CONffIDENCE: Contaminants in food and feed: Inexpensive detection for control of exposure

Evaluation in-house of an analytical method for perfluorinated compounds determination in fish

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Introduction:

PFCs are a class of organic compounds with a wide number of industrial applications due their physic-chemical properties, such as water and oil repellence, thermal and chemical stability These compounds have been employed in food packaging and paper products, in fire-extinguishing foam and insecticides. PFCs enter into the environment and have been detected in different water matrices and organisms.

WP1b is working in the analysis of 3 PFCs (perfluorooctanoic acid, PFOA; perfluorooctanesulfonate, PFOS and perfluorooctanesulfonamide, PFOSA) in real dietary food samples and the CSIC group have been assessed different real fish (n=15) and fish liver samples (n=12) from retail stores.

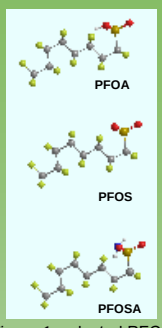
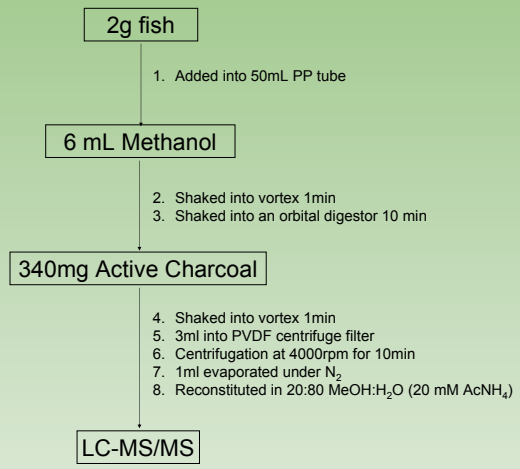


Figure 1: selected PFCs

Analytical Process:



Instrumental Parameters:

- LC system: Waters Alliance 2690 LC pump (Waters, Milford, MA, USA)
- Column: XTerra MS C18 3.5 µm (2.1 x 100 mm)
- Flow rate: 0.4µl/min
- Injection volume: 20µl
- Gradient elution mode: Water : MeOH (20mM Ammonium Acetate)
- Mass Spectrometrer: Quattro LC triple-quadruple mass spectrometer from Micromass (Manchester, UK)
- Ionization mode: orthogonal electrospray (ESI) in negative mode
- Adquisition mode: MRM
- Analysis time: 14min

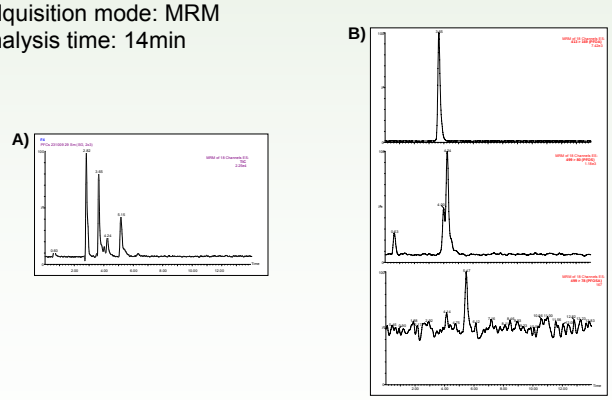


Figure 2: real spiked sample at 25ng/g level: **A)** Total Ion Chromatogram (TIC); **B)**MRM of selected PFCs.

Quality Parameters:

Table 1: Main parameters of the selected proceure developed by the ICT-Prague (concentration level: 25ng/g in sample).

	PFOS	PFOA	PFOSA
Recovery %	77	92	90
LOD (ng/g)	0.6	0.6	0.3
LOQ (ng/g)	2	2	1.5

LOD, LOQ: limit of detection and quantification, respectively.

Results:

Table 2: obtained results by the analysis of real samples from retail stores.

	Sample	PFOS	PFOA	PFOSA
Fish liver (ng/g)	1	< LOQ	< LOQ	< LOQ
	2	< LOQ	< LOQ	< LOQ
	3	< LOQ	n.d	n.d
	4	n.d	< LOQ	< LOQ
	5	n.d	< LOQ	< LOQ
	6	< LOQ	< LOQ	< LOQ
	7	< LOQ	< LOQ	< LOQ
	8	< LOQ	< LOQ	< LOQ
	9	< LOQ	< LOQ	< LOQ
	10	< LOQ	< LOQ	n.d
	11	2.1	n.d	n.d
	12	< LOQ	< LOQ	n.d
Fish muscle (ng/g)	1	n.d	< LOQ	< LOQ
	2	< LOQ	< LOQ	n.d
	3	n.d	< LOQ	n.d
	4	n.d	< LOQ	< LOQ
	5	n.d	< LOQ	< LOQ
	6	n.d	< LOQ	n.d
	7	< LOQ	< LOQ	< LOQ
	8	< LOQ	< LOQ	< LOQ
	9	< LOQ	< LOQ	< LOQ
	10	2.1	< LOQ	< LOQ
	11	2.8	< LOQ	< LOQ
	12	2.8	< LOQ	< LOQ
	13	n.d	< LOQ	n.d
	14	n.d	< LOQ	n.d
	15	n.d	n.d	n.d

Angler

Salmon

Conclusions:

PFCs have been detected in 100% of fish liver samples and in 93% of fish muscle samples. Most of the PFCs have been found below the LOQ. The next step should be the evaluation of the method for the analysis of PFCs in fish feed.

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