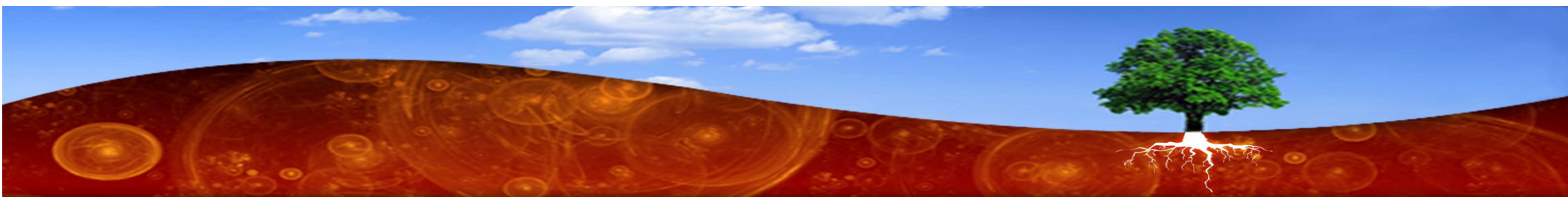


Detection of Perfluorooctanesulfonic Acid in Soil Samples

Brenna Arlyce Brown, PhD
Paracel Laboratories Ltd.
Calgary, AB

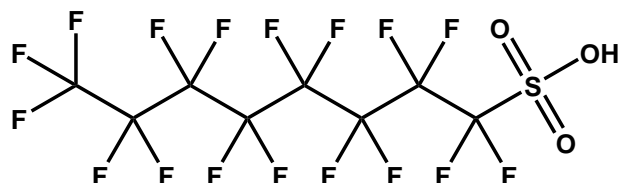


Outline

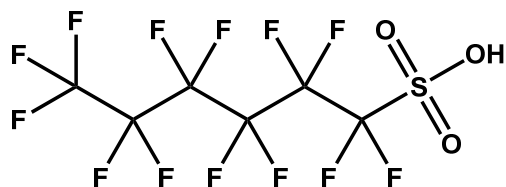
- Introduction to Perfluoronated compounds
 - Environmental Concerns
 - Health Concerns
- Introduction to DART-MS
- PFOS by DART-MS
- Conclusions



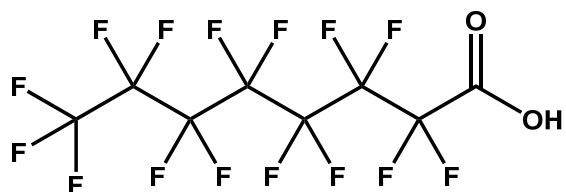
Perfluorinated Compounds



Perfluorooctanesulfonic acid – PFOS



Perfluorohexanesulfonic acid – PFHS



Perfluorooctanoic acid – PFOA

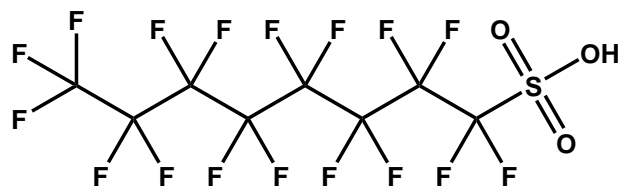


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Environ. Sci. Technol. 2001, 35, 1339-1342



Perfluoronated Compounds



C–F = 116 kcal/mol

C–H = 100 kcal/mol

- Hydrophobic and lipophobic
- Strength of C–F bond
- Fully oxidised, chemically stable
- Used in:
 - Surfactants
 - Insecticides
 - Textiles
 - Aqueous fire fighting foam



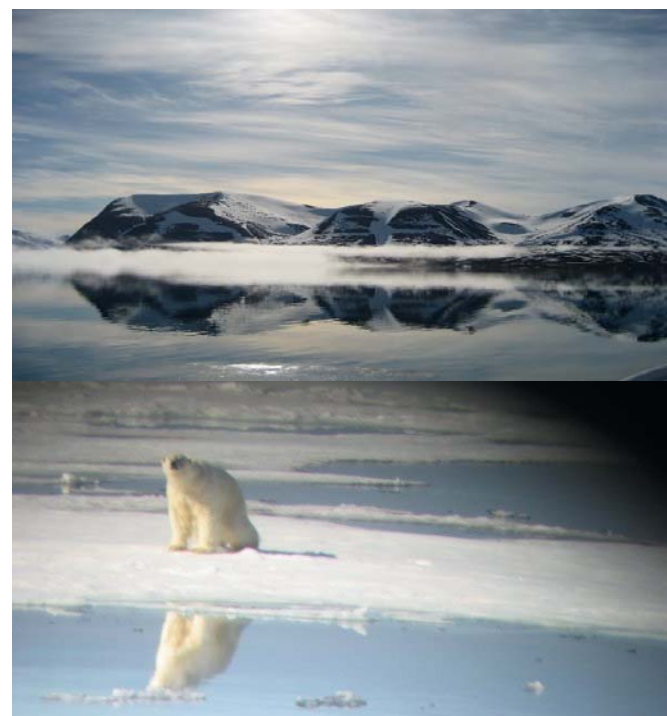
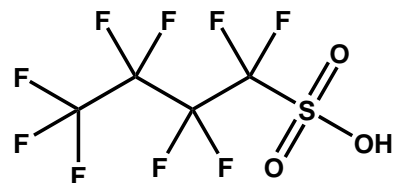
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Environment International, 2012, 39, 19-26



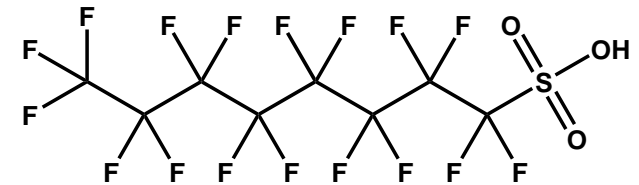
Perfluorinated Compounds – Persistent Organic Pollutants

- Produced since 1950s
- 2009 Stockholm Convention, PFOS listed as POP
- 1999 3M phase out PFOS from Scotchguard
- 2003 – 3M now uses perfluorobutanesulfonic acid

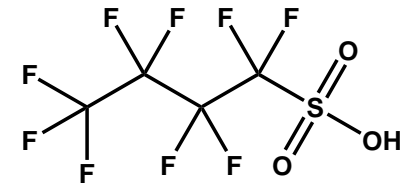


PFCs and Health Concerns

- Bioaccumulate in proteinaceous tissue
- Indicated carcinogenicity
- Impact:
 - Liver function
 - Cell organelles
 - Immune system
 - Neuroendocrine system



Half life in humans = 5.4 yrs

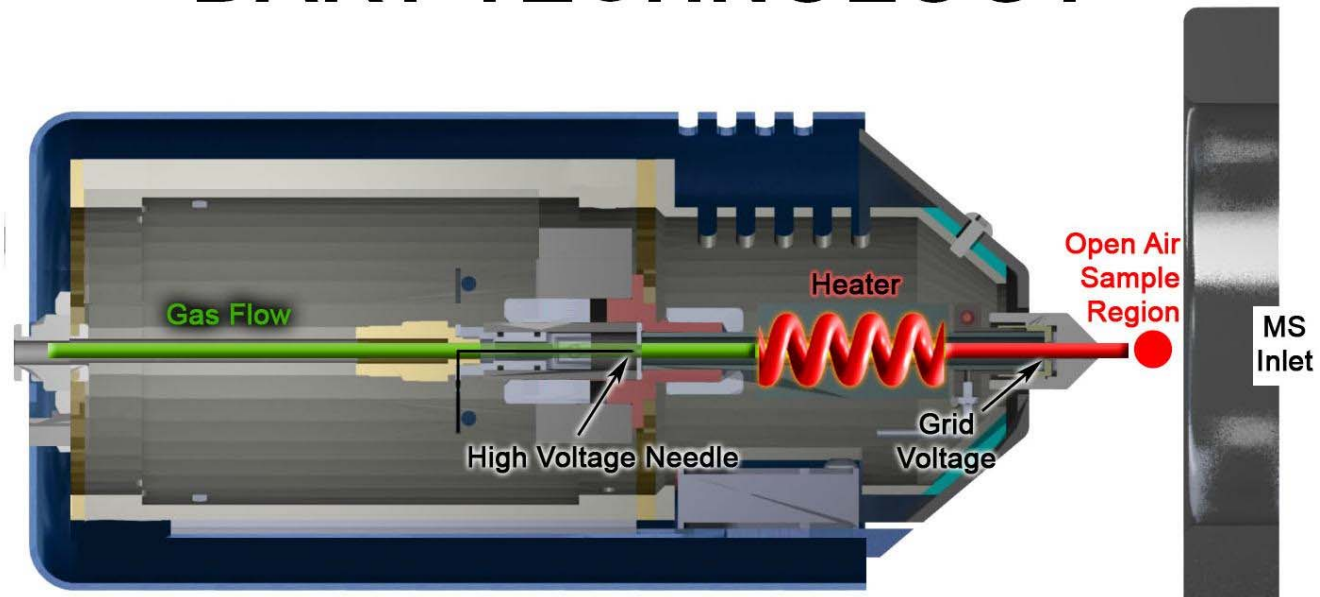


Half life in humans = 1 month



Direct Analysis in Real Time – DART

DART TECHNOLOGY

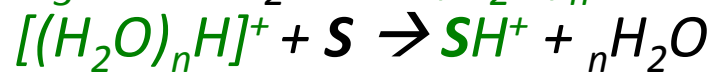
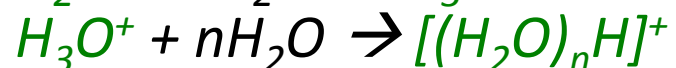
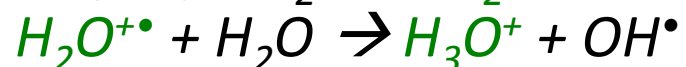
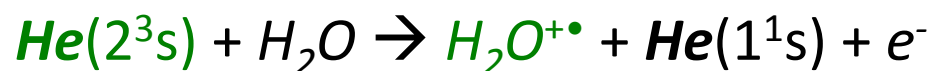


DART Principles

- Open air method – chemical ionisation
- Water present in the air is what ionizes sample



Where M is *excited* Helium ('positive' mode):



Cody, R.B.; Laramée, J.A.; Durst, H.D.. Anal. Chem., 2005, **77**(8): 2297-2302.

Song, L.; Dykstra, A.B.; Yao, H.; Bartmess, J.E. J. Am. Soc. Mass Spec. 2009, **20**(1): 42-50.



Advantages of DART

- Gases, liquids, solids
- Materials analysed directly on surfaces
- Raw, unprocessed body fluids
- Internal standards for quantification
- Samples do not have to be thermally stable or volatile
- Little to no fragmentation



Analyses Performed

- Four samples were analysed: 3 soil samples (extracted), one spike
- Temperature profile was obtained for the **blank spiked sample** to determine the optimal desorption temperature
- Additional testing of the samples was performed using the optimal temperature
- Samples were analyzed using QuickStrip cards and SPME (C18, C18/SCX (strong cation exchange), PDMS/DVB)
- Limit of detection was evaluated by performing a serial dilution of the stock blank spiked sample

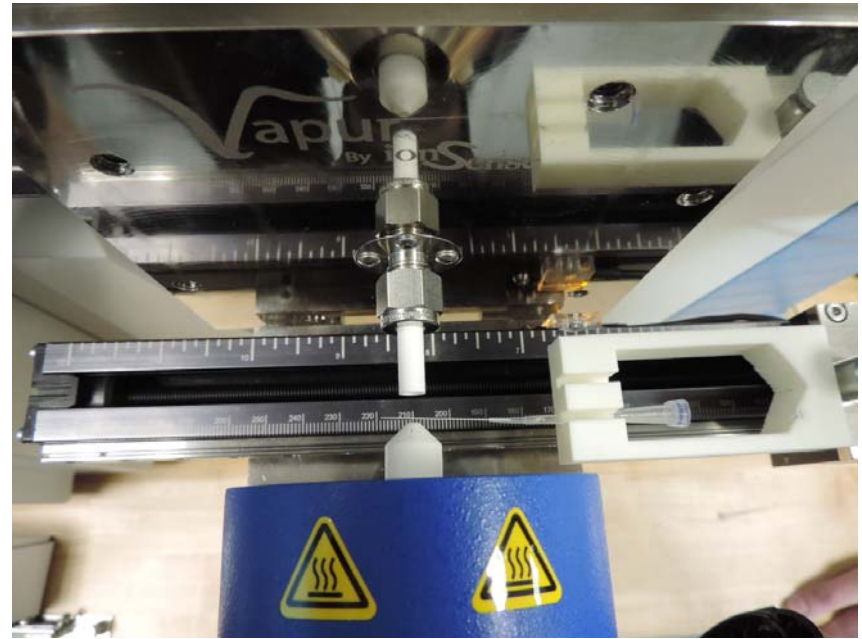
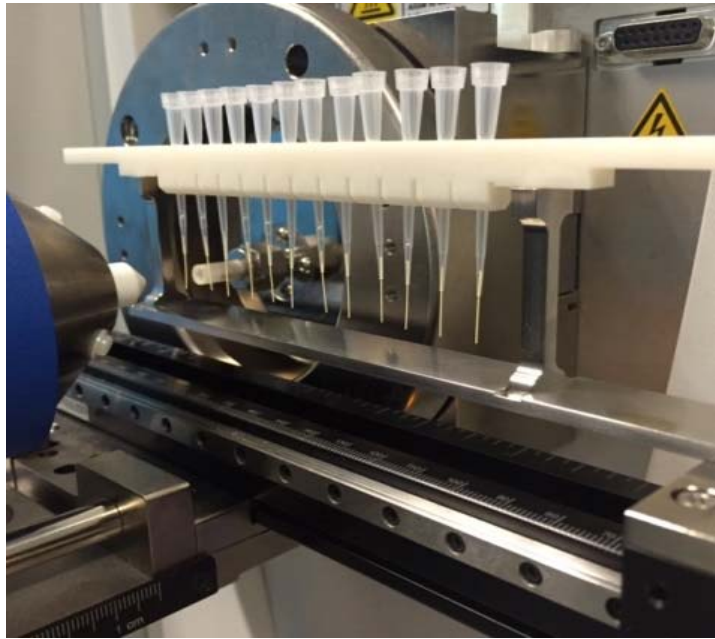


QuickStrip Cards

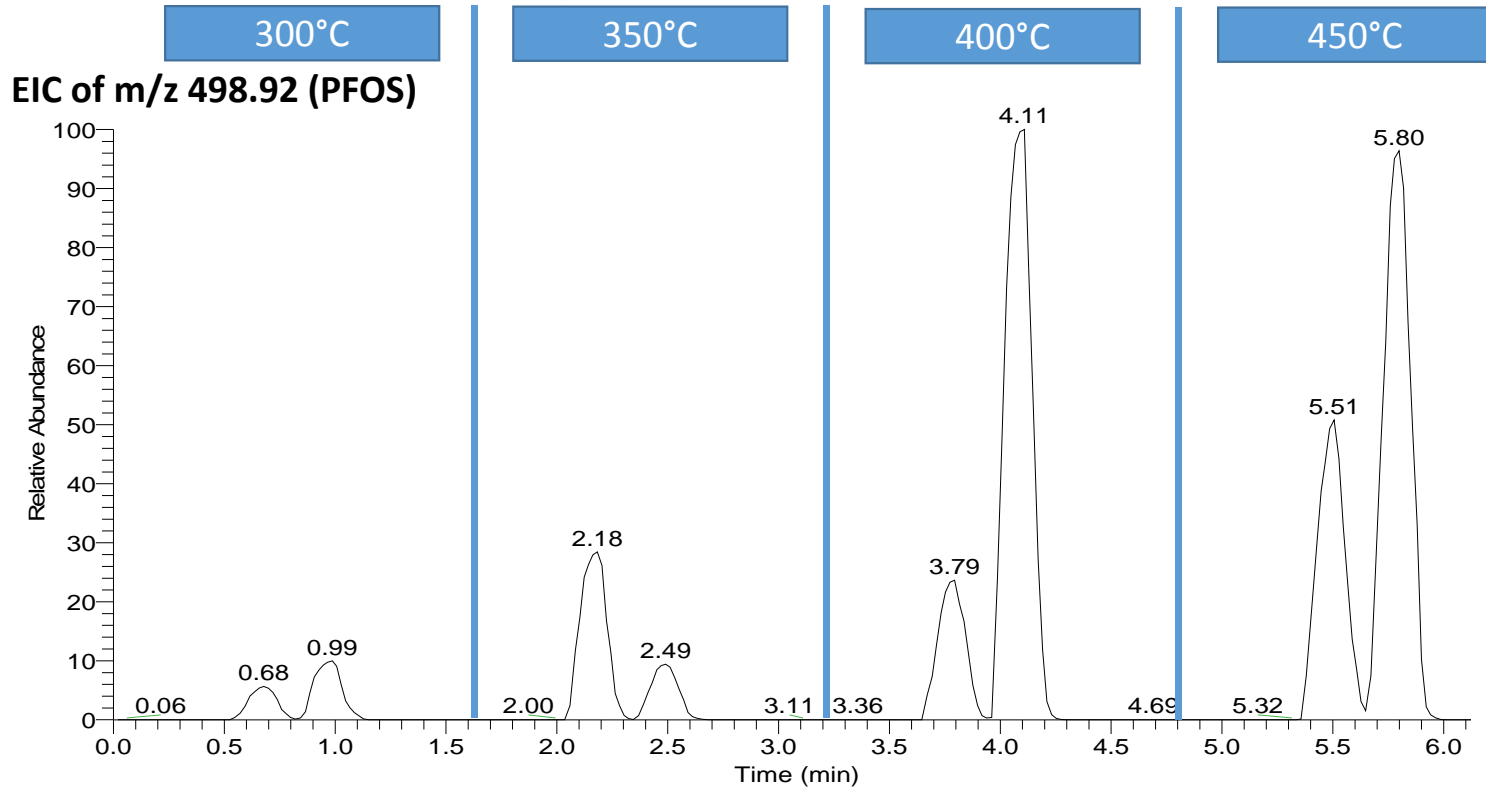
Stainless steel mesh – with
3 μL aliquot of liquid



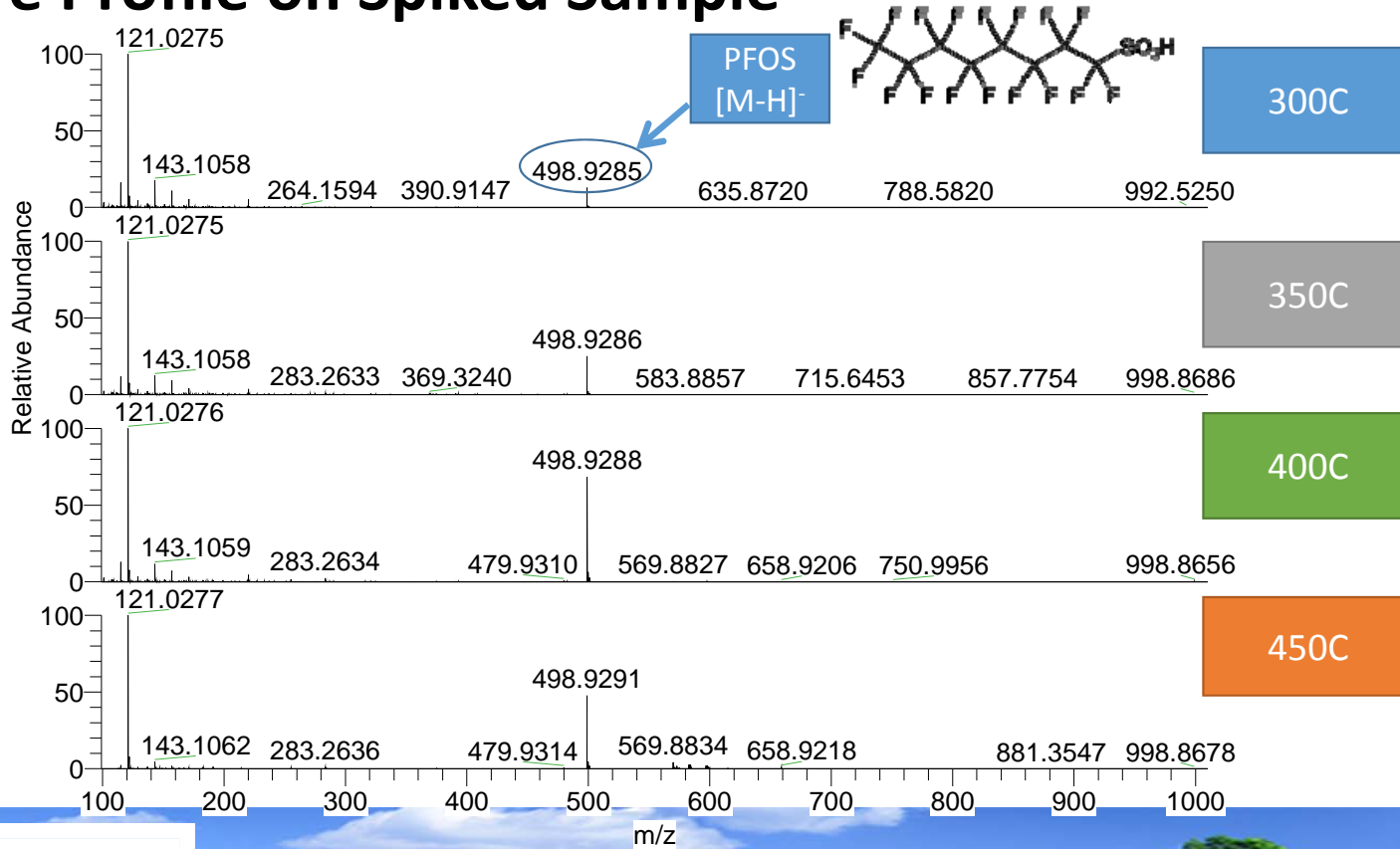
SPME C18



Temperature Profile Blank Spike



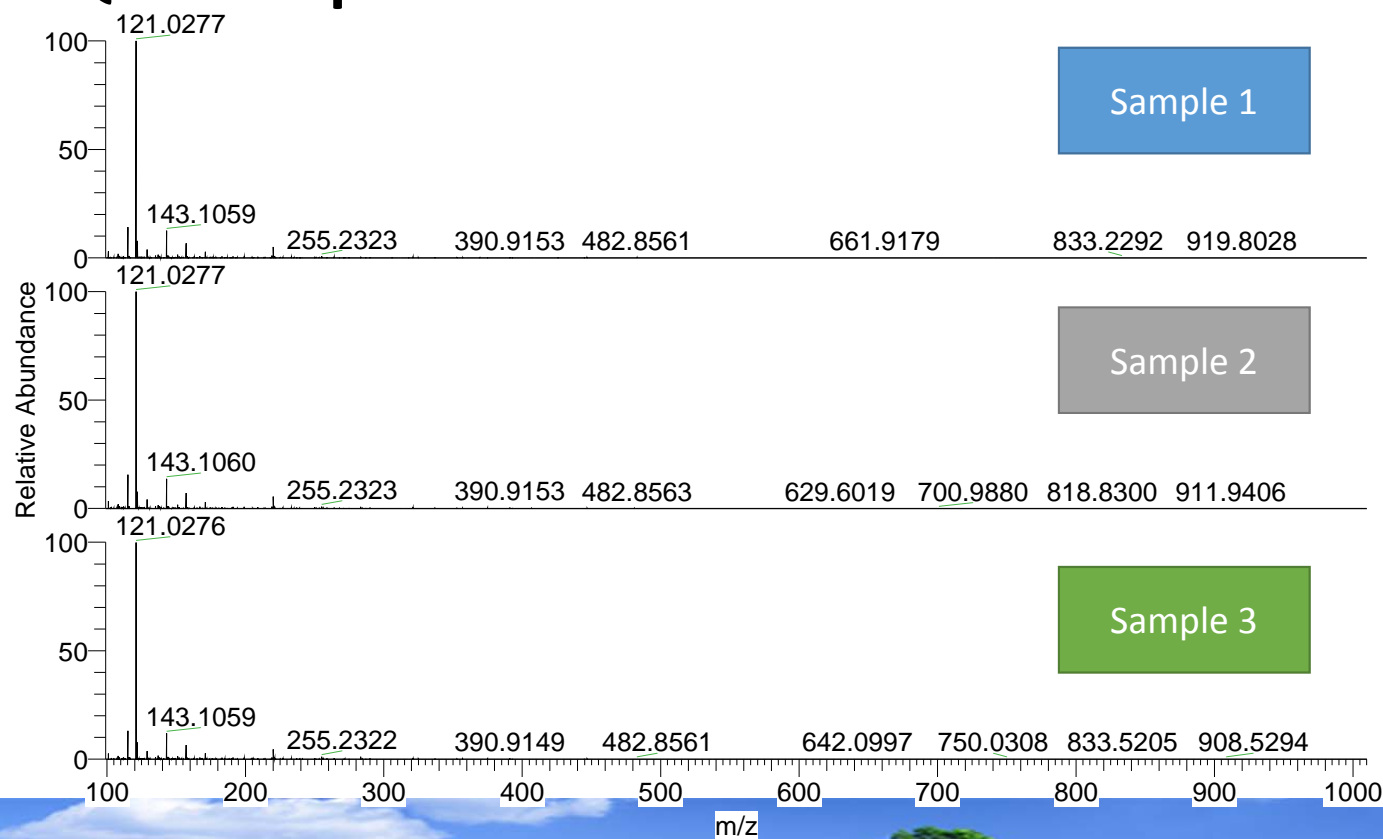
Temperature Profile on Spiked Sample



Samples Analysed with QuickStrip

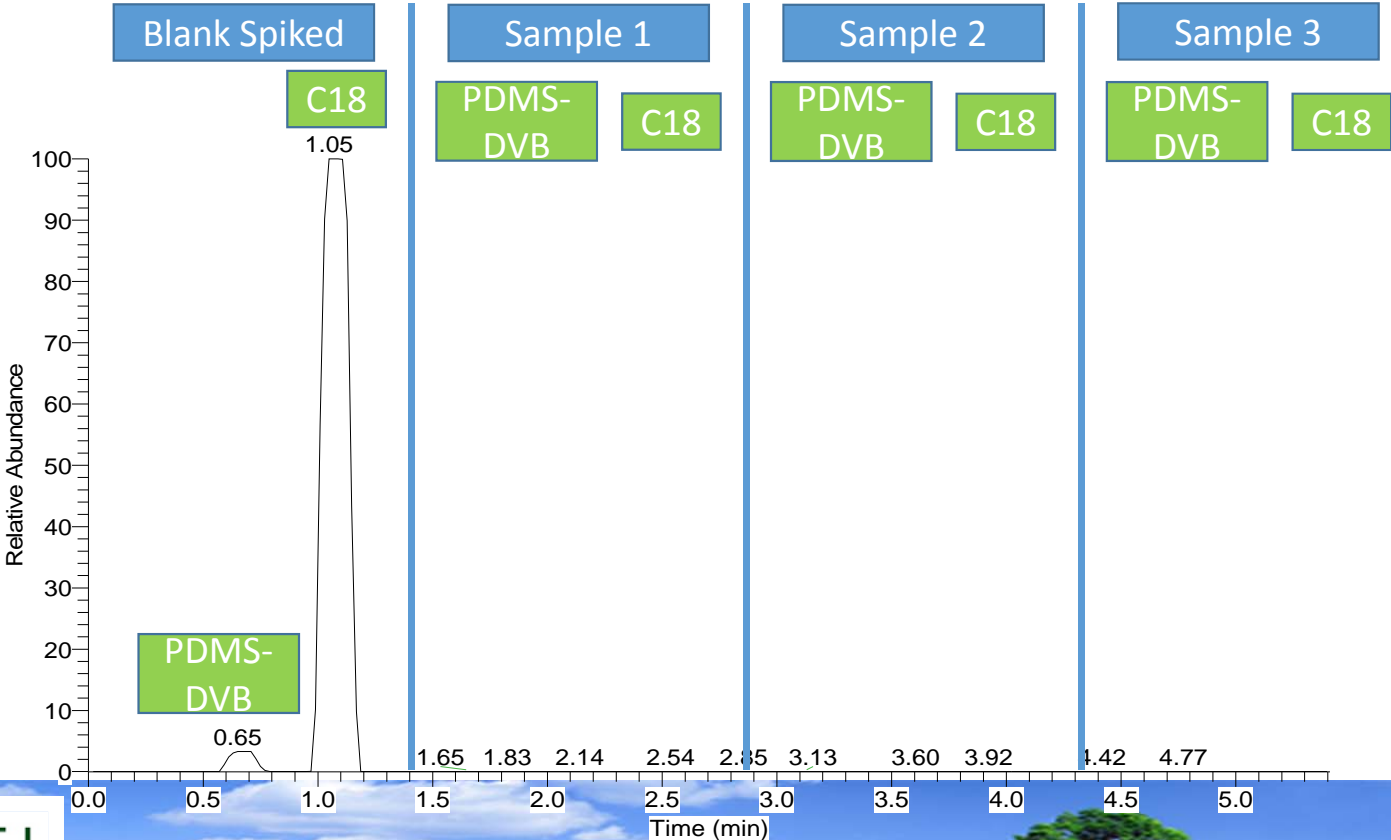
3 μL spotted on QuickStrip cards and analyzed at 400°C

m/z 498.92 (PFOS)



PDMS-DVB and C18 SPME

EIC of m/z
498.92 (PFOS)

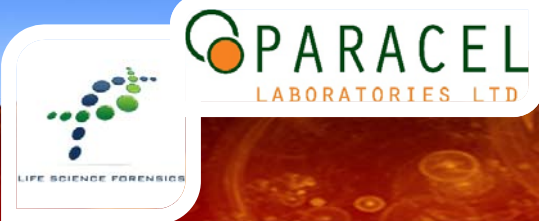
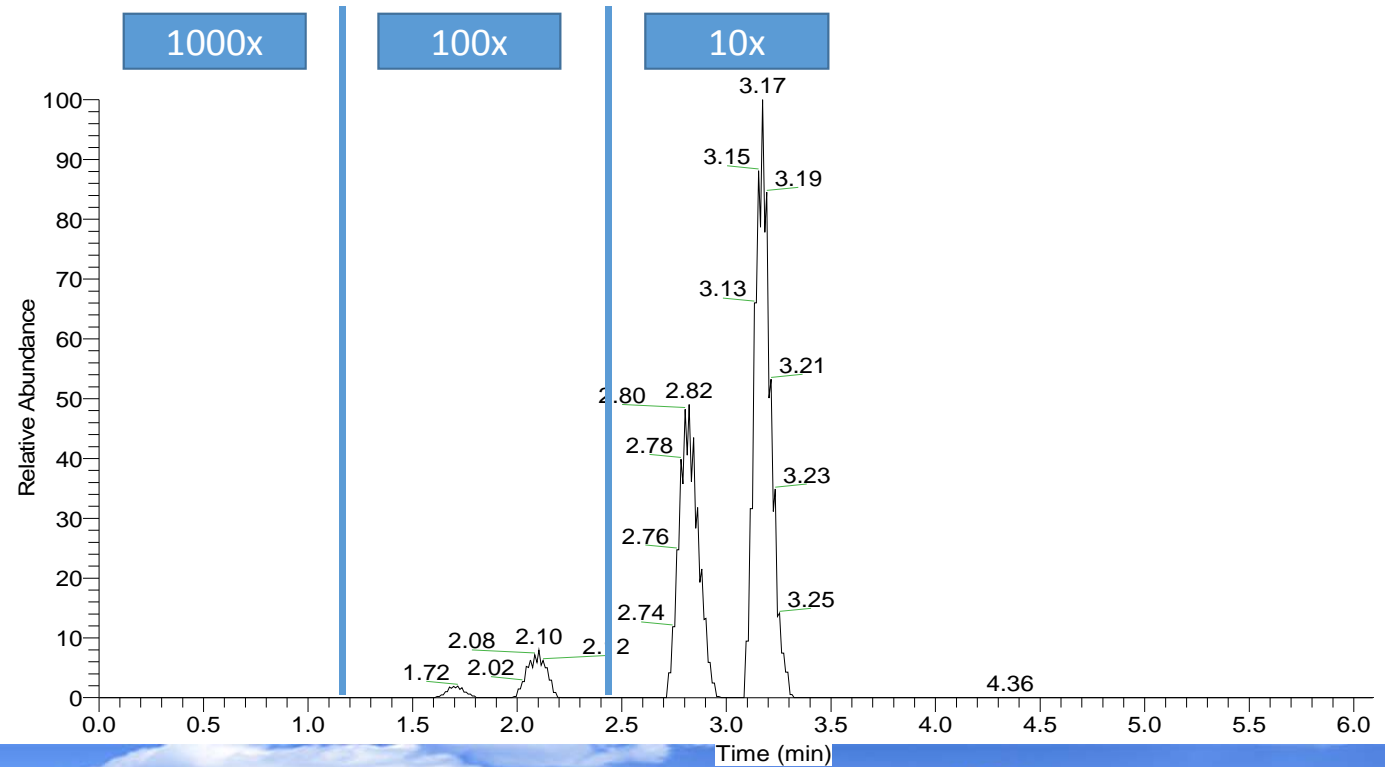


Limits of Detection – QuickStrip

EIC of m/z 498.92 (PFOS)

Serial dilution of the blank spiked sample

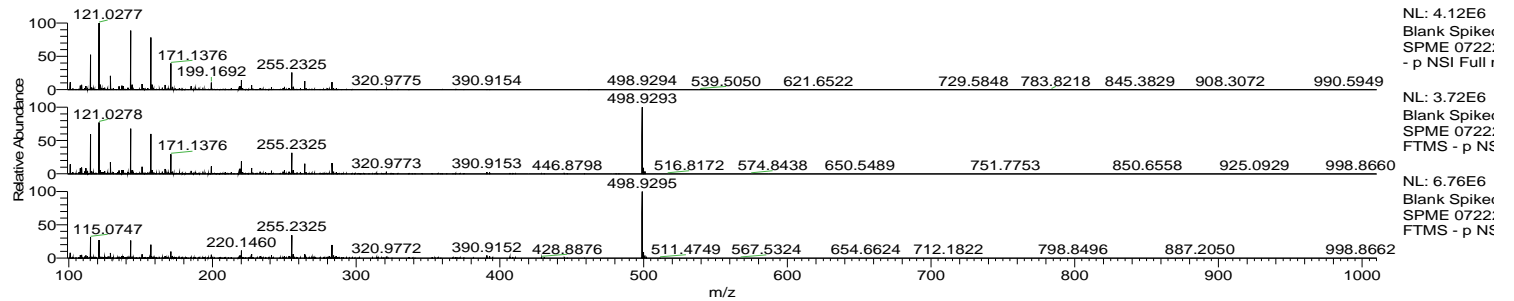
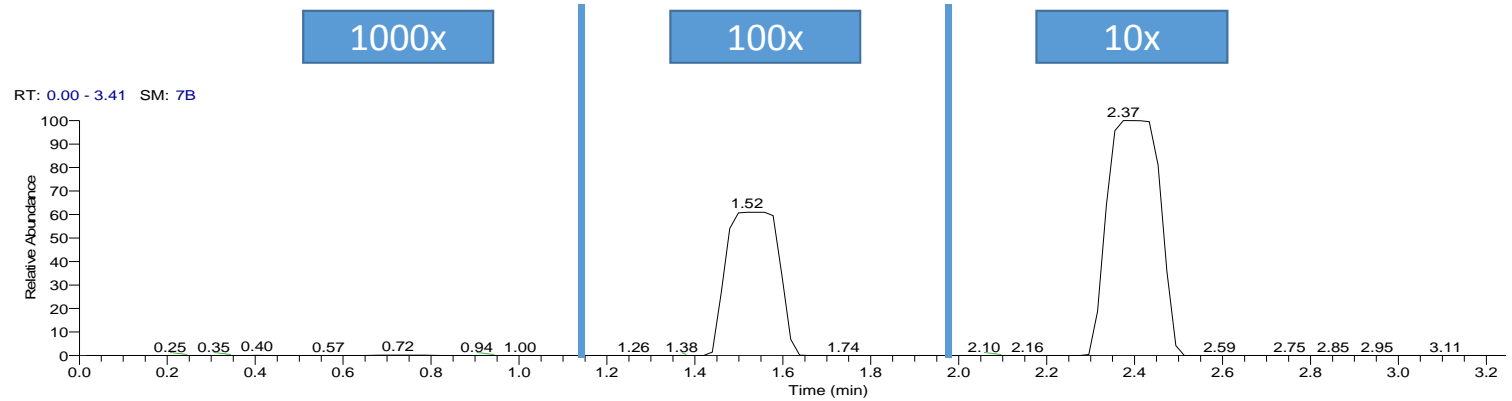
3 μ L spotted on QuickStrip cards and analyzed at 400°C



Limits of Detection – SPME C18

Desorbed at 400°C

Serial dilution of blank spiked sample



NL: 4.12E6
Blank Spiker
SPME 0722:
- p NSI Full r

NL: 3.72E6
Blank Spiker
SPME 0722:
FTMS - p NS

NL: 6.76E6
Blank Spiker
SPME 0722:
FTMS - p NS



Conclusions

- PFOS can be ionized by DART
- C18 SPME fibers was most effective in recovering PFOS
- PFOS was not found in any of the samples
- Limits of detection dependent on mass analyser rather than DART ionizer
- Possible to get low ppb detection limits



Acknowledgements

- Joseph LaPointe – Ionsense
- Ionsense analysis team
- Paracel and Life Science Forensics analysts

