



REFERENCE MATERIAL PRODUCT INFORMATION SHEET

NMIA MX019: PFAS in Soil

Property values

PFAS *	Abbreviation	CAS No.	Mass fraction (µg/kg)	Coverage factor (k)
Perfluorobutanoic acid	PFBA	375-22-4	1.70 ± 0.30	2.04
Perfluoropentanoic acid	PFPeA	2706-90-3	4.36 ± 0.80	2.02
Perfluorohexanoic acid	PFHxA	307-24-4	17.1 ± 2.5	2.04
Perfluoroheptanoic acid	PFHpA	375-85-9	3.47 ± 0.45	2.03
Perfluorooctanoic acid	PFOA	335-67-1	73.1 ± 8.2	2.04
Perfluorononanoic acid	PFNA	375-95-1	0.253 ± 0.052	2.16
Perfluorobutanesulfonic acid	PFBS	375-73-5	2.18 ± 0.33	2.03
Perfluorohexanesulfonic acid	PFHxS	355-46-4	53.7 ± 7.2	2.02
Perfluorohexanesulfonic acid - Total**	PFHxS-T	355-46-4	61.2 ± 8.2	2.02
Perfluorooctanesulfonic acid	PFOS	1763-23-1	910 ± 140	2.02
Perfluorooctanesulfonic acid - Total**	PFOS-T	1763-23-1	950 ± 140	2.02
N-Methyl perfluorooctane sulfonamide	MeFOSA	31506-32-8	2.22 ± 0.37	2.02
N-Ethyl perfluorooctane sulfonamidoethanol	EtFOSE	1691-99-2	4.9 ± 1.2	2.04
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	6:2 FTS	27619-97-2	5.38 ± 0.65	2.02

*All analytes are linear isomers only unless otherwise specified. The reported uncertainty is expanded to provide a level of confidence of 95%.

**Sum of linear and branched isomers.

Expiry: 03 September 2023

Batch No.: 2018.01

When stored in accordance with the instructions given on this certificate.

Description: This reference material consists of minimum 20 g of soil with incurred PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFBS, PFHxS, PFOS, PFOSA and fortified PFOA, MeFOSA, EtFOSE and 6:2 FTS, provided in a 50 mL polypropylene screw-capped tube.

Intended use: The reference material is intended for method validation and quality control purposes such as trend monitoring of analytical measurements to check if the process is under control.

Instructions for use: The recommended minimum sample size for analysis is 1 g of soil on an as received basis. The material must be thoroughly mixed before sub-sampling. After every use of the material, the tube should be closed tightly and returned to storage at -18°C.

Storage: The material should be stored at -18°C in the closed container as issued.

Metrological traceability: Property values of the PFAS analytes were determined via interlaboratory comparison. Participants employed validated methods using a variety of extraction and sample preparation techniques. For all analytes quantification was done by liquid chromatography-mass spectrometry, therefore property values are traceable to determination by LC-MS.

The property and indicative values were the unweighted mean value of the means of accepted sets of data from participant laboratories, all using calibrators from Wellington Laboratories. At the time of characterisation of MX019, calibrators from Wellington Laboratories had traceability statements for PFAS analytes to specific crystalline lots only. Therefore the metrological traceability of the measurands to SI has not been established.

Production: The material was prepared in September 2018 using contaminated soil mixed with uncontaminated soil from a Sydney landscape supplier which was fortified with four PFAS analytes. Each soil was air dried and sieved prior to mixing in a V-mixer. The prepared samples were divided into 20 g portions using a Retsch PT 100 rotary sample divider. Three hundred units of MX019 were produced. The reference material was distributed sequentially in polypropylene screw-capped tubes and stored at -18°C.

Analytical method: MX019 was characterised by an interlaboratory comparison study consisting of 11 laboratories world-wide. Methods applied for characterisation involved different sample extractions and clean-up followed by quantification using liquid chromatography mass spectrometry.

Homogeneity: Homogeneity testing was carried out in NMIA and was conducted using 10 units of MX019 selected randomly from the batch and analysed in duplicate in accordance with ISO Guide 35 [1]. Approximately 1 g of soil was spiked with 50 µL of a mixed labelled internal standard in methanol. The samples were extracted overnight by tumbling in alkaline methanol (0.01 N potassium hydroxide), then centrifuged and an 8 mL portion was evaporated under nitrogen. The dried extract was reconstituted in 1 mL of mobile phase, spiked with 50 µL of a mixed labelled recovery standard in methanol then filtered.

Instrument analysis was performed using a Waters Acquity I Class UPLC coupled with a Waters Xevo TQS mass spectrometer operating in multiple reaction monitoring mode. A volume of 3 µL of the extract was injected onto a Waters BEH C18 column (2.1 mm X 50 mm, 1.8 µm) with mobile phases (2 mM ammonium acetate in water and 2 mM ammonium acetate in methanol). The instrument mass accuracy was calibrated annually during preventative maintenance. An eight point calibration curve was established for each analytical batch. A reagent blank was extracted and analysed with each batch. Quantification was based on the use of the labelled standards using relative retention factors from the multipoint calibration. The analysis used calibration, internal standards and recovery standards supplied by Wellington Laboratories, Canada. The homogeneity results were used to calculate the within-bottle and between-bottle variances. The uncertainty in the property value incorporates these variances.

Stability: The material should be stored at -18°C in the closed container as issued. The long term stability at -18°C of the property values was assessed in August 2020, with ongoing monitoring performed to confirm stability over the life of the material. Stability of the material during transportation was also demonstrated by an isochronous accelerated stability study conducted at 40°C for up to four weeks.

The uncertainties in the property values incorporate a long-term storage stability component extrapolated to cover a period of certification of 12 months, and a transport stability component extrapolated to cover a period of 8 days at 40°C. [1].

Measurement uncertainty: Measurement uncertainties were estimated according to international standards [1,2] and National Measurement Institute standard operating procedures. All factors that could reasonably be expected to affect the measurement result were identified and the standard uncertainty of each evaluated. The standard uncertainties of the various components were combined as described in the Guide to the Expression of Uncertainty in Measurement [2]. The combined standard uncertainties were expanded to a level of confidence of 95% using coverage factors k calculated from the effective degrees of freedom obtained from the Welch-Satterthwaite equation.

Information Value Only

PFAS	Abbreviation	CAS No.	Mass fraction (µg/kg)	Coverage factor (k)
Perfluorooctane sulfonamide†	PFOSA	754-91-6	1.97 ± 0.63	2.06

†The above information value is reported for linear isomers only. This analyte exhibited statistically significant instability and deemed unsuitable as a property value. The reported uncertainty is expanded to provide a level of confidence of 95%.



Raluca Iavetz
Manager, Chemical Reference Values
01 September 2022
Report ID: MX019.2022.01

Accreditation No.198

References:

1. *ISO Guide 35:2017, Reference materials – Guidance for characterization and assessment of homogeneity and stability.*
2. *JCGM 100:2008 (GUM 1995 with minor corrections), Evaluation of measurement data – Guide to the expression of uncertainty in measurement, 1st edition (corrected 2010).*

This Certificate for NMIA MX019 supersedes any issued prior to 01 September 2022.

Legal notice: Terms and Conditions associated with the provision of this reference material can be found on the NMIA website.

END
