



# PFAS Testing Now in Brisbane and TOP Assay Challenges and Developments

## PFAS Analysis Capabilities

Due to the high demand of analysis for PFAS (Per- and polyfluoroalkylated substances), ALS is now offering PFAS analysis in Brisbane specifically to provide fast Turn-around time results for the Queensland market.

NATA assessment is complete, and Accreditation is expected to be awarded in early December 2017. ALS Brisbane will analyse Soil, Water and Product matrices for Standard Level, Low Level and TOP Assay suites. Super Trace analysis and Biota will continue to be analysed in the ALS Sydney Laboratory.

This along with developments in the TOP Assay method to help ensure full oxidation of samples (outlined below) gives ALS the most comprehensive PFAS testing capabilities in Australia.

## METHOD AND LOR INFORMATION

### ALS METHOD CODE

EP231 - PFAS Short Suite (12 Analytes)  
EP231X - PFAS Full Suite (28 Analytes)  
EP231X (TOP) - PFAS TOP Assay (28 Analytes)  
EP231-LL - PFAS Low Level Short Suite  
EP231X-LL - PFAS Low Level Full Suite  
EP231X-LL (TOP) - PFAS Low Level TOP Assay  
EP231-ST - PFAS Super Trace Short Suite  
EP231X-ST - PFAS Super Trace Full Suite

### LIMIT OF DETECTION

#### WATER

EP231, EP231X, EP231X (TOP) - 0.01-0.1µg/L  
EP231-LL, EP231X-LL, EP231X-LL (TOP) - 0.002-0.01µg/L  
EP231-ST, EP231X-ST - 0.0003-0.002 µg/L

#### SOIL

EP231, EP231X, EP231X (TOP) - 0.0002-0.001 mg/kg

## Total Oxidisable Precursor (TOP) Assay

The Total Oxidisable Precursor (TOP) Assay is an oxidative sample pre-treatment method aimed at converting the PFAS within a sample into the stable perfluoroalkyl acids that can be quantified by conventional LCMSMS analytical techniques, thereby providing a greater understanding of the extent of PFAS within a sample. The oxidation is achieved by hydroxyl radicals, formed under heated alkaline conditions from prescribed amounts of potassium persulfate (KPS) and sodium hydroxide (NaOH) added to the sample prior to incubation.

## Challenges with current methodology

Extensive testing on a diverse range of foam products has demonstrated that under the standard conditions of the assay exhaustion of the oxidant (hydroxyl radicals) is possible unless samples are pre-diluted.

The primary source of exhaustion is not through conversion of PFAS, but from competition by other oxidisable material present in the formulations. This material is typically at higher proportions than the PFAS (i.e. solvents, non-fluorinated hydrocarbon surfactants and polysaccharide or protein thickeners as present in alcohol-resistant foams).

For environmental samples, exhaustion of the oxidant has been observed in matrices with elevated levels of organic carbon. Incomplete oxidation is evidenced by the persistence of fluorotelomers post assay (rather than the expected conversion to perfluorinated carboxylic acids) and the formation of fluorotelomer sulfonic acids from fluorotelomer sulfonamide derivatives (evidence of alkaline hydrolysis). Incomplete oxidation may significantly underestimate the post assay PFAS concentration where substantial concentrations of "precursor" compounds are present.

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# Total Oxidisable Precursor (TOP) Assay Developments

Previously, the only solution to avoiding incomplete oxidation was pre-dilution of the sample to reduce interference from the non-PFAS oxidisable material. However, dilution is only useful if the raised limits of reporting still permit practical data interpretation and assessment against relevant criteria.

ALS has recently completed a collaborative investigation and carried out extensive verification using increased dosage of oxidant for TOP Assay. Of critical importance in this modified technique is maintaining the stoichiometric ratio of oxidant (KPS) to hydroxide (NaOH). Also crucial is ensuring the pH is maintained within a range that promotes effective propagation of hydroxyl radicals and avoids potential chain shortening. Shortening of the perfluorinated alkyl chain below C4 will be unaccounted for, as this is the shortest chain length detected in the current analytical suite.

Waste water samples from a confidential project with high BOD concentrations (>3,000mg/L) had consistently demonstrated incomplete oxidation under standard dose conditions. This was evidenced by the persistence of fluorotelomer sulfonic acids following the oxidation step. As shown in Figure 1 (below), through increased oxidant dosage, effective conversion was achieved. The option of increasing dosage on difficult matrices provides benefits in terms of reduced frequency of repeats (and associated reporting delays) as well as preservation of limits of reporting. Ultimately a combined approach of dilution and increased dosage may be required for particularly challenging matrices in order to meet practical reporting requirements. In such instances, understanding the data objectives of the project is key.

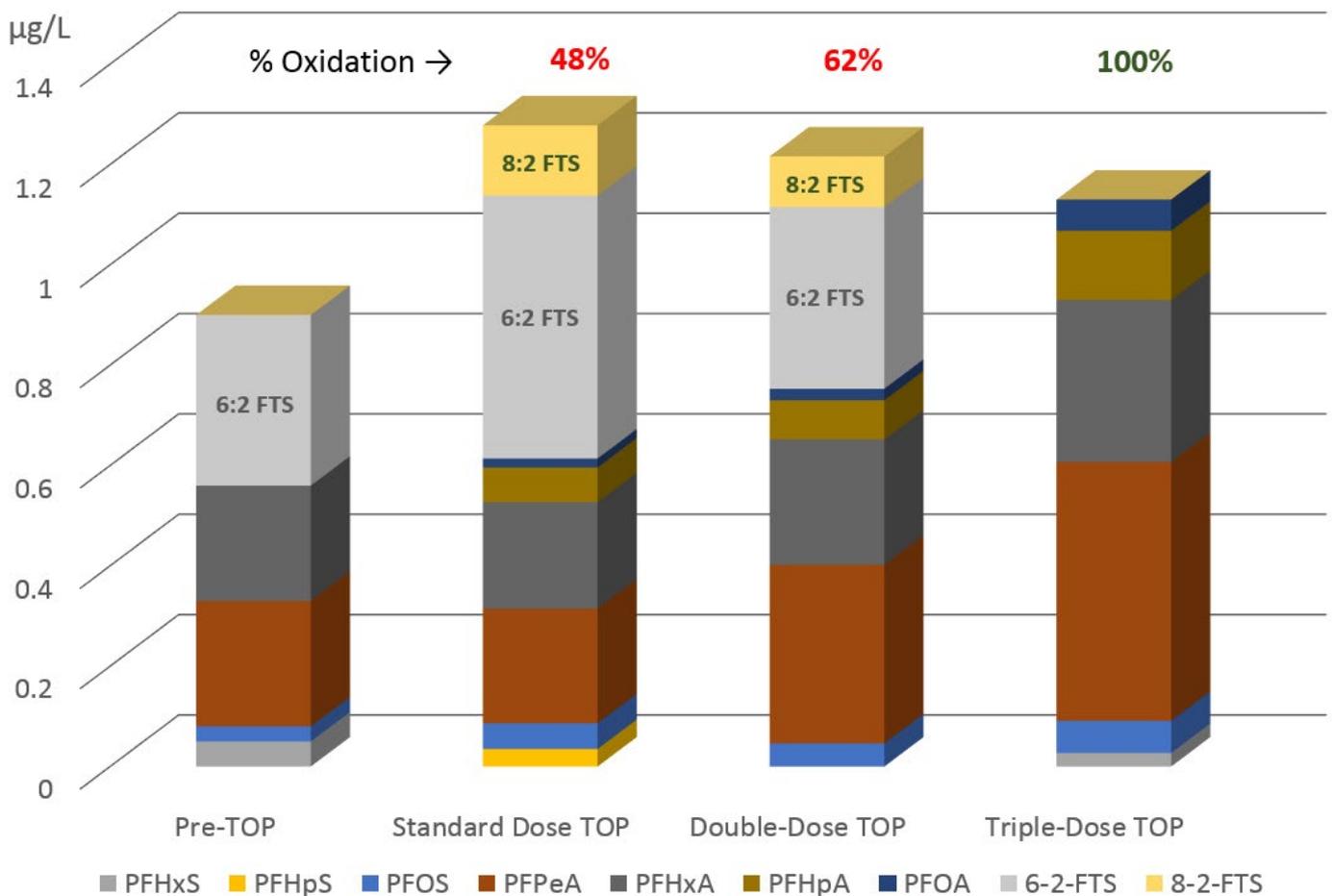


Figure 1. Demonstration of the effect of increased dose on completeness of oxidation. Waste water sample with BOD >3,000mg/L. Incomplete oxidation evidenced by persistence of fluorotelomer sulfonic acids. % Oxidation calculated as the proportion of Perfluoroalkyl acids (PFAA) to Sum of PFAS.

## ACKNOWLEDGEMENT

ALS would like to acknowledge Arcadis for technical input on method development and use of project data.

## REFERENCES

Erika F. Houtz and David L. Sedlak, Environ. Sci. Technol. 2012, 9342-9349

## OTHER ALS ENVIROMAILS ON PFAS

[EnviroMail 38](#) - PFOS and PFOA – June 2009 | [EnviroMail 67](#) - Testing-of-Extended-PFCs - March 2013 | [EnviroMail 86](#) - PFCs in Landfill leachate - February 2015 | [EnviroMail 94](#) - PFOS PFOA and why my results don't agree - Aug 2015 | [EnviroMail 106](#) - PFAS Naming Conventions, Extended Suites and Summation Reporting | [EnviroMail 110](#) - Identifying Hidden PFAS Chemicals in Environmental Samples and Firefighting Foams

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