P-015 PFAS Screening test: An essential tool for analytical decision making

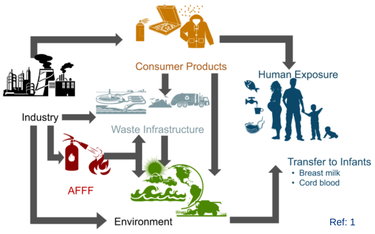
Rosamond Tshumah-Mutingwende*, Tomohiro Seki and Akihiko Tsujimura

Eurofins Nihon Kankyo K.K., NK Lab R&D Group, Japan

BACKGROUND

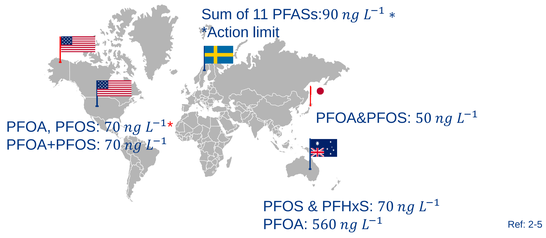
What are PFASs?

- Per-and polyfluoroalkyl substances
- Man-made compounds
- Consist of fluorine atoms attached to a carbon chain



Ref: 1

What has been done?



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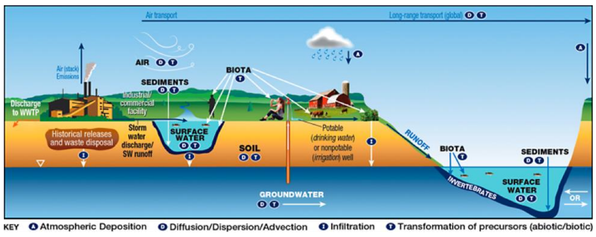
PFAS analytical methods

Method	Matrix	Quantitation method	Number of analytes
Japan Ministry of Environment	Surface water and groundwater	Internal standard / Isotope dilution	2
JIS K 045070-10	Industrial water / waste water	Internal Standard	2 (19)
USEPA 537.1	Drinking water	Internal standard	14
ISO 25101:2009	Drinking water		
USEPA 533	Drinking water	Isotope dilution	25
DIN 38407-42:2011-03	Drinking water/ wastewater/ sludge	Internal/External standard calibration	10
DIN 38414-14:2011-08	Soil / sludge	-	-
ASTM D7979	Groundwater, surface water, drinking water, waste water	External Calibration	21
ASTM D7968	Soil	External Calibration	21

Rationale



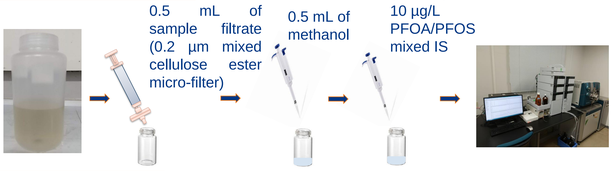
- PFASs are insusceptible to conventional waste water treatment techniques.
- Once in environment, PFAS precursors trend to undergo biotic and abiotic transformation into the fully fluorinated perfluoroalkyl compounds such as PFOA and PFOS
- This makes it difficult to estimate PFAS concentrations in contaminated sites, hence, in samples reaching the laboratory –thus, increasing the risk of cross-contamination.
- **Therefore, the objective of this study is to introduce a rapid screening method for PFAS analysis in water samples.**
- This will be useful in deciding sample volumes for solid phase extraction (SPE), dilution factors (DF) and preventing cross-contamination in laboratories by segregating samples based on their concentrations.

Ref: 6-7

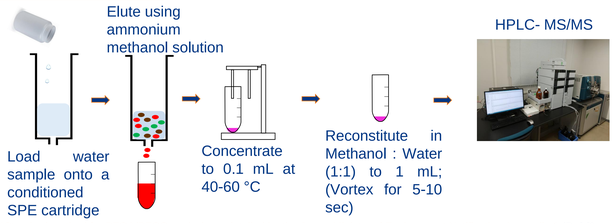
METHODOLOGY

A. Sample preparation

i. Screening method



ii. Standard method*



*Standard method for PFOA/PFOS analysis as detailed by the Japan Ministry of Environment Notification Appendix 1 (Ref:8).

B. Comparison of Screening and standard methods

Water samples were divided into 2 batches. The first batch was prepared according to the screening test pre-treatment procedure and the second batch using the standard method for PFOA/PFOS analysis as detailed in (Ref:8). Both batches were analyzed using the same HPLC gradient conditions. The screening and standard method sample concentrations were compared and the coefficient of determination was reported.

C. Optimization of LC gradient conditions

The gradient conditions were optimized in order to reduce the screening method analysis time. Several gradient conditions were tested and the optimum method which allowed us to obtain the best results (good peak shapes and surrogate recoveries) at a shorter analysis time was selected.

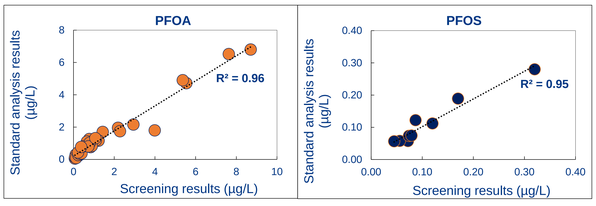
D. Comparison of the Internal and External standard calibration method

In most overseas laboratories, screening is often performed using the external standard calibration method. However, in this study, isotopes were added as internal standard in order to confirm the accuracy of our analysis. Analyte concentration were then calculated using the internal and external standard calibration methods and the results were compared.

RESULTS & DISCUSSIONS

B. Comparison of the screening and standard methods (Different sample pre-treatment/ similar HPLC gradient condition)

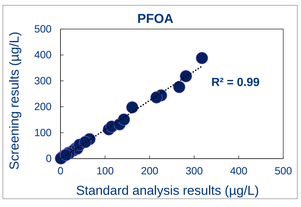
The observed high coefficients of determination (R^2) of 0.96 and 0.95 for PFOA and PFOS respectively; reveal the closeness of the screening and standard analysis results.



C. Optimization of LC gradient conditions

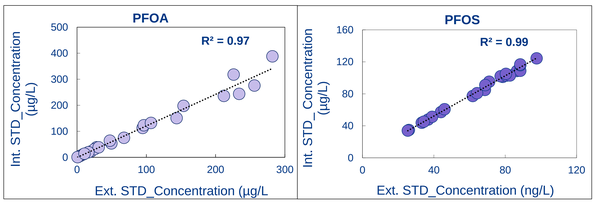
Keeping all other factors such as the mobile phase solutions, analytical, delay and guard column; Ion spray voltage and column temperature amongst others; HPLC conditions were optimized as follows:

Item	Old HPLC condition	Optimized HPLC conditions
Gradient / Analysis time (min)	15.10	10.01
Flowrate (mL/min)	0.2	0.4
Injection volume (µL)	1	2
Dwell time (msec)	150	100



- The observed high coefficient of determination (R^2) of 0.99 for PFOA reveal the closeness of the screening and standard analysis results.
- This also shows that the HPLC gradient conditions were successfully optimized.

D. Comparison of the Internal and External standard calibration method



- The observed high coefficients of determination (R^2) of 0.97 and 0.99 for PFOA and PFOS respectively; reveal the closeness of the results quantified using external and internal standard calibration methods.
- Thus, huge cost savings can be achieved by not using the Internal standard (mixed PFOA/PFOS surrogate solution) and utilizing the external calibration method.

CONCLUSIONS

- The screening process is an essential tool for analytical decision making.
- By implementing the screening process into our PFAS analysis routine, we could achieve the following:
  - Efficiently decide sample dilution factors
  - Decide sample volumes for SPE (for Standard analysis procedure)
  - Minimize the risk of contamination by segregating samples based on their concentrations
- Results of the comparison between the IS and external standard method, show a good correlation between these two methods. Therefore, the external standard calibration method was adopted as the screening method for the above purpose.
- Furthermore, huge cost savings are achieved by using the external standard calibration method since the internal standard (mixed PFOA/PFOS surrogate solution) addition step is eliminated.
- Also, the external standard calibration method enables us to analyze very low concentration samples by increasing the injection volume and observing the analyte peaks which would have been impossible to see at lower injection volumes.

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