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EnviroNote News

UPDATE on Per- and Polyfluorinated Alkyl Substances (PFASs)

Government of Western Australia
Department of Environment Regulation

On 24 February 2016, the Government of Western Australia published its' Interim Guideline on the Assessment and Management of Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS) - Contaminated Sites Guidelines. This document includes guidance on

- Assessing and managing PFAS contamination
- Assessing risks to human health, the environment and environmental values
- The availability and derivation of generic assessment levels
- The remediation and management of PFAS impacted sites

Importantly from a laboratory analysis perspective it contains advice on methodologies and the

- use of polypropylene or HDPE sample containers with polypropylene lids;
- use of a mixed linear/branched standard for calibration purposes and the approach used in quantification;
- report analytical results representing the concentration of summed linear and branched isomers; and
- include the uncertainty in measurement in the laboratory reporting.

EPA United States Environmental Protection Agency — Emerging Contaminants – Perfluorooctane Sulfonate (PFOS) and Perfluorooctanoic Acid (PFOA)

Eurofins follows standard USEPA methodology for waters and soils and the recommended sample containers, preservation requirements and respective holding times are outlined below.

US EPA Method 537 ver 1.1 - Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC-MS/MS)

SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Samples must be collected in a 250-mL polypropylene bottle fitted with a polypropylene screw-cap. The preservation reagent, Trizma®, is added to each sample bottle as a solid prior to shipment to the field (or prior to sample collection).

FIELD REAGENT BLANKS (FRB)

A FRB must be handled along with each sample set. The sample set is composed of samples collected from the same sample site and at the same time. At the laboratory, fill the field blank sample bottle with reagent water and preservatives, seal, and ship to the sampling site along with the sample bottles. For each FRB shipped, an empty sample bottle (no preservatives) must also be shipped. At the sampling site, the sampler must open the shipped FRB and pour the preserved reagent water into the empty shipped sample bottle, seal and label this bottle as the FRB. The FRB is shipped back to the laboratory along with the samples and analysed to ensure that PFASs were not introduced into the sample during sample collection/handling.

SAMPLE SHIPMENT AND STORAGE – Samples must be chilled during shipment and must not exceed 10°C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10 °C when the samples are received at the laboratory. Samples stored in the lab must be held at or below 6 °C until extraction, but should not be frozen.

SAMPLE AND EXTRACT HOLDING TIMES – Results of the sample storage stability study indicated that all compounds listed in this method have adequate stability for 14 days when collected, preserved, shipped and stored as described above. Therefore, water samples should be extracted as soon as possible but must be extracted within 14 days. Extracts must be stored at room temperature and analysed within 28 days after extraction.

EPA-821-R-11-007 Draft Procedure for Analysis of Perfluorinated Carboxylic Acids and Sulfonic Acids in Sewage Sludge and Biosolids by HPLC/MS/MS Dec. 2011

SAMPLE COLLECTION, PRESERVATION, STORAGE, AND HOLDING TIMES

Sample collection – Collect samples in amber high density polyethylene (HDPE) containers with propylene caps/lids, following conventional sampling practices designed to obtain a sample that is representative of the material of interest. Lids and other materials containing PTFE must be avoided, due to possible leaching of fluorinated materials.

Collect a sample of sewage sludge or biosolids sufficient to yield at least 0.5 g of wet solids for analysis, plus enough sample to allow the determination of % solids determination (moisture) and to provide volume for QC samples. Larger samples are recommended to ensure that they are more representative of the bulk source of the material. Holding times – EPA has not yet conducted a formal holding time study and will conduct one after the procedure is finalised. Until that time, default holding times that begin at the time of sample collection are as follows:

- Begin sample extraction within 60 days of collection (to be validated).
- Analyse extracts within 30 days of extraction (to be validated).
- Store all samples and extracts at less than 4 ºC in HDPE containers.

ISOTOPE DILUTION LC-MS/MS

Labelled internal standard solution is spiked directly into samples prior to extraction. Labelled compounds are used to quantify unlabelled target compounds and perform recovery correction. Isotope dilution is used for calibration of each native compound for which an exact labelled analogue is available. Internal standard calibration is applied to the determination of the native compounds that do not have exact labelled analogues, and that are not being quantified by isotope dilution. Internal standard calibration is also used to quantify the labelled compounds themselves.

Results You Can Trust

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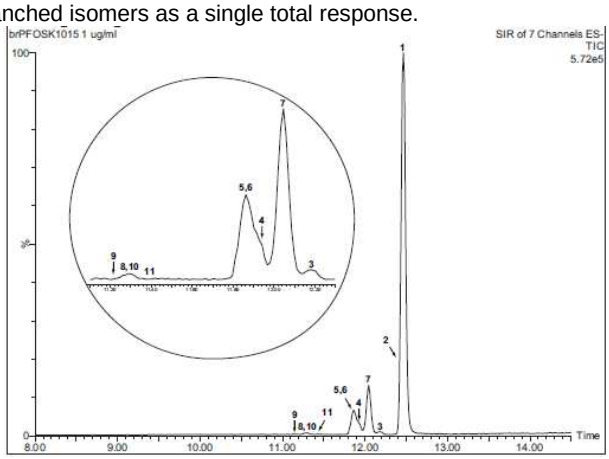
QUANTIFICATION OF LINEAR AND BRANCHED ISOMERS

Some PFASs consist of linear and branched isomers, depending on manufacturing processes viz electrochemical fluorination (ECF) or fluorotelomer process. Therefore, peak integration in samples ensures that the PFAS target peaks include the linear and branched isomers as a single total response.



NOTE: At the time of writing this note only PFOS was available as certified standard containing both linear and branched isomers. Eurofins uses br-PFOSK Potassium Perfluorooctanesulfonate Solution/Mixture of Linear and Branched Isomers that is compliant with NATA's Policy Circular 11 Policy on Metrological Traceability. All other PFASs are quantified using linear standards and where linear and branched isomers are present they are included and appropriately notated on the report.

A NIST reference material 8447 containing perfluorinated sulfonic acids with both linear and branched isomers were analysed by three laboratories using LC-MS/MS techniques. Eurofins was Lab 1 and as can be there was very good agreement between the three laboratories and with the supplied reference values and associated measurement uncertainties indicating that the methodologies are giving comparable results for this reference standard.

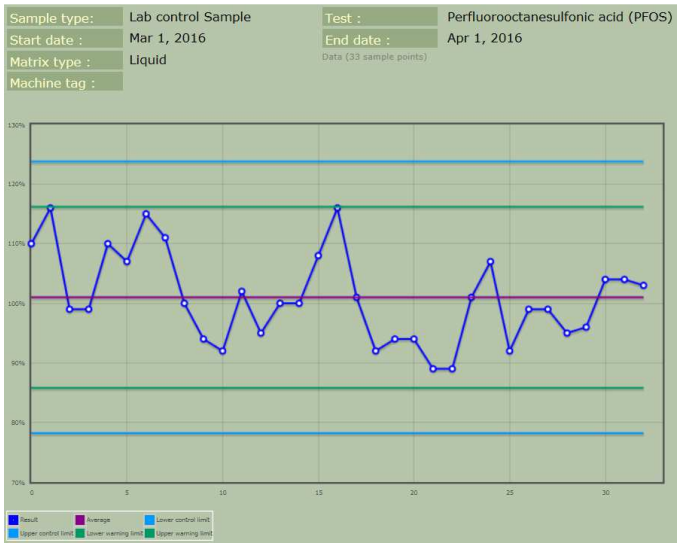
NIST – Reference Material laboratory comparison

Laboratory	PFBS	PFHxS	PFOS
NIST Sample	42.3 ± 2.3	55.2 ± 1.7	56.6 ± 2.5
Lab 1	41.95	54.66	57.04
Lab 2	43.7	57.4	59.3
Lab 3	33.77	51.89	56.17
%RPD range	1 to 22	1 to 6	1 to 4

MEASUREMENT UNCERTAINTY
(or the number after the ± sign!)

When reporting the result of a measurement of a physical quantity, it is obligatory that some quantitative indication of the quality of the result be given so that those who use it can assess its reliability. Without such an indication, measurement results cannot be compared, either among themselves or with reference values given in a specification or standard. It is therefore necessary that there be a readily implemented, easily understood, and generally accepted procedure for characterizing the quality of a result of a measurement, that is, for evaluating and expressing its uncertainty.

At Eurofins we use control charts as a mechanism of measuring and monitoring whether laboratory processes are in control viz are stable, with variation only coming from sources common to the process then no corrections or changes to process control parameters are needed or desired.



If the control chart indicates that the monitored process is not in control then the process is stopped immediately and procedures initiated to bring the system back in control. The control chart is a basic tool of quality control. Measurement uncertainty data is estimated from within-laboratory data on bias and precision has been calculated by using the procedures outlined in ASTM E2554-13 Standard Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques. The MU data calculated for PFOS over the last month shows an average recovery from laboratory control samples of 101% with a measurement uncertainty of 15.2% or 101 ± 15.2%. Measurement uncertainty values for other PFASs can be found by clicking on this link

Eurofins | mgt Experience

Eurofins | mgt's Brisbane centre-of-excellence have considerable experience in the analysis of PFASs in a range of matrices and obtained excellent results in a recent proficiency study conducted by the National Measurement Institute.

Please contact your local Analytical Service Manager or one of our Business Development Team listed below for further details.

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