	Soil	16/07/2024	16/07/2024
MFG0290-04	Sample 01	Sludge	16/07/2024	16/07/2024
MFG0290-05	Sample 02	Sludge	16/07/2024	16/07/2024

#### Semi-volatile TRH (Sludge)

Envirolab I D	Units	PQL	MFG0290-04	MFG0290-05
Your Reference			Sample 01	Sample 02
Date Sampled			16/07/2024	16/07/2024
TRH C10-C14	mg/kg	50	<50	<50
TRH C15-C28	mg/kg	100	1900	1400
TRH C29-C36	mg/kg	100	210	<100
Total +ve TRH C10-C36	mg/kg	50	2100	1400
TRH >C10-C16	mg/kg	50	190	160
TRH >C16-C34 (F3)	mg/kg	100	1900	1300
TRH >C34-C40 (F4)	mg/kg	100	<100	<100
Total +ve TRH >C10-C40	mg/kg	50	2100	1500
	%			

Your Reference: Revision: R-00

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I norganics -	General	Chemical	Parameters	(Soil)
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Envirolab I D	Units	PQL	MFG0290-01	MFG0290-02	MFG0290-03
Your Reference			R04-1	R04-2	R04-3
Date Sampled			16/07/2024	16/07/2024	16/07/2024
Total Fluoride	mg/kg	50	52	64	73
Fluoride	mg/kg	0.50	0.50	0.55	0.51

OSE II met the standard for PFAS see above

Your Reference: Revision: R-00

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#### I norganics - Moisture (Soil)

Moisture	%	0.10	30	32	35
Date Sampled			16/07/2024	16/07/2024	16/07/2024
Your Reference			R04-1	R04-2	R04-3
Envirolab I D	Units	PQL	MFG0290-01	MFG0290-02	MFG0290-03

#### PFAS Extended List (Soil)

Envirolab I D	Units	PQL	MFG0290-01	MFG0290-02	MFG0290-03
Your Reference			R04-1	R04-2	R04-3
Date Sampled			16/07/2024	16/07/2024	16/07/2024
Perfluorobutanesulfonic acid (PFBS)	µg/kg	0.10	0.91	0.60	0.88
Perfluoropentanesulfonic acid (PFPeS)	µg/kg	0.10	1.2	0.75	0.95
Perfluorohexanesulfonic acid (PFHxS)	µg/kg	0.10	16	11	15
Perfluoroheptanesulfonic acid (PFHpS)	µg/kg	0.10	2.9	2.1	3.1
Perfluorooctanesulfonic acid (PFOS)	µg/kg	0.10	460	370	580
Perfluorodecanesulfonic acid (PFDS)	µg/kg	0.20	58	53	76
Perfluorobutanoic acid (PFBA)	µg/kg	0.20	0.51	0.42	0.48
Perfluoropentanoic acid (PFPeA)	µg/kg	0.20	0.44	0.37	0.42
Perfluorohexanoic acid (PFHxA)	µg/kg	0.10	1.2	0.87	1.0
Perfluoroheptanoic acid (PFHpA)	µg/kg	0.10	0.32	0.22	0.30
Perfluorooctanoic acid (PFOA)	µg/kg	0.10	2.2	1.3	2.0
Perfluorononanoic acid (PFNA)	µg/kg	0.10	<0.30	<0.30	<0.30
Perfluorodecanoic acid (PFDA)	µg/kg	0.50	<0.50	<0.50	<0.50
Perfluoroundecanoic acid (PFUnDA)	µg/kg	0.50	<0.50	<0.50	<0.50
Perfluorododecanoic acid (PFDoDA)	µg/kg	0.50	<0.50	<0.50	<0.50
Perfluorotridecanoic acid (PFTrDA)	µg/kg	0.50	<0.50	<0.50	<0.50
Perfluorotetradecanoic acid (PFTeDA)	µg/kg	5.0	<5.0	<5.0	<5.0
4:2 Fluorotelomer sulfonic acid (4:2 FTS)	µg/kg	0.10	<0.10	<0.10	<0.10
5:2 Fluorotelomer sulfonic acid (6:2 FTS)	µg/kg	0.10	<0.10	<0.10	<0.10
8:2 Fluorotelomer sulfonic acid (8:2 FTS)	µg/kg	0.20	<0.20	<0.20	<0.20
10:2 Fluorotelomer sulfonic acid (10:2 FTS)	µg/kg	0.20	<0.20	<0.20	<0.20
Perfluorooctane sulfonamide (FOSA)	µg/kg	1.0	7.7	6.9	9.8
-Methyl perfluorooctane sulfonamide MeFOSA)	µg/kg	1.0	<1.0	<1.0	<1.0
N-Ethyl perfluorooctane sulfonamide EtFOSA)	µg/kg	1.0	<1.0	<1.0	<1.0
N-Methyl perfluorooctane sulfonamidoethanol	µg/kg	1.0	<1.0	<1.0	<1.0
N-Ethyl perfluorooctane sulfonamidoethanol	µg/kg	5.0	<5.0	<5.0	<5.0
N-Methyl perfluorooctane sulfonamidoacetic acid	µg/kg	0.20	<0.20	<0.20	<0.20
N-Ethyl perfluorooctane sulfonamidoacetic acid	µg/kg	0.20	<0.20	<0.20	<0.20
	%				
	%				
Total +ve PFHxS+PFOS	µg/kg	0.10	480	380	600
Total +ve PFOA+PFOS	µg/kg	0.10	460	370	580
Fotal +ve PFAS	µg/kg	0.10	550	450	690
	%				
	%				
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	%				

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#### PFAS Extended List (Soil)

Envirolab I D	Units	PQL	MFG0290-01	MFG0290-02	MFG0290-03	
Your Reference			R04-1	R04-2	R04-3	
Date Sampled			16/07/2024	16/07/2024	16/07/2024	
	%					
	%					
	%					
	%					
	%			Extr	ction Internal Standard 13C2 PFUnDA	
	%				Extraction Internal Standard 13C2 PFDoDA	
	%				Extraction Internal Standard 13C2 PFTeDA	
	%				Extraction Internal Standard 13C2 PFTe	DA
	%				Extraction Internal St.	andard 13C2 4:21
	%				Fvi	raction_Internal S
	%					
	%					Ex
	%					

#### **Result Comments**

l dentifier	Description
[4]	For PFAS Extracted Internal Standards denoted with ## or being outside the 50-150% acceptance range, the respective target analyte results may be unaffected, in other circumstances the PQL has been raised to accommodate the outlier(s).

## Method Summary

Method I D	Methodology Summary
INORG-008	Moisture content determined by heating at 105+/-5 °C for a minimum of 12 hours.
INORG-026	Fluoride determined by ion selective electrode (ISE) based on APHA latest edition, 4500-F-C. Solids are reported from a 1:5 water extract unless otherwise specified.
INORG-138	Total Fluoride by Combustion Ion Chromatography
ORG-020	Soil samples are extracted with Dichloromethane/Acetone and waters with Dichloromethane and analysed by GC-FID. F2 = (>C10-C16)-Naphthalene as per NEPM B1 Guideline on Investigation Levels for Soil and Groundwater (HSLs Tables 1A (3, 4)). Note Naphthalene is determined from the VOC analysis. Note, the Total +ve TRH PQL is reflective of the lowest individual PQL and is therefore "Total +ve TRH" is simply a sum of the positive individual TRH fractions (>C10-C40).
ORG-029	Soil/solid and sorbent samples are extracted with basified Methanol. Waters and soil/sorbent extracts are directly injected and/or concentrated/extracted using SPE. TCLP/ASLP leachates are centrifuged, the supernatant is then analysed (including amendment with solvent) - as per the option in AS4439.3. Analysis is undertaken with LC-MSMS. PFAS results include the sum of branched and linear isomers where applicable. Please note that PFAS results are corrected for Extracted Internal Standards (QSM 5.4 Table B-15 terminology), which are mass labelled analytes added prior to sample preparation to assess matrix effects and verify processing of the sample. PFAS canalytes without a commercially available mass labelled analogue are corrected vs a closely eluting mass labelled PFAS compound. Surrogates are also reported, in this context they are mass labelled PFAS compound. Surrogates are also reported, in (not used for result) correction). Enviration is used discretionally to remove interfering matrix components. Please contact the laboratory if estimates of Measurement Uncertainty are required as per WA DER.

#### **Result Definitions**

Identifier	Description
NR	Not reported
NEPM	National Environment Protection Measure
NS	Not specified
LCS	Laboratory Control Sample
RPD	Relative Percent Difference
>	Greater than
<	Less than
PQL	Practical Quantitation Limit
INS	Insufficient sample for this test
NA	Test not required
NT	Not tested
DOL	Samples rejected due to particulate overload (air filters only)
RFD	Samples rejected due to filter damage (air filters only)
RUD	Samples rejected due to uneven deposition (air filters only)
##	Indicates a laboratory acceptance criteria outlier, for further details, see Result Comments and/or QC Comments

#### **Quality Control Definitions**

#### Blank

This is the component of the analytical signal which is not derived from the sample but from reagents, glassware etc, and is determined by processing solvents and reagents in exactly the same manner as for samples.

#### Surrogate Spike

Surrogates are known additions to each sample, blank, matrix spike and LCS in a batch, of compounds which are similar to the analyte of interest, however are not expected to be found in real samples.

#### LCS (Laboratory Control Sample)

This comprises either a standard reference material or a control matrix (such as a blank sand or water) fortified with analytes representative of the analyte class. It is simply a check sample.

#### Matrix Spike

A portion of the sample is spiked with a known concentration of target analyte. The purpose of the matrix spike is to monitor the performance of the analytical method used and to determine whether matrix interferences exist.

#### Duplicate

This is the complete duplicate analysis of a sample from the process batch. The sample selected should be one where the analyte concentration is easily measurable.

#### Laboratory Acceptance Criteria

Duplicate sample and matrix spike recoveries may not be reported on smaller jobs, however, were analysed at a frequency to meet or exceed NEPM requirements. All samples are tested in batches of 20. The duplicate sample RPD and matrix spike recoveries for the batch were within the laboratory acceptance criteria. Filters, swabs, wipes, tubes and badges will not have duplicate data as the whole sample is generally extracted during sample extraction. Spikes for Physical and Aggregate Tests are not applicable. For VOCs in water samples, three vials are required for duplicate or spike analysis.

General Acceptance Criteria (GAC) - Analyte specific criteria applies for some analytes and is reflected in QC recovery tables.

Duplicates: >10xPQL - RPD acceptance criteria will vary depending on the analytes and the analytical techniques but is typically in the range 20%-50% - see ELN-P05 QAQC tables for details (available on request); <10xPQL - RPD are higher as the results approach PQL and the estimated measurement uncertainty will statistically increase. Matrix Spikes, LCS and Surrogate recoveries: Generally 70-130% for inorganics/metals; 60-140% for organics (+/-50% surrogates) and 10-140% for labile SVOCs (including labile surrogates), ultra trace organics and speciated phenols is acceptable.

In circumstances where no duplicate and/or sample spike has been reported at 1 in 10 and/or 1 in 20 samples respectively, the sample volume submitted was typically insufficient in order to satisfy laboratory QA/QC protocols.

#### **Miscellaneous Information**

When samples are received where certain analytes are outside of recommended technical holding times (THTs), the analysis has proceeded. Where analytes are on the verge of breaching THTs, every effort will be made to analyse within the THT or as soon as practicable.

Where sampling dates are not provided, Envirolab are not in a position to comment on the validity of the analysis where recommended technical holding times may have been breached. We have taken the sampling date as being the date received at the laboratory.

Two significant figures are reported for the majority of tests and with a high degree of confidence, for results <10\*PQL, the second significant figure may be in doubt i.e. has a relatively high degree of uncertainty and is provided for information only.

Measurement Uncertainty estimates are available for most tests upon request.

Analysis of aqueous samples typically involves the extraction/digestion and/or analysis of the liquid phase only (i.e. NOT any settled sediment phase but inclusive of suspended particles if present), unless stipulated on the Envirolab COC or by correspondence. Notable exceptions include certain Physical Tests (pH/EC/BOD/COD/Apparent Colour etc.), Solids testing, Total Recoverable metals and PFAS where sediment/solids are included by default.

Urine Analysis - The BEI values listed are taken from the 2022 edition of

Air volume measurements are not covered by Envirolab's NATA accreditation.

# Data Quality Assessment Summary MFG0290

#### **Client Details**

Client Your Reference Date I ssued Hibbs & Associates Pty Ltd S13167 23/07/2024

#### **Recommended Holding Time Compliance**

No recommended holding time exceedances

## **Quality Control and QC Frequency**

QC Туре	Compliant	Details
Blank	Yes	No Outliers
LCS	Yes	No Outliers
Duplicates	No	Duplicate Outliers Exist - See detailed list below
Matrix Spike	No	Matrix Spike Outliers Exist - See detailed list below
Surrogates / Extracted Internal Standards	No	Surrogates / Extracted ISTD Outliers Exist - See detailed list below
QC Frequency	Yes	No Outliers

Surrogates/Extracted Internal Standards, Duplicates and/or Matrix Spikes are not always relevant/applicable to certain analyses and matrices. Therefore, said QC measures are deemed compliant in these situations by default. See Laboratory Acceptance Criteria for more information

# Data Quality Assessment Summary MFG0290

## Recommended Holding Time Compliance

Analysis	Sample Number(s)	Date Sampled	Date Extracted	Date Analysed	Compliant
sTRH   Soil	4-5	16/07/2024	18/07/2024	18/07/2024	Yes
Fluoride   Soil	1-3	16/07/2024	19/07/2024	19/07/2024	Yes
Total Fluoride by CIC   Soil	1-3	16/07/2024	23/07/2024	23/07/2024	Yes
Moisture   Soil	1-3	16/07/2024	18/07/2024	19/07/2024	Yes
PFAS EXT-ISTD   Soil	1-3	16/07/2024	18/07/2024	20/07/2024	Yes
PFAS-Extended   Soil	1-3	16/07/2024	18/07/2024	20/07/2024	Yes

#### **Outliers: Duplicates**

Sample I D	Duplicate ID	Analyte	% Limits	RPD
BFG3021-DUP3#	DUP3	TRH >C16-C34 (F3)	50.00	200[5]
DRG-029  PFAS Ext	tended List (Soil)  Batch	BFG3018		
Sample I D	Duplicate ID	Analyte	% Limits	RPD
MFG0290-01	DUP2	Perfluorononanoic acid (PFNA)	50.00	200[5]
NORG-138  I norga	anics - General Chemical	Parameters (Soil)   Batch BFG3753		
Sample I D	Analyte		% Limits	% Recovery
MFG0290-02	Total Flu	ıoride	70 - 130	##[2]
)RG-020  Semi-vol	latile TRH (Soil)  Batch I	3FG3021		
RG-020  Semi-vol Sample I D	latile TRH (Soil)  Batch I Analyte		% Limits	% Recovery

BFG3021-MS1#	TRH >C34-C40 (F4)	60 - 140	##[3]
BFG3021-MS1#	TRH C29-C36	60 - 140	##[3]

## ORG-029| PFAS Extended List (Soil)| Batch BFG3018

Sample I D	Analyte	% Limits	% Recovery
MFG0290-02	Perfluorooctanesulfonic acid (PFOS)	60 - 140	##[1]

# Data Quality Assessment Summary MFG0290

## Outliers: Surrogate / Extracted Internal Standards

#### ORG-029| PFAS Extended List (Matrix)| Batch BFG3018

Sample I D	Analyte	% Limits	% Recovery
MFG0290-01	Extraction Internal Standard 13C5 PFNA	50 - 150	37.7 [4]
	Extraction Internal Standard 13C2 8:2FTS	50 - 150	## [4]
MFG0290-02	Extraction Internal Standard 13C5 PFNA	50 - 150	35.7 [4]
	Extraction Internal Standard 13C2 8:2FTS	50 - 150	## [4]
MFG0290-03	Extraction Internal Standard 13C5 PFNA	50 - 150	32.8 [4]
	Extraction Internal Standard 13C2 8:2FTS	50 - 150	## [4]

#### **Quality Control MFG0290**

## ORG-020| Semi-volatile TRH (Soil) | Batch BFG3021

Analyte	Units	PQL	Blank	DUP1 BFG3021-DUP1# Samp   QC   RPD %	DUP2 BFG3021-DUP2# Samp   QC   RPD %	LCS %	Spike % BFG3021-MS1#
TRH C10-C14	mg/kg	50	<50	<50 <50 [NA]		104	97.1
TRH C15-C28	mg/kg	100	<100	<100 <100 [NA]		88.1	80.7
TRH C29-C36	mg/kg	100	<100	<100 <100 [NA]		91.5	##[3]
TRH >C10-C16	mg/kg	50	<50	<50 <50 [NA]		81.8	80.2
TRH >C16-C34 (F3)	mg/kg	100	<100	<100 <100 [NA]		91.0	77.1
TRH >C34-C40 (F4)	mg/kg	100	<100	<100 <100 [NA]		83.4	##[3]

Analyte	Units	PQL	Blank	DUP3 BFG3021-DUP3#	DUP4 BFG3021-DUP4#	LCS %	
-				Samp   QC   RPD %	Samp   QC   RPD %		
TRH C10-C14	mg/kg	50		<50 <50 [NA]		[NA]	
TRH C15-C28	mg/kg	100		<100 <100 [NA]		[NA]	
TRH C29-C36	mg/kg	100		<100 <100 [NA]		[NA]	
TRH >C10-C16	mg/kg	50		<50 <50 [NA]		[NA]	Surrogate o-Te
TRH >C16-C34 (F3)	mg/kg	100		117 <100 200 [5]		[NA]	
TRH >C34-C40 (F4)	mg/kg	100		<100 <100 [NA]		[NA]	

# The QC reported was not specifically part of this workorder but formed part of the QC process batch.

# INORG-026 | I norganics - General Chemical Parameters (Soil) | Batch BFG3251

Analyte	Units	PQL	Blank	DUP1 MFG0290-01 Samp   QC   RPD %	DUP2 BFG3251-DUP2# Samp   QC   RPD %	LCS %	<b>Spike %</b> MFG0290-02
Fluoride	mg/kg	0.50	<0.50	0.504 0.502 0.416	30.5 30.5 0.00	81.8	87.0

# The QC reported was not specifically part of this workorder but formed part of the QC process batch.

#### INORG-138 | Inorganics - General Chemical Parameters (Soil) | Batch BFG3753

Analyte	Units	PQL	Blank	DUP1 MFG0290-01 Samp   QC   RPD %	LCS %	<b>Spike %</b> MFG0290-02
Total Fluoride	mg/kg	50	<50	52.0 51.7 0.598	98.0	##[2]

#### INORG-008 | Inorganics - Moisture (Soil) | Batch BFG3013

				DUP1	DUP2	LCS %
Analyte	Units	PQL	Blank	BFG3013-DUP1#	BFG3013-DUP2#	
				Samp   QC   RPD %	Samp   QC   RPD %	
Moisture	%	0.1		23.3 26.6 13.3	12.1 11.0 9.17	[NA]

# The QC reported was not specifically part of this workorder but formed part of the QC process batch.

# Quality Control MFG0290

## ORG-029| PFAS Extended List (Soil) | Batch BFG3018

Analyte	Units	PQL	Blank	DUP1 MFG0290-01 Samp   QC   RPD %	DUP2 MFG0290-01 Samp   QC   RPD %	LCS %	<b>Spike %</b> MFG0290-02
Perfluorobutanesulfonic acid (PFBS)	µg/kg	0.10	<0.10	0.909 0.774 16.0	0.909 0.736 20.9	101	99.6
Perfluoropentanesulfonic acid (PFPeS)	µg/kg	0.10	<0.10	1.15 0.980 16.3	1.15 0.725 45.6	101	113
Perfluorohexanesulfonic acid (PFHxS)	µg/kg	0.10	<0.10	15.7 12.6 21.9	15.7 11.5 30.8	95.9	105
Perfluoroheptanesulfonic acid (PFHpS)	µg/kg	0.10	<0.10	2.94 2.34 22.6	2.94 2.20 28.7	104	110
Perfluorooctanesulfonic acid (PFOS)	µg/kg	0.10	<0.10	461 398 14.6	461 371 21.5	97.1	##[1]
Perfluorodecanesulfonic acid (PFDS)	µg/kg	0.20	<0.20	57.8 48.3 17.9	57.8 46.4 22.0	95.4	71.2
Perfluorobutanoic acid (PFBA)	µg/kg	0.20	<0.20	0.506 0.457 10.2	0.506 0.362 33.1	96.9	103
Perfluoropentanoic acid (PFPeA)	µg/kg	0.20	<0.20	0.438 0.394 10.6	0.438 0.365 18.0	98.1	102
Perfluorohexanoic acid (PFHxA)	µg/kg	0.10	<0.10	1.23 1.13 8.40	1.23 0.924 28.3	95.3	101
Perfluoroheptanoic acid (PFHpA)	µg/kg	0.10	<0.10	0.316 0.290 8.71	0.316 0.229 31.8	96.6	97.6
Perfluorooctanoic acid (PFOA)	µg/kg	0.10	<0.10	2.17 1.64 27.7	2.17 1.43 41.3	98.9	104
Perfluorononanoic acid (PFNA)	µg/kg	0.10	<0.10	<0.30 <0.30 [NA]	## ## [NA] [5]	102	101
Perfluorodecanoic acid (PFDA)	µg/kg	0.50	<0.50	<0.50 <0.50 [NA]	<0.50 <0.50 [NA]	99.2	109
Perfluoroundecanoic acid (PFUnDA)	µg/kg	0.50	<0.50	<0.50 <0.50 [NA]	<0.50 <0.50 [NA]	93.4	113
Perfluorododecanoic acid (PFDoDA)	µg/kg	0.50	<0.50	<0.50 <0.50 [NA]	<0.50 <0.50 [NA]	98.4	105
Perfluorotridecanoic acid (PFTrDA)	µg/kg	0.50	<0.50	<0.50 <0.50 [NA]	<0.50 <0.50 [NA]	96.3	109
Perfluorotetradecanoic acid (PFTeDA)	µg/kg	5.0	<5.0	<5.0 <5.0 [NA]	<5.0 <5.0 [NA]	94.6	95.5
4:2 Fluorotelomer sulfonic acid (4:2 FTS)	µg/kg	0.10	<0.10	<0.10 <0.10 [NA]	<0.10 <0.10 [NA]	109	114
6:2 Fluorotelomer sulfonic acid (6:2 FTS)	µg/kg	0.10	<0.10	<0.10 <0.10 [NA]	<0.10 <0.10 [NA]	107	130
8:2 Fluorotelomer sulfonic acid (8:2 FTS)	µg/kg	0.20	<0.20	<0.20 <0.20 [NA]	<0.20 <0.20 [NA]	105	108
10:2 Fluorotelomer sulfonic acid (10:2 FTS)	µg/kg	0.20	<0.20	<0.20 <0.20 [NA]	<0.20 <0.20 [NA]	98.3	127
Perfluorooctane sulfonamide (FOSA)	µg/kg	1.0	<1.0	7.67 6.77 12.4	7.67 6.51 16.4	98.4	97.4
N-Methyl perfluorooctane sulfonamide (MeFOSA)	µg/kg	1.0	<1.0	<1.0 <1.0 [NA]	<1.0 <1.0 [NA]	104	110
N-Ethyl perfluorooctane sulfonamide (EtFOSA)	µg/kg	1.0	<1.0	<1.0 <1.0 [NA]	<1.0 <1.0 [NA]	104	110
N-Methyl perfluorooctane sulfonamidoethanol	µg/kg	1.0	<1.0	<1.0 <1.0 [NA]	<1.0 <1.0 [NA]	97.4	99.3
N-Ethyl perfluorooctane sulfonamidoethanol	µg/kg	5.0	<5.0	<5.0 <5.0 [NA]	<5.0 <5.0 [NA]	105	114
N-Methyl perfluorooctane sulfonamidoacetic acid	µg/kg	0.20	<0.20	<0.20 <0.20 [NA]	<0.20 <0.20 [NA]	105	102
N-Ethyl perfluorooctane sulfonamidoacetic acid	µg/kg	0.20	<0.20	<0.20 <0.20 [NA]	<0.20 <0.20 [NA]	92.0	107

Your Reference:S13167Revision: R-00Certificate of Analysis Generated: 23/07/2024 18:14

# Quality Control MFG0290

#### **QC** Comments

l dentifier	Description
[1]	Spike recovery is not applicable due to the relatively high analyte background in the sample (>3* spike level). However, the LCS recovery is within acceptance criteria.
[2]	Spike recovery is outside routine acceptance criteria (70-130%), this may be due to suspected non-homogeneity and/or matrix interference effects. However, an acceptable recovery was achieved for the LCS.
[3]	Spike recovery is outside routine acceptance criteria (60-140%), this may be due to suspected non-homogeneity and/or matrix interference effects. However, an acceptable recovery was achieved for the LCS.
[4]	For PFAS Extracted Internal Standards denoted with ## or being outside the 50-150% acceptance range, the respective target analyte results may be unaffected, in other circumstances the PQL has been raised to accommodate the outlier(s).
[5]	Duplicate %RPD may be flagged as an outlier to routine laboratory acceptance, however, where one or both results are <10*PQL, the RPD acceptance criteria increases exponentially.

Complete test report below, with the Bacteria count MPN test at the end of the report.



Our Reference: S13167-R06

11 November 2024

CMTA International Pty Ltd 158 Garretts Road, Longford, Vic 3851 Australia

Attention: Mr Joel Farhadian

Dear Mr Farhadian,

#### re: ASSESSMENT OF PFAS CONTAMINATED SOIL - ROUND 6 - DAY 90

#### 1. Introduction

CMTA International Pty Ltd (Client) engaged Hibbs & Associates Pty Ltd (Hibbs) to collect soil samples containing Per- and polyfluoroalkyl substances (PFAS) including various amounts of Perfluoro-octanesulfonic acid (PFOS), Perfluoro-octanoic acid (PFOA) and Perfluoro-hexane-sulfonic acid (PFHxS) PFAS/PFOS from a simple pilot plant designed to reduce the amount of PFAS/PFOS/PFHxS via microbiological degradation. PFAS are a group of synthetically manufactured chemicals known as "forever chemicals" due to their persistence in the environment.

The Department of Defence in Australia have historically used AFFF foam on military bases in emergency situations and in their firefighting exercises, this legacy is well documented in the public domain. It was reported that the PFAS contaminated soil sample was provided by Defence, we have no further details about the history or the origin of the sample.

The contaminated soil was collected from a site confirmed to have been contaminated with PFAS. The soil samples were collected at and tested for a suit of PFAS chemicals to determine the baseline level of contamination before remediation of the soil. The soil samples were collected and tested for 28 PFAS analytes, total fluoride (organic and inorganic) and soluble inorganic fluoride to determine the baseline level of contamination which existed in the soil sample prior to bioremediation of the soil. Samples were also collected at intervals specified by the Client during the remediation process to assess the effectiveness of the process.

These samples were sent to a NATA accredited laboratory for analysis to determine the concentration of PFAS (per-and poly-fluoroalkyl substances) at a specific time interval after the treatment was added, these are the fourth set of samples collected 90 days after the biodegradation chemicals and biological additives were combined with the test soil. Subjecting the contaminated soil with water, air and additives (post-baseline) which are designed to breakdown PFAS in an accelerated time frame. The samples were collected from an irrigated plastic container (pilot plant) with no drainage, which was stored at 7 Erebus Street, Keilor Park, VIC 3042, refer to Appendix B for methodology. The water level was observed to be at least 2 to 3 centimetres above the height of the submerged soil refer to Appendix A.

It is the understanding of Hibbs that the methodology and the surfactant/enzyme combination are proprietary and as such Hibbs do not know what the composition of the remediation chemicals or the basis from which this methodology has been developed. Hibbs sole purpose is to ensure that the samples are collected correctly and analysed by a reputable NATA accredited laboratory to conduct PFAS testing and to report the levels of PFAS after each round of testing.

HIBBS & ASSOCIATES PTY LTD ABN 12 608 093 134 Suite B, 255 Rawson Street, Auburn NSW 2144 P.O. Box 4266, Homebush NSW 2140 P +61 2 9746 3244E info@hibbs.com.auW www.hibbs.com.au



# 2. Objectives

The Client is seeking to determine the efficacy of a new technology introduced for the remediation of PFAS contaminated soil. The objective of this sampling programme is to:

- determine the baseline concentrations of PFAS in the contaminated soil prior to treating the subject soil or commencement of remediation trial.
- determine PFAS concentrations during remediation at specific time intervals specified in the methodology provided by the Owner of the new technology. The objective is to provide an indication of the effectivity or otherwise of the remediation process.
- determine the microbiological activity and Total Organic Fluoride content of the subject soil.

The testing post treatment schedule is listed below, the same sampling method will be used for each round of testing until the desired level of remediation is achieved. The sampling regime was specified by the Client.

- 7 days (Round 2) (20/06/2024)
- 15 days (Round 3) (28/06/2024)
- 30 days (Round 4) (16/07/2024)
- 45 days (Round 5) (1/08/2024)
- 90 days (Round 6) (11/09/2024)



# 3. Sampling Methodology

Samples were collected manually from the container (pilot plant) that stores the test soil collected by the Client, refer to Appendix A. Due to the treatment method, the soil samples contained additional water and a stainless-steel shovel was used to scoop the samples into jars. The shovel was also used to drain excess water before pouring the samples into the jars. The samples were transferred into sample jars provided by the laboratory. A unique sample identifier was used for each sample collected, based on the sampling point number, and the samples were delivered to a NATA accredited laboratory for the chemical analysis along with a chain of custody (COC) form, refer to Appendix C.

For each round of testing, the three-sampling standard method will be used. Each of the three sample extractions will not be mixed, but each is kept separately in a jar and labelled sample 1, 2, 3. Sample locations are shown in the diagram below Figure 1.

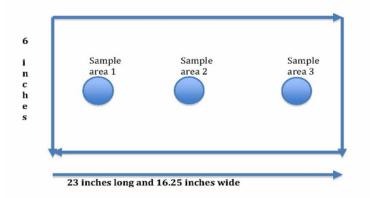


Figure 1: Sampling Locations



#### 4. **Testing Methodology**

The tests which are listed as being commercially available at Envirolab for PFAS and are NATA certified include PFAS speciation (28 PFAS analytes), Total Oxidizable Precursor (TOP) Assay, Total Fluoride (organic and inorganic), and soluble Inorganic Fluoride. Of these five tests only the TOP test was not conducted. In addition to the four tests conducted being PFAS speciation (28 PFAS analytes), Total Fluoride (organic and inorganic), soluble Inorganic Fluoride and Total Organic Fluoride, a total microbial plate count was also conducted on the subject soil. Total Organic Fluoride was performed to provide some insight into the wildly fluctuating results we are receiving for total fluoride. From this testing we can determine if it is the organic or inorganic fluoride which may be attributed to these variations in the data. A microbiological aerobic plate count was performed to provide an indication of the microbial activity in the subject soil.

4.1	PFAS Speciation

PFAS Extended suite	Acronym	PQL (µg/ kg)
Perfluorobutanesulfonic acid (PFBS)	PFBS	0.1
Perfluoropentanesulfonic acid (PFPeS)	PFPeS	0.1
Perfluorohexanesulfonic acid (PFHxS)	PFHxS	0.1
Perfluoroheptanesulfonic acid (PFHpS)	PFHpS	0.1
Perfluorooctanesulfonic acid (PFOS)	PFOS	0.1
Perfluorodecanesulfonic acid (PFDS)	PFDS	0.2
Perfluorobutanoic acid (PFBA)	PFBA	0.2
Perfluoropentanoic acid (PFPeA)	PFPeA	0.2
Perfluorohexanoic acid (PFHxA)	PFHxA	0.1
Perfluoroheptanoic acid (PFHpA)	PFHpA	0.1
Perfluorooctanoic acid (PFOA)	PFOA	0.1
Perfluorononanoic acid (PFNA)	PFNA	0.1
Perfluorodecanoic acid (PFDA)	PFDA	0.5
Perfluoroundecanoic acid (PFUnDA)	PFUnDA	0.5
Perfluorododecanoic acid (PFDoDA)	PFDoDA	0.5
Perfluorotridecanoic acid (PFTrDA)	PFTrDA	0.5
Perfluorotetradecanoic acid (PFTeDA)	PFTeDA	5
4:2 Fluorotelomer sulfonic acid (4:2 FTS)	4:2 FTS	0.1
6:2 Fluorotelomer sulfonic acid (6:2 FTS)	6:2 FTS	0.1
8:2 Fluorotelomer sulfonic acid (8:2 FTS)	8:2 FTS	0.2
10:2 Fluorotelomer sulfonic acid (10:2 FTS)	10:2 FTS	0.2
Perfluorooctane sulfonamide (FOSA)	FOSA	1
N-Methyl perfluorooctane sulfonamide (MeFOSA)	MeFOSA	1
N-Ethyl perfluorooctane sulfonamide (EFOSA)	<b>E</b> FOSA	1
N-Methyl perfluorooctane sulfonamidoethanol	MeFOSE	1
N-Ethyl perfluorooctane sulfonamidoethanol	<b>E</b> FOSE	5

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PFAS Extended suite	Acronym	PQL (µg/ kg)
N-Methyl perfluorooctane sulfonamidoacetic acid	MeFOSAA	0.2
N-Ethyl perfluorooctane sulfonamidoacetic acid	EFOSAA	0.2
PFAS Short suite		PQL (µg/kg)
Perfluorohexanesulfonic acid (PFHxS)	PFHxS	0.1
Perfluorooctanesulfonic acid (PFOS)	PFOS	0.1
Perfluorooctanoic acid (PFOA)	PFOA	0.1
6:2 Fluorotelomer sulfonic acid (6:2 FTS)	6:2 FTS	0.1
8:2 Fluorotelomer sulfonic acid (8:2 FTS)	8:2 FTS	0.2

PFAS speciation is based on the USEPA 1633 (this method is using Basified methanol or Methanolic ammonium hydroxide) running through a liquid chromatography separation technology and identified with a specific detector using a mass spectrophotometer i.e. Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) and references ASTM D7968-14 Determination of Perfluorinated Compounds in Soil by Liquid.

#### 4.2 Total Oxidisable Precursor

TOP (Total Oxidisable Precursor) Assay for Organic and Inorganic Florine reports Oxidisable precursors. Pre-treatment step consists of oxidant digestion under strong alkaline conditions at 85°C for 6 hours. The digestion converts previously undetectable PFAS to PFCA and PFSA. Treated samples are then neutralised and analysed via LC-MS/MS. The process enables detection of the component previously not available for analysis. The sample is run through a liquid chromatograph separation instrument technique and identified with a specific detector using a mass spectrophotometer i.e. Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS).

#### 4.3 Total Fluoride (Inorganic/ Organic)

The soil sample is placed into an oxygen bomb and ignited, the resulting pressure and temperature are able to convert all of the carbon in the sample to carbon dioxide. The residue is run through an ion chromatography column to identify the anions present. Total fluoride is measured and represents all organic and inorganic fluoride originally present in the sample.

#### 4.4 Total Soluble Inorganic Fluoride

Soluble Inorganic fluorine is measured by taking a 1:5 water extraction, then analyze for soluble inorganic fluoride using a fluoride ion selective electrode, however this will only measure soluble inorganic fluorides. Likewise for Organic fluoride, we could do a Total Oxidizable Precursor Assay (TOP assay) which would break down longer chain Poly-Flouro Akyl Substances into shorter chains.



## 5. Assessment Criteria

The results of this testing do not have a regulatory limit to compare to as such, rather they will be compared to future tests conducted at scheduled intervals post treatment. Treatment Evaluation of the remediation process cannot be achieved until after the treatment has commenced following the initial round of baseline testing.

The samples will be analysed for the extended list of PFAS in soil, moisture, and fluoride.

The PFAS National Environmental Management Plan 2.0 (NEMP) has developed guidance around exposure direct and indirect ecological exposure and industrial/commercial exposure. Eventually testing results can be used to compare to values specified in the NEMP.

#### Table 5.1: PFAS NEM P Exposures

	PFOS <sup>1</sup> mg/kg	PFOA <sup>2</sup> mg/kg	Sum of PFHxS <sup>3</sup> and PFOS mg/kg	
EQL	0.0001	0.0001	0.0002	
PFAS NEMP 2020 Ecological direct exposure	1	10		
PFAS NEMP 2020 Ecological indirect exposure	0.01			
PFAS NEMP 2020 Industrial/ commercial (HIL D)		50	20	
PFAS NEMP 2020 Public open space (HIL C)		10	1	
PFAS NEMP 2020 Residential with minimal opportunities for soil access (HIL B)		20	2.0	
PFAS NEMP 2020 Residential with garden/accessible soil (HIL A)		0.1	0.01	

The above values from the existing NEMP guidance criteria provide some reference to estimate the degree of PFAS contamination present in the soil. It should however be noted that this is a very small number of bio-persistent analytes used as the key-markers to fully characterise total PFAS contamination and as such should be interpreted with caution.

It is well documented that terminal products from the degradation of straight and branched chain PFAS compounds with chain lengths >8 carbon atoms long, will ultimately break down to PFOS, PFHxS and PFOA. It is very well established that these chemicals do not readily breakdown any further once they have been degraded (oxidised) to these entities, hence why they are described as terminal PFAS degradation products in the literature. Therefore, any reduction in the levels of PFOS, PFHxS or PFOA is a very strong indication of terminal PFAS entities being broken down using biological processing for the remediation of PFAS contaminated soils.

Defence is seeking evidence of demineralisation or the removal of fluorine from the organic carbon chains from Per and poly fluoro alkyl substances after the remediation process. This is not straightforward and only under specific circumstances will this be clearcut or easily demonstrated.

. CMTA International Pty Ltd - Reference No. S13167-R06 158 Garretts Road, Longford, Vic 3851 Australia: Assessment of PFAS Contaminated Soil - Round 6 - Day 90

<sup>1</sup> PFOS: Perfluorooctanesulfonic acid

<sup>2</sup> PFOA: Perfluorooctanoic acid

<sup>3</sup> PFHxS: Perfluorohexanesulfonic acid



## 6. Results

## 6.1 Total Aerobic Microbial Plate Count 22C<sup>0</sup>

	Units	RO6-01	RO6-02	R06-03
Aerobic Plate Count	cfu/g (Dry Weight)	9200000	9200000	1.9x10 <sup>7</sup>

Average=1.25 x10<sup>7</sup> cfu/g



#### **Results Cont'd**

#### Table 6.1: Summary of Laboratory Analytical Results

NEM P Crite	eria			M oisture C	Content (%)	Total Fluoride (mg/ kg	Total Fluoride (mg/ kg)		kg)	PFOA <sup>2</sup> (µg/ kg)		Sum of PFHxS <sup>3</sup> and	
PFAS NEME	2020 Ecologica	l direct exposure						1000		10,000			
PFAS NEME	2020 Ecologica	l indirect exposure		T				10					
PFAS NEN	PFAS NEMP 2020 Industrial/ commercial (HIL D)										50,000		
Round	Sample Name	Sample Date	Sampling Round	Lab Result (%)	Variation	Lab Result (mg/kg)	Variation	Lab Result (µg∕kg)	Variation	Lab Result (µg/kg)	Variation	Lab Result (µg/kg)	Variation
	S13167-R01-1	13/06/2024	Baseline	12	Baseline	230	Baseline	1100	Baseline	4.1	Baseline	1100	Baseline
1	\$13167-R01-2	13/06/2024	Baseline	13	Baseline	240	Baseline	1100	Baseline	4.1	Baseline	1100	Baseline
	S13167-R01-3	13/06/2024	Baseline	12	Baseline	240	Baseline	970	Baseline	3.1	Baseline	1100	Baseline
	S13167-R02-1	20/06/2024	R02-7 days	36	200%	170	-26%	1600	45%	4.6	12%	1600	45%
2	S13167-R02-2	20/06/2024	R02-7 days	31	138%	230	-4%	1300	18%	3.4	-17%	1300	18%
	S13167-R02-3	20/06/2024	R02-7 days	30	150%	180	-25%	910	-6%	3.7	19%	940	-15%
	S13167-R03-1	28/06/2024	RO3-15 days	33	175%	300	30%	940	-15%	3.1	-24%	960	-13%
3	S13167-R03-2	28/06/2024	RO3-15 days	34	162%	380	58%	1200	9%	4.3	5%	1200	9%
	S13167-R03-3	28/06/2024	RO3-15 days	32	167%	239	0%	1100	13%	4.1	32%	1100	0%
	S13167-R04-1	16/07/2024	RO4-30 days	30	150%	52	-77%	460	-58%	2.2	-46%	480	-56%
4	S13167-R04-2	16/07/2024	RO4-30 days	32	146%	64	-73%	370	-66%	1.3	-68%	380	-65%
	S13167-R04-3	16/07/2024	RO4-30 days	35	192%	73	-70%	580	-40%	2	-35%	600	-45%
	S13167-R05-1	1/08/2024	RO5-45 days	27	125%	100.00	-57%	270	-75%	0.75	-82%	280	-75%
5	\$13167-R05-2	1/08/2024	RO5-45 days	30	131%	180	-25%	340	-69%	1	-76%	350	-68%
	S13167-R05-3	1/08/2024	RO5-45 days	34	183%	190	-21%	450	-54%	1.4	-55%	460	-58%
	\$13167-R06-1	11/09/2024	RO5-90 days	25	108%	360.00	57%	540	-51%	0.93	-77%	550	-50%
6	S13167-R06-2	11/09/2024	RO5-90 days	24	85%	230	-4%	430	-61%	0.76	-81%	440	-60%
	S13167-R06-3	11/09/2024	RO5-90 days	29	142%	400	67%	660	-32%	1.2	-61%	670	-39%
Average	\$13167-R01	13/06/2024	R01 - Baseline	12.33	Baseline	236.67	Baseline	1056.667	Baseline	3.77	Baseline	1100.0	Baseline
Per round	S13167-R02	20/06/2024	R02 - 7 days	32.33	162%	193.33	-18%	1270	20%	3.90	4%	1280.0	16%
	S13167-R03	28/06/2024	R03 - 15 days	33	168%	306.33	29%	1080	2%	3.83	2%	1086.7	-1%
	S13167-R04	28/06/2024	R04 - 30 days	32.33	162%	63.00	-73%	470	-56%	1.83	-51%	486.7	-56%
	S13167-R05	1/08/2024	RO5-45 days	30.33	146%	156.7	-34%	353.3	-67%	1.05	-72%	363.3	-67%
	S13167-R06	11/09/2024	RO6-90 days	26	111%	330.0	39%	543.3	-49%	0.96	-74%	553.3	-50%

1 PFOS: Perfluorooctanesulfonic acid 2 PFOA: Perfluorooctanoic acid 3 PFHxS: Perfluorohexanesulfonic acid

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# Comparison of Laboratory Results After Six Rounds of Testing

Analyte	Ruoride	Total flouride	M oisture Content	Perfluorobutane sulfonic acid (PFBS)	Perfluorodecanesulfonic add (PFDS)	Perfluorohexane sulfonic acid (PFHxS)	Perfluorohexanoicacid (PFHxA)	Perfluorooctane sulfonamide (PFOSA)	Perfluorooctanesulfonic acid (PFOS)	Perfluorooctanoicacid (PFOA)	Perfluoropentane sulfonic acid (PFPeS)	Perfluoropentanoic add (PFPeA)	Sum of PFAS	Sum of PFHXS and PFOS	Sum of USEPA PFAS (PFOS+ PFOA)*
	mg/ kg	mg/ kg	%	µg/ kg	µg/ kg	µg/ kg	µg/ kg	µg/ kg	µg/ kg	µg/ kg	µg/ kg	µg/ kg	µg/ kg	µg/ kg	µg/ kg
ROUND 1	<0.50	237	12	2.1	15	32	3.5	9.6	1000	4	2.3	0.76	1,107	1,052	1040
ROUND 2	<0.50	193	32	1.8	64	26	2.9	10	1300	3.9	2.3	0.69	1,367	1,280	1270
ROUND 3	<0.50	303	33	1.5	85	26	2.4	9.6	1100	3.8	1.9	0.41	1,200	1,087	1080
ROUND 4	0.52	63	32	0.8	62	14	1.2	8.1	470	1.8	1.0	0.41	563	487	470
ROUND 5	0.76	157	30	0.4	43	8.8	0.6	5.1	353	1.1	0.59	0.30	417	363	353
ROUND 6	1.2	330	26	0.4	42	6.2	0.8	4.3	543	1.0	0.49	0.36	603	553	543

## Table 6.2: Summary of Laboratory Analytical Results After Six Rounds

Yellow	No Trend Observed since last round
Green	Decrease in concentration since last round
Red	Increase in concentration since last round

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## 7. Results Discussion

- The analytical results for Round 6 (R06) reflect the period 90 days after the water and proprietary chemicals/surfactants/enzymes were applied to the PFAS-contaminated soil in the pilot plant. During this period, soil moisture levels dropped to an average of 26%, representing a further decrease from 30% in the previous round (R05). Previous overwatering and poor mixing of the soil over this extended period has led to a higher variability of results in this round compared to the previous round.
- The aerobic microbial activity in R06 has shown a decline, with an average count of 1.25 x 10<sup>7</sup> cfu/g, compared to 2.3 x 10<sup>7</sup> cfu/g in R05. This reduction suggests a potential impact on microbial efficiency, and a deterioration of the system. i.e. no longer sustainable. Further monitoring is therefore not recommended as this represents a reduction in microbial activity potentially below levels required for effective remediation.
- Given the above two dot points it is recommended to end this trial and disregard the original and last rounds due to system instabilities resulting in increased variability within the trending of results. This may be the result of incomplete mixing, depletion of the original soil mass, reduction in microbial activity and/or approaching the limits of detection for some PFAS analytes.
- The total fluoride concentration in R06 showed a notable increase, rising from 157 mg/kg in R05 to 330 mg/kg in R06, indicating a 39% increase. This is a significant deviation from the earlier round. The fluctuations observed in the fluoride concentrations were previously identified as originating from insoluble inorganic fluoride present in the system which remain unexplained at this time.
- From Round 4 to Round 6 there has been a steady increase in soluble inorganic fluoride. This
  could potentially be used as a marker for mineralisation organic fluoride and is supported by
  exhibiting a proportionate increase to the decrease in organic fluoride with positive upward trend
  once surpassing the detection limit of the method.
- PFOS concentrations in Round 6 (R06) experienced a partial rebound after a notable decline in previous rounds. While the PFOS levels remain reduced by 49% compared to the baseline, the increase from 353 µg/kg (R05) to 543 µg/kg is unexpected and further evidence to suggest the system is not in a steady state and should be terminated to prevent producing spurious results.
- Similarly, as PFOS accounts for more than 94% of all organic fluoride present, deviations in the analytical result of this analyte have a carry-on effect to other key parameters such the sum of PFHxS and PFOS, the sum of PFOS and PFOA and the Total sum of PFAS. In R06 PFHxS and PFOS equals 553.3 µg/kg from a R05 result of 363 µg/kg, this still represents a 57% reduction compared to the original baseline. In R06 PFOS and PFOA equals 543 µg/kg from a R05 result of 353 µg/kg, this still represents a 57% reduction compared to the original baseline.
- The PFOA concentrations in R06 decreased further, maintaining a 74% reduction compared to the baseline, with a slight reduction from 1.05 µg/kg in R05 to 0.96 µg/kg in R06. This consistent decline in PFOA levels continues to highlight the effectiveness of the treatment process in mitigating PFOA contamination.
- $^{\circ}~$  PFBS levels remained constant from R05 to R06 with a result of 0.4  $\mu g/kg$ , maintaining an 80 % reduction compared to the baseline.
- PFDS levels were reduced marginally with a result of 41  $\mu$ g/kg in RF06 from 42  $\mu$ g/kg in R05, maintaining a 80 % reduction compared to the baseline.

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- PFHxS levels decreased from 8.8 µg/kg in RO5 to 6.2 in RO6 maintaining a 76% reduction compared to the baseline.
- PFHxA levels increased marginally from 0.6 μg/kg in RO5 to 0.8 in RO6 maintaining a 76% reduction compared to the baseline.
- PFOSA levels decreased from 5.1 μg/kg in RO5 to 4.3 μg/kg in RO6 maintaining a 57% reduction compared to the baseline.
- PFPeS levels marginally decreased from 0.59 μg/kg in RO5 to 0.49 μg/kg in RO6 maintaining a 79% reduction compared to the baseline.
- \* PFPeA levels increased marginally from 0.30  $\mu g/kg$  in RO5 to 0.36 in RO6 maintaining a 48% reduction compared to the baseline.
- Overall, while significant progress has been made in the reduction of PFAS concentrations, the
  reduction in microbial activity, the increased variability in fluoride levels and the slight rebound in
  PFOS concentrations indicate the system is no longer in equilibrium and/or sustainable to facilitate
  producing reliable / reproducible results. It is our recommendation to terminate the trial and the
  first and last round of results should be discarded when the system was not in a steady state to
  present the most reliable set of data.



## Conclusions

- Moisture Content:
  - Samples R06-1, R06-2, and R06-3 show a decrease in moisture content compared to round 5 (R05). The average moisture content is now 26%, which is a reduction from the previous average of 30%. While this level remains above the critical threshold necessary to sustain microbial activity (20 to 30%), the ongoing decrease in moisture levels could potentially impact the effectiveness of the microbial remediation process. A consistent moisture level is vital for optimizing the remediation outcomes in these soil samples.
- Total Fluoride:
  - The total fluoride levels in samples R06-1, R06-2, and R06-3 exhibit a significant increase compared to round 5 (R05), where fluoride levels had decreased. The rise from 157 mg/kg in R05 to 330 mg/kg in R06 indicates a partial rebound in fluoride contamination. Given that this is a closed system, fluctuations in this figure are unexpected.
- Soluble Inorganic Flouride
  - Soluble inorganic fluoride is steadily increasing at a concentration within the same magnitude which is consistent with what would be expected from the demineralisation of the PFAS species presently observed in this subject soil.
- PFOS1:
  - PFOS levels have experienced a rebound in round 6 (R06), rising from 353 μg/kg in R05 to 543 μg/kg in R06. Despite remaining reduced by 49% compared to baseline levels, this increase highlights the need for ongoing monitoring and possibly additional remediation measures to sustain reductions in PFOS contamination across all samples.
- PFOA2:
  - PFOA levels in round 6 (R06) have shown a slight reduction from 1.05 µg/kg in R05 to 0.96 µg/kg in R06, marking a continued decline in PFOA contamination across all samples. This consistent decrease underscores the effectiveness of the treatment process in mitigating PFOA contamination.
- Sum of PFHxS3 and PFOS:
  - The sum of PFHxS and PFOS levels averaged 553.3 µg/kg in round 6 (R06), which represents a 50% reduction compared to baseline. Although this reflects a positive trend in reducing these contaminants, the fluctuations observed necessitate further examination to determine whether these levels will stabilize.
- PFAS
  - Overall, while significant reductions in PFAS substances have been achieved, including key
    markers like PFOS and PFOA, the increased variability in fluoride levels and the rebound in
    PFOS concentrations indicate a need for continuous monitoring and possible adjustments to
    the remediation strategy to maintain effective contaminant reductions over time.



## 8. Recommendations

- Test the subject soil to elucidate the different species of bacteria which are present
- Further testing of the inorganic fluoride to better understand the huge variations observed in Total Fluoride from round to round.

Questions regarding this report should addressed to Robert Gale on +61 419 929 038.

Yours sincerely, HIBBS & ASSOCIATES PTY LTD

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Photograph 1 : Sampling Locations





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## OSE II How-To Soil For PFOS, PFAS, PETRO CHEMICAL,

# PESTICIDES, HERBICIDES

## ON SOIL Demonstration/ TEST Illustrated

I. Required Items to perform test/ demonstration

<u>A. CONTAINER:</u> You need a container that is approximately, 60.96 cm long X 45.72 cm wide, by 15.24 to 20.32 cm deep, the 20.32 cm depth is preferred.

Or a 40 liter aquarium.

B. <u>SOIL:</u> You will need spread the 5 kg of soil to spread to a depth of 10.2 cm covering the Entire bottom of the plastic container 1m3 of sandy soil equals 1500 kg approximately.





Note you can collect soil with PFASalready present, and, if so, this would eliminate the need to apply a contaminant to soil in the plastic container. If contaminated soil is not present, the regular soil will do.



C. Approximately 1500 kg of soil per m3, this test will use 5kg, where 15.33 ml or 16ml of OSE II will be utilized with 800 ml of fresh water.



D. 5 kg of PFAS Contaminated soil will be utilized:

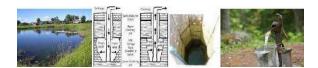
D. <u>WATER:</u> There are three options for water to use, fresh, brackish (mix of salt and fresh water), or sea water. The water must be from an unpolluted natural water source. The water you choose will be used to *maintain a soil moisture level of 60% or more* and will be from the same source of water used when, at the beginning of the test, you mix OSE II with water to apply it to the oiled soil.  The soil that will be used for this laboratory test will be sourced from a natural fresh water source.

Note: The reason one uses a native source of water is because OSE II activates the oil eating microbes naturally present in the water. If an oil spill took place on a river, or pond, lake, inlet, marsh, bay etc. you would use the water in THAT SPILL LOCATION. If you were to use tap water, OSE II would not show a result or a very slow one because tap water is treated, it contains very little, if any bacteria and has been treated/ purified using chemicals. Because OSE II contains no microbes but instead works WITH the oil eating bacteria/ microbes living in their native environments, your demonstration soil and water will need to approximate natural conditions as much as possible.

Examples of usable water sources:



Fresh Water Lake, then a river, and a stream



Fresh water pond or well



Brackish water (where fresh water meets ocean/ sea water



#### Ocean or sea water

Water can be collected in pails or bottles. Collect at least 2 liters before starting the demonstration, and start the aeration of the container of water within an hour after first applying OSE II. You should use the water within an hour of collecting it, if possible. Mechanical aeration is not needed if you are going to replace the water each day.

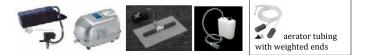


Refreshed Water: If you do not replace water daily, you will need to aerate the water and refresh the water in the pails or bottles at least every three days. Ideal conditions would be extracted fresh water each day if possible.

E. <u>AERATOR</u>: If you cannot refresh the water <u>each day</u> then you will need a small aquarium aerator with a plastic tube to keep the water oxygenated.

F two (2) small aerators and a couple of meters of tubing with 8 spliters for the tubing

1 aerator and tubing for the water, and one for the test vessel



F. <u>SMALL GARDEN TOOLS' SPATULA:</u> You will need small utensils' garden tools to turn the soil. Ensure they are clean and free of contaminants such as pesticides etc.



G. <u>SAMPLE COLLECTION JARS</u>. You will need at least 24 Teflon sealed glass jars to take samples to the lab. (Consult with your lab if necessary to ensure you do not use contaminated jars) The laboratory you use for testing may supply these or may tell you where to procure them. The lab will also tell you the amount of soil they will need to perform a test; this varies between laboratories, so request this information before starting the demonstration based on the US EPA test method 533. You will also need stick on labels for the jars.



 ${\rm H.}\,\underline{\rm A\,spoon}$  or similar implement to mix the samples together when placed in a jar.

I. JAR LABELS: Stick-on labels for the jars.

J ICE CHEST: You will need a small ice chest to take samples to the laboratory.

An Hour before placing a sample extraction in the ice chest place ice in the ice chest to cool the temperature down inside the ice chest.

K. <u>GLOVES</u>: Surgical cloves are required when testing with PFAS, OSE II can wash it the soil off, if hazardous waste (such as PCB's or pesticides etc.) are being tested, surgical gloves will be necessary.

L <u>OSEII</u>: 1, 8 ounce bottle of OSE II will be used for the test. However, if you extract soil that has already been contaminated with PFAS, then you will need to perform a lab test to see what the concentration of the PFAS, if it has not been performed, so the amount of OSE II required can be calculated. For this test the Defense force will have previously tested the soil for contamination levels.





M. <u>MOISTURE METER</u>: You will need a lawn moisture meter, they are easily procured from most hardware stores and are very economical, generally around 25.00\$ US or less, or the soil can be saturated with water until you see puddling on the surface of the soil.

N. <u>MIX/ APPLICATOR TOOLS</u>; you will need a pale or sprayer to mix and apply OSE II to the PFAS contaminated soil, you will need containers that hold a liter or more of liquid.



O. <u>LOG BOOK:</u> A log book to keep track of what is done each day and any changes observed.

### II. CALCULATIONS/ PROPORTIONS:





The soil will be spread to a depth of 10.2 cm The container is approximately, 60.96 cm long X 45.72 cm wide, by 15.24 to 20.32 cm deep, the 20.32 cm depth is preferred 1.5 kg of soil can contain up to 10% by volume of contaminant.

2. You will need to use 16.0 ml of OSE II from the 8-ounce bottle of OSE II, the 16 ml is required for this test.

3. The water to OSE II dilution ratio required, is 25 parts water to 1 part OSE II. 25 times 16 ml equals 375 ml of water that you will be required to mix with 16 ml of OSE II. The total liquid volume is 391ml.

4. You will need at least 2 liters of water on standby, to maintain the required moisture level. For each day you do not resupply the fresh water you will need 2 liters of water being aerated for when additional water is required, it is preferred you collect fresh water daily.

REMEMBER: The soil must be kept at a minimum of 60% moisture level throughout the entire test. If the soil is allowed to dry out during this time, you will not achieve the results as the necessary microbes will die off. If you do not have a meter to measure the moisture level, then make sure you can see water slightly pudding on the soils surface.

NOTE: If you start your demonstration/ test using fresh water then mix OSE II with fresh water and use the same fresh water source to maintain the moisture level, if you use sea water or brackish water then use sea water or brackish water to maintain the moisture level.

**5.** There will be two applications of the OSE II and water mixture, day one will contain 90% of the OSE II and water and day 21 the second application will be applied which is 10% of the required OSE II and water required.

III. DOCUMENTATION & PROCEDURE: Take pictures or video of each step and keep a log/ record with date/ times of all actions carried out for the duration of the demonstration.

1. Get all the supplies, oil, water and containers required to perform the demonstration staged at a site that will allow easy access for the demonstration.

#### 2. Take the PFAS contaminated soil and cover the entire bottom of the plastic demonstration container to a depth of approximately 10.2 cm

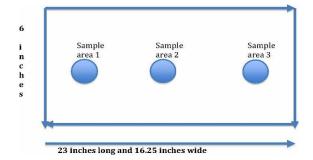
3. Take the tubing to be used for aeration from the bottom of the plastic container and make a four pronged aeration field, x^+ example below. You will need to poke approximately one hole each 5 cm apart for the length of the tubes. The tubes will need to be covered in soil and weighted on the ends.

#### See drawing below.



Note: Take Sample/s of the contaminated soil from the bottom of the soil. The sample size will be determined by the laboratory that is utilized, consult directly with the laboratory utilized for soil sample volume and the type of jars with Teflon sealed lids required.

- 4. 'THREE SAMPLE STANDARD': This method while *scientifically valid* and Where this method is selected or required, each of the three sample extractions are <u>not be mixed</u>, but each is kept separate and placed in separate jars and labeled as sample area 1, 2 and 3.
- Note: In this case, there would be three samples taken for each sampling date required as opposed to one composite sample for each date interval e.g. 7, 15, 30 and 45, and potentially 60 days if required. Soil sampling diagram below.



1. Day 1, the first application of OSE II and water, which is 90% of the total required volume. Mix 14.4 ml of OSE II with 360 liters of fresh water for a total volume of 374.4 ml and stir, this will require a container with the capacity of 400 ml.

2. Turn aerator on in the test vessel, air should start rising through the soil.

3. Apply the 374.4 ml of OSE II and water mixture evenly across the soil's surface until all the mixture has been emptied from the container. Spraying is the preferred method of application, but if you cannot access a sprayer, mixing it with water and pouring it over the spill site evenly can be performed.

4.24 hours after applying the OSE II and water mixture, measure the moisture content of the soil, with the moisture meter, or visually observe the soil and apply the necessary water to get the moisture above 60%; which should show liquid puddling on the soils surface, extra water is not a problem.

5.7 days after applying the OSE II and water mixture, extract the three soil samples, following the soil sampling diagram above, III. 4..

6.21 days after the initial application of OSE II, mix 1.6 ml of OSE II with 40 ml of water for a total volume of 41.6 ml, using the original mixing container with the capacity of 400 ml. This represents the second application of OSE II and water, the 10% by volume of the required OSE II and water.

7. Apply the 41.6 ml of OSE II and water mixture evenly across the soil's surface until all the mixture has been applied. Spraying is the preferred method of application, but if you cannot access a sprayer, mixing it with water and pouring it over the spill site evenly can be done.

NOTE: Make sure the correct jars are utilized the correct amount of soil is extracted from each sampling area and placed into separate jars, labeled, and then placed in the ice chest, which should have ice placed in it 1 hour before collecting soil samples.

#### IV. SAMPLING/ TESTING INTERVALS:

Note: USEPA method 1633 which covers soils and solids as well as ground water should be utilized. See link <a href="https://www.epa.gov/water-research/pfas-analytical-methods-development-and-sampling-research#Standard">https://www.epa.gov/water-research/pfas-analytical-methods-development-and-sampling-research#Standard</a>

Method 1633	EPA method for measuring 40 PFAS in wastewater, surface water, groundwater, soil, biosolids, sediment, landfill leachate, and fish tissue.
	EPA and the Department of Defense collaborated on the development of this method.

1.7 days after applying the OSE II and water mixture, extract the three soil samples, following the soil sampling diagram above, section III. step 4..

2.15 days after applying the OSE II and water mixture, extract the three soil samples, following the soil sampling diagram above, section III. step 4..

3.30 days after applying the OSE II and water mixture, extract the three soil samples, following the soil sampling diagram above, section III. step 4..

4.45 days after applying the OSE II and water mixture, extract the three soil samples, following the soil sampling diagram above, section III. step 4..

NOTE: if required, keep extracting soil samples until the required standard is met for Australia.

5.60 days after applying the OSE II and water mixture, extract the three soil samples, following the soil sampling diagram above, section III. step 4..

6.75 days after applying the OSE II and water mixture, extract the three soil samples, following the soil sampling diagram above, section III. step 4..

7.90 days after applying the OSE II and water mixture, extract the three soil samples, following the soil sampling diagram above, section III. step 4..

NOTE: Extract the samples from the same point/ area each time and place the three samples in a jar, mix the samples, seal the lid and label the jar. If using 3 separate sample standard, put samples in separate jars and label each of the samples area 1, 2 and 3 for Day 7, 15, 30, 45 ect respectively.

#### V. MAINTAINCE:

1. Check the moisture level each day with the moisture meter; write down in a log the moisture level. If the moisture level is below 60% add water until the moisture level is above 50% and you can see the water puddling on the surface of the soil, and note how much water was added.

2. Turn or till the soil three times a week with the small spatula, and make a note of each time the soil is turned in your written log.

3. Each time the soil is to be tilled scrape any oil on the sides of the container onto the soil then till/stir.

4. Video the set up of the equipment, and OSE II, and the mixing and application of OSE II, then take pictures or a short video each day until the test day extractions are completed.

VI. Observations

Write down initial observations as well as smells. Each time a noticeable change/ observations or action takes place note it with date, time and what was observed exactly. Also take pictures as often as possible or video.

ENG. Chace Smith Steven Pedigo



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Sample ID	information	Depth	Date sampled		Suffe	E		<b>U</b> 1		Tests Re	quired			Comments
1				Type of sample	PFAS extended s (28 analytes)	Total FI by fusion	N IS	POCENT PRICE	spc ciation					Provide as much information about sample as you ca
	RO6-1		11.09.24	wet	V	V	V	_	V					
-	R06 - 2		11.09.24	west .	v	V	V	1	1					
3	R06-3		11.09.24	SOIL	V	V	V	~	-					
	by (Company): Hibbs	11		Received by (Cor		ELS	, М	elb			112		ab Use Only	~
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Envirolab Services Pty Ltd ABN 37 112 535 645 - 002 25 Research Drive Croydon South VIC 3136 ph +61 3 9763 2500 melbourne@envirolab.com.au www.envirolab.com.au

## Certificate of Analysis MFI 0195

Client Details						
Client	Hibbs & Associates Pty Ltd					
Contact	George Dervusoski					
Address	PO Box 4266, HOMEBUSH, NSW, 2140					
Sample Details						
Your Reference	S13167					
Number of Samples	3 Soil					
Date Samples Received	11/09/2024					
Date Instructions Received	11/09/2024					
Analysis Details						
	m the client. Results relate specifically to the samples as received. asis for soils and on an as received basis for other matrices.					
Date Results Requested by	18/09/2024					
Date of Issue	18/09/2024					
NATA Accreditation Number 2901. This document shall not be reproduced except in full. Accredited for compliance with ISO/ IEC 17025. Tests not covered by NATA are denoted with *.						
Authorisation Details						
Results Approved By	Azrin Akram, Senior Chemist Chaminda Gunasekara, Inorganics Supervisor Tianna Milburn, Senior Chemist					
Laboratory Manager Pamela Adams						

## Samples in this Report

Envirolab I D	Sample I D	Matrix	Date Sampled	Date Received
MFI0195-01	R06-1	Soil	11/09/2024	11/09/2024
MFI0195-02	R06-2	Soil	11/09/2024	11/09/2024
MFI0195-03	R06-3	Soil	11/09/2024	11/09/2024

### Sample Comments

General Comment

Sample(s) received in sample containers that don't conform to recommended containers. Hence the analytical data may be affected.

		. ,			
Envirolab I D	Units	PQL	MFI0195-01	MFI0195-02	MFI0195-03
Your Reference			R06-1	R06-2	R06-3
Date Sampled			11/09/2024	11/09/2024	11/09/2024
Total Fluoride	mg/kg	50	360	230	400
Fluoride	mg/kg	0.50	1.2	1.2	1.2

### Inorganics - General Chemical Parameters (Soil)

Your Reference: Revision: R-00

### I norganics - Moisture (Soil)

Moisture	%	0.10	25	24	29
Date Sampled			11/09/2024	11/09/2024	11/09/2024
Your Reference			R06-1	R06-2	R06-3
Envirolab I D	Units	PQL	MFI0195-01	MFI0195-02	MFI0195-03

### PFAS Extended List (Soil)

Envirolab I D	Units	PQL	MFI0195-01 R06-1	MFI0195-02 R06-2	MFI0195-03 R06-3
Your Reference Date Sampled			11/09/2024	11/09/2024	11/09/2024
Perfluorobutanesulfonic acid (PFBS)	µg/kg	0.10	0.40	0.37	0.49
Perfluoropentanesulfonic acid (PFPeS)	µg/kg	0.10	0.47	0.42	0.57
Perfluorohexanesulfonic acid (PFHxS)	µg/kg	0.10	6.2	5.3	7.1
Perfluoroheptanesulfonic acid (PFHpS)	µg/kg	0.10	1.1	0.92	1.4
Perfluorooctanesulfonic acid (PFOS)	µg/kg	0.10	540	430	660
Perfluorodecanesulfonic acid (PFDS)	µg/kg	0.20	43	33	49
Perfluorobutanoic acid (PFBA)	µg/kg	0.20	<0.50 [3]	<0.50 [3]	<0.50 [3]
Perfluoropentanoic acid (PFPeA)	µg/kg	0.20	0.33	0.29	0.45
Perfluorohexanoic acid (PFHxA)	µg/kg	0.10	0.85	0.69	1.0
Perfluoroheptanoic acid (PFHpA)		0.10	0.16	0.14	0.20
Perfluorooctanoic acid (PFOA)	μg/kg μg/kg	0.10	0.93	0.76	1.2
Perfluorononanoic acid (PFNA)		0.10	<0.10	<0.10	<0.10
	µg/kg				
Perfluorodecanoic acid (PFDA)	µg/kg	0.50	<0.50	<0.50	<0.50
Perfluoroundecanoic acid (PFUnDA)	µg/kg	0.50	<0.50	<0.50	<0.50
Perfluorododecanoic acid (PFDoDA)	µg/kg	0.50	<0.50	<0.50	<0.50
Perfluorotridecanoic acid (PFTrDA)	µg/kg	0.50	<0.50	<0.50	<0.50
Perfluorotetradecanoic acid (PFTeDA)	µg/kg	5.0	<5.0	<5.0	<5.0
4:2 Fluorotelomer sulfonic acid (4:2 FTS)	µg/kg	0.10	<0.10	<0.10	<0.10
6:2 Fluorotelomer sulfonic acid (6:2 FTS)	µg/kg	0.10	<0.10	<0.10	<0.10
8:2 Fluorotelomer sulfonic acid (8:2 FTS)	µg/kg	0.20	<0.20	<0.20	<0.20
10:2 Fluorotelomer sulfonic acid (10:2 FTS)	µg/kg	0.20	<0.20	<0.20	<0.20
Perfluorooctane sulfonamide (FOSA)	µg/kg	1.0	4.6	3.4	5.5
-Methyl perfluorooctane sulfonamide MeFOSA)	µg/kg	1.0	<1.0	<1.0	<1.0
N-Ethyl perfluorooctane sulfonamide (EtFOSA)	µg/kg	1.0	<1.0	<1.0	<1.0
N-Methyl perfluorooctane sulfonamidoethanol	µg/kg	1.0	<1.0	<1.0	<1.0
N-Ethyl perfluorooctane sulfonamidoethanol	µg/kg	5.0	<5.0	<5.0	<5.0
N-Methyl perfluorooctane sulfonamidoacetic acid	µg/kg	0.20	<0.20	<0.20	<0.20
N-Ethyl perfluorooctane sulfonamidoacetic acid	µg/kg	0.20	<0.20	<0.20	<0.20
	%				
	%				
Total +ve PFHxS+PFOS	µg/kg	0.10	550	440	670
Total +ve PFOA+PFOS	µg/kg	0.10	540	430	660
Total +ve PFAS	µg/kg	0.10	600	480	730
	%				
	%				
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	%				

Your Reference: S13167 Revision: R-00 Certificat

S13167 Certificate of Analysis Generated: 18/09/2024 16:18

### PFAS Extended List (Soil)

Envirolab I D	Units	PQL	MFI0195-01	MFI0195-02	MFI0195-03		
Your Reference			R06-1	R06-2	R06-3		
Date Sampled			11/09/2024	11/09/2024	11/09/2024		
	%						
	%						
	%						
	%						
	%			Extr	Extraction Internal Standard 13C2 PFUnDA		
	%				Extraction Internal Standard 13C2 PFDoDA		
	%				Extraction Internal Standard 13C2 PFTeDA		
	%				Extraction Internal Standard 15C2 FFTEDA		
	%				Extraction Internal Standau	rd 13C2 4:2	
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	%						

## Subcontracted Micro - Certificate: 215273 - Analysed By ALS (Scoresby) (Soil)

Envirolab I D	Units	PQL	MFI0195-01	MFI0195-02	MFI0195-03
Your Reference			R06-1	R06-2	R06-3
Date Sampled			11/09/2024	11/09/2024	11/09/2024
Aerobic Plate Count - 22C	cfu/g dry wt		9200000 [6]	9200000 [6]	1.9E+07 [6]

### **Result Comments**

l dentifier	Description	
[2]	For PFAS Extracted Internal Standards denoted with ## or being outside the 50-150% acceptance range, the respective target analyte results may be unaffected, in other circumstances the PQL has been raised to accommodate the outlier(s).	
[3]	PQL(s) has/have been raised due to matrix interference.	
[6]	The sub-contracting laboratory did not provide analysis and/or preparation and/or sample receipt dates. The date(s) the sample(s) was/were received at the sub-contracting laboratory has/have been used to assess holding time(s).	

## Method Summary

Method I D	Methodology Summary
INORG-008	Moisture content determined by heating at 105+/-5 °C for a minimum of 12 hours.
INORG-026	Fluoride determined by ion selective electrode (ISE) based on APHA latest edition, 4500-F-C. Solids are reported from a 1:5 water extract unless otherwise specified.
INORG-138	Total Fluoride by Combustion Ion Chromatography
ORG-029	Soil/solid and sorbent samples are extracted with basified Methanol. Waters and soil/sorbent extracts are directly injected and/or concentrated/extracted using SPE. TCLP/ASLP leachates are centrifuged, the supernatant is then analysed (including amendment with solvent) - as per the option in AS4439.3. Analysis is undertaken with LC-MSMS. PFAS results include the sum of branched and linear isomers where applicable. Please note that PFAS results are corrected for Extracted Internal Standards (QSM 5.4 Table B-15 terminology), which are mass labelled analytes added prior to sample preparation to assess matrix effects and verify processing of the sample. PFAS analytes without a commercially available mass labelled analogue are corrected vs a closely eluting mass labelled PFAS compound. Surrogates are also reported, in this context they are mass labelled PFAS compounds added prior to extraction but are used as monitoring compounds only (not used for result correction). Envicarb (or similar) is used discretionally to remove interfering matrix components. Please contact the laboratory if estimates of Measurement Uncertainty are required as per WA DER.
SUB-004	Subcontracted to ALS Water - Accreditation number 992

### **Result Definitions**

Identifier	Description
NR	Not reported
NEPM	National Environment Protection Measure
NS	Not specified
LCS	Laboratory Control Sample
RPD	Relative Percent Difference
>	Greater than
<	Less than
PQL	Practical Quantitation Limit
INS	Insufficient sample for this test
NA	Test not required
NT	Not tested
DOL	Samples rejected due to particulate overload (air filters only)
RFD	Samples rejected due to filter damage (air filters only)
RUD	Samples rejected due to uneven deposition (air filters only)
##	Indicates a laboratory acceptance criteria outlier, for further details, see Result Comments and/or QC Comments

#### **Quality Control Definitions**

#### Blank

This is the component of the analytical signal which is not derived from the sample but from reagents, glassware etc, and is determined by processing solvents and reagents in exactly the same manner as for samples.

#### Surrogate Spike

Surrogates are known additions to each sample, blank, matrix spike and LCS in a batch, of compounds which are similar to the analyte of interest, however are not expected to be found in real samples.

#### LCS (Laboratory Control Sample)

This comprises either a standard reference material or a control matrix (such as a blank sand or water) fortified with analytes representative of the analyte class. It is simply a check sample.

#### Matrix Spike

A portion of the sample is spiked with a known concentration of target analyte. The purpose of the matrix spike is to monitor the performance of the analytical method used and to determine whether matrix interferences exist.

#### Duplicate

This is the complete duplicate analysis of a sample from the process batch. The sample selected should be one where the analyte concentration is easily measurable.

#### Laboratory Acceptance Criteria

Duplicate sample and matrix spike recoveries may not be reported on smaller jobs, however, were analysed at a frequency to meet or exceed NEPM requirements. All samples are tested in batches of 20. The duplicate sample RPD and matrix spike recoveries for the batch were within the laboratory acceptance criteria. Filters, swabs, wipes, tubes and badges will not have duplicate data as the whole sample is generally extracted during sample extraction. Spikes for Physical and Aggregate Tests are not applicable. For VOCs in water samples, three vials are required for duplicate or spike analysis.

General Acceptance Criteria (GAC) - Analyte specific criteria applies for some analytes and is reflected in QC recovery tables.

Duplicates: >10xPQL - RPD acceptance criteria will vary depending on the analytes and the analytical techniques but is typically in the range 20%-50% - see ELN-P05 QAQC tables for details (available on request); <10xPQL - RPD are higher as the results approach PQL and the estimated measurement uncertainty will statistically increase. Matrix Spikes, LCS and Surrogate recoveries: Generally 70-130% for inorganics/metals; 60-140% for organics (+/-50% surrogates) and 10-140% for labile SVOCs (including labile surrogates), ultra trace organics and speciated phenols is acceptable.

In circumstances where no duplicate and/or sample spike has been reported at 1 in 10 and/or 1 in 20 samples respectively, the sample volume submitted was typically insufficient in order to satisfy laboratory QA/QC protocols.

#### **Miscellaneous Information**

When samples are received where certain analytes are outside of recommended technical holding times (THTs), the analysis has proceeded. Where analytes are on the verge of breaching THTs, every effort will be made to analyse within the THT or as soon as practicable.

Where sampling dates are not provided, Envirolab are not in a position to comment on the validity of the analysis where recommended technical holding times may have been breached. We have taken the sampling date as being the date received at the laboratory.

Two significant figures are reported for the majority of tests and with a high degree of confidence, for results <10\*PQL, the second significant figure may be in doubt i.e. has a relatively high degree of uncertainty and is provided for information only.

Measurement Uncertainty estimates are available for most tests upon request.

Analysis of aqueous samples typically involves the extraction/digestion and/or analysis of the liquid phase only (i.e. NOT any settled sediment phase but inclusive of suspended particles if present), unless stipulated on the Envirolab COC or by correspondence. Notable exceptions include certain Physical Tests (pH/EC/BOD/COD/Apparent Colour etc.), Solids testing, Total Recoverable metals and PFAS where sediment/solids are included by default.

Urine Analysis - The BEI values listed are taken from the 2022 edition of

Air volume measurements are not covered by Envirolab's NATA accreditation.

## Data Quality Assessment Summary MFI0195

### **Client Details**

Client Your Reference Date I ssued Hibbs & Associates Pty Ltd S13167 18/09/2024

### **Recommended Holding Time Compliance**

No recommended holding time exceedances

## **Quality Control and QC Frequency**

QC Туре	Compliant	Details
Blank	Yes	No Outliers
LCS	Yes	No Outliers
Duplicates	No	Duplicate Outliers Exist - See detailed list below
Matrix Spike	No	Matrix Spike Outliers Exist - See detailed list below
Surrogates / Extracted Internal Standards	No	Surrogates / Extracted ISTD Outliers Exist - See detailed list below
QC Frequency	Yes	No Outliers

Surrogates/Extracted Internal Standards, Duplicates and/or Matrix Spikes are not always relevant/applicable to certain analyses and matrices. Therefore, said QC measures are deemed compliant in these situations by default. See Laboratory Acceptance Criteria for more information

## Data Quality Assessment Summary MFI0195

## **Recommended Holding Time Compliance**

Analysis	Sample Number(s)	Date Sampled	Date Extracted	Date Analysed	Compliant
Fluoride   Soil	1-3	11/09/2024	18/09/2024	18/09/2024	Yes
Total Fluoride by CIC   Soil	1-3	11/09/2024	17/09/2024	17/09/2024	Yes
Moisture   Soil	1-3	11/09/2024	13/09/2024	16/09/2024	Yes
PFAS EXT-ISTD   Soil	1-3	11/09/2024	13/09/2024	14/09/2024	Yes
PFAS-Extended   Soil	1-3	11/09/2024	13/09/2024	14/09/2024	Yes
APC-22C   Soil	1	11/09/2024	12/09/2024	16/09/2024	Yes
	2-3	11/09/2024	16/09/2024	16/09/2024	Yes

## **Outliers: Duplicates**

INORG-138   Inorganics - General Chemical Parameters (Soil)   Batch BFI 2902					
Sample I D	Duplicate I D	Analyte	% Limits	RPD	
MFI0195-01	DUP2	Total Fluoride	30.00	41.7[4]	

### **Outliers: Matrix Spike**

### ORG-029| PFAS Extended List (Soil)| Batch BFI 2353

Sample I D	Analyte	% Limits	% Recovery
MFI0195-02	Perfluorodecanesulfonic acid (PFDS)	60 - 140	##[1]
MFI0195-02	Perfluorooctanesulfonic acid (PFOS)	60 - 140	##[1]

### Outliers: Surrogate / Extracted Internal Standards

### ORG-029| PFAS Extended List (Matrix)| Batch BFI 2353

Sample I D	Analyte	% Limits	% Recovery
MFI0195-01	Extraction Internal Standard 13C5 PFNA	50 - 150	## [2]
MFI0195-03	Extraction Internal Standard 13C5 PFNA	50 - 150	## [2]

## **Quality Control MFI0195**

## INORG-138 | Inorganics - General Chemical Parameters (Soil) | Batch BFI 2902

Analyte	Units	PQL	Blank	DUP1 BFI2902-DUP1# Samp   QC   RPD %	DUP2 MFI0195-01 Samp   QC   RPD %	LCS %	Spike % BFI2902-MS1#
Total Fluoride	mg/kg	50	<50	398 339 16.1	358 235 41.7 [4]	104	98.7

# The QC reported was not specifically part of this workorder but formed part of the QC process batch.

### INORG-026 | Inorganics - General Chemical Parameters (Soil) | Batch BFI 3158

Analyte	Units	PQL	Blank	<b>DUP1</b> MFI0195-01 Samp   QC   RPD %	LCS %	Spike % MFI0195-02
Fluoride	mg/kg	0.50	<0.50	1.25 1.22 [NA]	120	113

### INORG-008 | Inorganics - Moisture (Soil) | Batch BFI 2350

				DUP1	DUP2	LCS %
Analyte	Units	PQL	Blank	MFI0195-01	BFI2350-DUP2#	
				Samp   QC   RPD %	Samp   QC   RPD %	
Moisture	%	0.1		24.7 25.3 2.48	0.110 0.320 [NA]	[NA]

# The QC reported was not specifically part of this workorder but formed part of the QC process batch.

### ORG-029| PFAS Extended List (Soil) | Batch BFI 2353

erfluorobutanesulfonic acid (PFBS)				0 1001000.00	0 1001000.00		MFI0195-02
		0.10	-0.10	Samp   QC   RPD %	Samp   QC   RPD %	90.2	97.7
erfluoropentanesulfonic acid (PFPeS)	µg/kg	0.10	<0.10	0.402 0.479 [NA]	0.402 0.426 [NA]		
	µg/kg	0.10	<0.10	0.466 0.550 16.5	0.466 0.532 [NA]	93.2	109
erfluorohexanesulfonic acid (PFHxS)	µg/kg	0.10	<0.10	6.23 7.35 16.5	6.23 7.31 16.0	88.2	96.4
erfluoroheptanesulfonic acid (PFHpS)	µg/kg	0.10	<0.10	1.11 1.33 17.9	1.11 1.40 22.8	87.4	99.2
erfluorooctanesulfonic acid (PFOS)	µg/kg	0.10	<0.10	545 613 11.8	545 669 20.4	87.5	##[1]
erfluorodecanesulfonic acid (PFDS)	µg/kg	0.20	<0.20	43.3 45.7 5.31	43.3 48.8 11.8	92.5	##[1]
erfluorobutanoic acid (PFBA)	µg/kg	0.20	<0.20	<0.50 <0.50 [NA][3]	<0.50 <0.50 [NA][3]	95.3	96.4
erfluoropentanoic acid (PFPeA)	µg/kg	0.20	<0.20	0.327 0.367 [NA]	0.327 0.433 [NA]	93.7	100
erfluorohexanoic acid (PFHxA)	µg/kg	0.10	<0.10	0.851 0.999 16.0	0.851 0.916 7.40	92.3	95.2
erfluoroheptanoic acid (PFHpA)	µg/kg	0.10	<0.10	0.160 0.196 [NA]	0.160 0.170 [NA]	98.2	97.9
erfluorooctanoic acid (PFOA)	µg/kg	0.10	<0.10	0.930 1.10 16.7	0.930 1.07 14.4	94.8	101
erfluorononanoic acid (PFNA)	µg/kg	0.10	<0.10	<0.10 <0.10 [NA]	<0.10 <0.10 [NA]	90.7	93.0
erfluorodecanoic acid (PFDA)	µg/kg	0.50	<0.50	<0.50 <0.50 [NA]	<0.50 <0.50 [NA]	102	112
erfluoroundecanoic acid (PFUnDA)	µg/kg	0.50	<0.50	<0.50 <0.50 [NA]	<0.50 <0.50 [NA]	103	110
erfluorododecanoic acid (PFDoDA)	µg/kg	0.50	<0.50	<0.50 <0.50 [NA]	<0.50 <0.50 [NA]	93.1	97.9
erfluorotridecanoic acid (PFTrDA)	µg/kg	0.50	<0.50	<0.50 <0.50 [NA]	<0.50 <0.50 [NA]	101	120
erfluorotetradecanoic acid (PFTeDA)	µg/kg	5.0	<5.0	<5.0 <5.0 [NA]	<5.0 <5.0 [NA]	96.9	100
:2 Fluorotelomer sulfonic acid (4:2 FTS)	µg/kg	0.10	<0.10	<0.10 <0.10 [NA]	<0.10 <0.10 [NA]	109	108
:2 Fluorotelomer sulfonic acid (6:2 FTS)	µg/kg	0.10	<0.10	<0.10 <0.10 [NA]	<0.10 <0.10 [NA]	99.6	120
:2 Fluorotelomer sulfonic acid (8:2 FTS)	µg/kg	0.20	<0.20	<0.20 <0.20 [NA]	<0.20 <0.20 [NA]	108	107
0:2 Fluorotelomer sulfonic acid (10:2 FTS)	µg/kg	0.20	<0.20	<0.20 <0.20 [NA]	<0.20 <0.20 [NA]	106	138
erfluorooctane sulfonamide (FOSA)	µg/kg	1.0	<1.0	4.63 5.18 [NA]	4.63 5.37 14.8	89.5	99.4
-Methyl perfluorooctane sulfonamide MeFOSA)	µg/kg	1.0	<1.0	<1.0 <1.0 [NA]	<1.0 <1.0 [NA]	98.5	102
-Ethyl perfluorooctane sulfonamide EtFOSA)	µg/kg	1.0	<1.0	<1.0 <1.0 [NA]	<1.0 <1.0 [NA]	89.1	91.0
-Methyl perfluorooctane sulfonamidoethanol	µg/kg	1.0	<1.0	<1.0 <1.0 [NA]	<1.0 <1.0 [NA]	103	103
-Ethyl perfluorooctane sulfonamidoethanol	µg/kg	5.0	<5.0	<5.0 <5.0 [NA]	<5.0 <5.0 [NA]	100	107
-Methyl perfluorooctane sulfonamidoacetic cid	µg/kg	0.20	<0.20	<0.20 <0.20 [NA]	<0.20 <0.20 [NA]	97.0	95.6
-Ethyl perfluorooctane sulfonamidoacetic cid	µg/kg	0.20	<0.20	<0.20 <0.20 [NA]	<0.20 <0.20 [NA]	90.1	97.3

Your Reference: Revision: R-00 S13167 Certificate of Analysis Generated: 18/09/2024 16:18

# Quality Control MFI0195

## ORG-029| PFAS Extended List (Soil) | Batch BFI 2353

Analyte	Units	PQL	Blank	DUP1 MFI0195-01 Samp   QC   RPD %	DUP2 MFI0195-01 Samp   QC   RPD %	LCS %	<b>Spike %</b> MFI0195-02
				Extraction Intern	al Standard 13C3 PFPeA	%	94.
				E)	xtraction Internal Standard 13C2 PFHx.		%
					Extraction Internal Stand	ard 13C4 PFHpA Internal Standard 13C4	%
					Extraction		Standard 13C5 PFNA
						Extr	action Internal Standard 13C
							Extraction Interna

## QC Comments

l dentifier	Description
[1]	Spike recovery is not applicable due to the relatively high analyte background in the sample (>3* spike level). However, the LCS recovery is within acceptance criteria.
[2]	For PFAS Extracted Internal Standards denoted with ## or being outside the 50-150% acceptance range, the respective target analyte results may be unaffected, in other circumstances the PQL has been raised to accommodate the outlier(s).
[3]	PQL(s) has/have been raised due to matrix interference.
[4]	Duplicate %RPD may be flagged as an outlier to routine laboratory acceptance, however, where one or both results are <10*PQL, the RPD acceptance criteria increases exponentially.