



METHOD STATEMENT

Determinand:

PFOS (Heptadecafluorooctanesulfonic acid), PFOA (Perfluorooctanoic acid), DEHP (Diethylhexyl phthalate) and Triclosan

Matrix:

Treated sewage effluents and surface waters

Principle of Method:

PFOS, PFOA, DEHP and Triclosan are extracted from an aqueous matrix via online SPE, utilising Thermo Scientific's EQuan LC system, equipped with a Hypersil Gold aQ pre-concentration column. The compounds are then backflushed from the pre-concentration column via a gradient run and are quantified by high resolution, accurate mass (HRAM) liquid chromatography mass spectrometry (LC-MS).

Sampling and Sample Preparation:

Samples should be taken in an STL101 bottle. No preservative is required. Samples are stored between 3 ± 2°C prior to analysis.

Samples are stable for times stated below, from sampling.

DEHP	3 days (in-house data)
PFOS	5 days (in-house data)
PFOA	5 days (in-house data)
Triclosan	7 days (in-house data)

Interferences:

The LC-MS system operates at a mass spectral resolution of 70,000 FWHM and therefore the technique is extremely selective, however in theory any substance with an equivalent LC retention time, and which generates ions within 0.005 mass units of the analyte may interfere.

Performance of Method:

Range of Method without dilution:

PFOS and PFOA: 0.09ng/l – 100ng/l
 DEHP: 200ng/l – 2,000ng/l
 Triclosan: 14ng/l – 10,000ng/l

Determinand	LOD µg/l	MRL µg/l	Low Std		High Std	
			%RSD	%Bias	%RSD	%Bias
DEHP	0.13	0.20	11.33	6.71	12.34	7.51
PFOS	0.0000676	0.000009	2.64	3.66	6.32	-2.20
PFOA	0.0000539	0.000009	4.94	5.48	3.11	-0.72
Triclosan	0.0064	0.010	5.97	2.18	3.81	-1.64



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Determinand	Spike	River Stanley Upstream Surface Water		River Stanley Treated Sewage	
		%RSD	%Rec.	%RSD	%Rec.
DEHP	400ng/L	-	-	8.47	101.1
	1600ng/L	6.23	105.4	4.25	102.9
PFOS	20ng/L	-	-	4.68	107.2
	80ng/L	5.53	94.41	4.15	96.9
PFOA	20ng/L	-	-	5.64	101.1
	80ng/L	5.17	95.87	2.38	94.76
Triclosan	2000ng/L	-	-	4.83	102.9
	8000ng/L	4.05	100.3	2.81	99.96

Uncertainty of Measurement:

The reported uncertainty is an expanded uncertainty calculated using a coverage factor of 2, which gives a level of confidence of approximately 95%.

Determinand	Uncertainty of Measurement %
DEHP	17.9
PFOS	16.7
PFOA	14.5
Triclosan	8.4

References:

In-house method - no external references