



METHOD STATEMENT

Determinand:

Ranitidine, Benzotriazole, Imidacloprid, Bisphenol S, Perfluoropentanoic Acid (PFPeA), Climbazole, Perfluorobutanesulfonic acid (PFBS), Perfluorohexanoic acid (PFHxA), Ammonium 2,3,3,3-tetrafluoro-2-(heptafluoropropoxy)propanoate (GEN-X), Perfluoroheptanoic acid (PFHpA), Perfluorohexanesulfonic acid (PFHxS), 6:2 Fluorotelomer Sulfonamide Alkylbetaine (6:2 FTAB), 6:2 Fluorotelomer Sulfonate (6:2 FTS), Perfluorooctanoic acid (PFOA), Perfluorooctanesulfonic acid (PFOS), Fipronil, Triclocarban

Matrix:

Surface Waters, Treated & Untreated Sewage Effluents

Principle of Method:

The compounds of interest 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 at $3 \pm 2^\circ\text{C}$ and analysed within 14 days of sampling for all compounds apart from Ranitidine which has 10 days and Benzotriazole which has 15 days.

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 5ppm of the analytes' monoisotopic mass, may interfere.

Performance of Method:

LOD, Precision and Bias

Determinand	LOD, ug/L				MRL ug/L	Low Std		High Std	
	Treated Sewage	Surface Water	Ground Water	Treated Sewage		%RSD	%Bias	%RSD	%Bias
Ranitidine	0.00890	0.00230	0.000804	0.0225	0.1	3.10	-2.89	2.87	-0.63
Benzotriazole	0.944	0.0140	0.00399	0.361	0.1	5.43	-1.24	2.26	-0.59
Imidacloprid	0.00205	0.000462	0.000293	0.00644	0.001	4.57	-2.81	1.53	-0.43
Bisphenol S	0.00426	0.00243	0.00284	0.0233	0.01	4.52	-3.87	2.30	-0.57
PFPeA	0.00191	0.000623	0.000332	0.00467	0.001	4.11	-2.35	2.13	-0.22
Climbazole	0.00297	0.00170	0.00149	0.0110	0.1	3.91	-1.39	1.98	-0.29
PFBS	0.000426	0.0000973	0.000179	0.00176	0.001	2.37	-2.52	2.21	-0.48
PFHxA	0.00126	0.000292	0.000192	0.00159	0.001	4.24	-2.99	1.32	-0.70
GenX	0.00129	0.000334	0.000147	0.00816	0.001	4.07	-2.81	1.13	-1.87
PFHpA	0.000591	0.000230	0.000158	0.00137	0.001	2.37	-2.81	2.21	-0.35
PFHxS	0.000424	0.0000788	0.000107	0.00112	0.001	2.37	-3.63	1.94	-0.94
6:2FTAB	0.00535	0.000507	0.000995	0.0221	0.001	8.26	0.76	8.76	-4.30
6:2FTS	0.00249	0.000326	0.000176	0.00176	0.001	2.21	-1.73	2.21	1.39
PFOA	0.00108	0.000232	0.0000668	0.000594	0.00009	2.37	-4.04	2.01	-0.38
PFOS	0.00830	0.000151	0.0000233	0.0200	0.00009	10.01	-3.95	4.45	-0.77
Fipronil	0.000939	0.000216	0.000234	0.00227	0.01	6.47	-3.14	2.03	0.28
Triclocarban	0.00132	0.00110	0.00115	0.00736	0.1	4.83	-1.99	1.63	0.30



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Matrix Spike Recoveries

Determinand	Treated Sewage 10%		Treated Sewage 60%		Surface Water 60%		Untreated Sewage 60%	
	%RSD	%Rec	%RSD	%Rec	%RSD	%Rec	%RSD	%Rec
Ranitidine	3.99	94.7	4.91	101	3.98	98.5	6.93	99.3
Benzotriazole	10.61*	106.0*	9.61*	95.7*	4.26	103.1	5.49	104.4
Imidacloprid	3.14	96.9	1.61	101	1.64	99.6	6.95	98.8
Bisphenol S	3.09	97.1	3.03	99.8	2.61	99.3	6.18	97.7
PFPeA	6.46	100	3.43	102	2.18	102	6.62	99.7
Climbazole	2.84	96.5	3.73	101	2.35	101	7.37	98.7
PFBS	2.93	96.8	2.24	99.2	1.34	99.1	6.60	98.4
PFHxA	2.33	97.1	3.33	101	2.28	100	6.38	97.7
GenX	4.78	95.9	3.04	99.9	1.58	101	6.92	98.5
PFHpA	2.29	95.7	2.42	99.2	1.49	99.1	7.52	97.8
PFHxS	3.46	94.8	2.68	96.8	1.08	97.3	6.73	96.1
6:2FTAB	18.19	93.9	9.73	98.2	9.70	128	12.50	109
6:2FTS	2.60	95.3	2.32	99.1	1.69	101	6.95	94.9
PFOA	4.54	84.0	5.70	105	2.53	102	2.48	102
PFOS	*	*	5.38	118	2.92	101	11.27	118
Fipronil	2.55	95.5	2.79	100	1.77	101	5.83	99.2
Triclocarban	5.28	96.2	3.93	98.5	2.49	98.9	7.25	98.2

* Data affected by very high levels of compound in sample matrix which exceeded the calibrated range of the method.

Uncertainty of Measurement:

Determinand	Uncertainty of Measurement %
Ranitidine	13.3
Benzotriazole	11.6*
Imidacloprid	9.38
Bisphenol S	9.08
PFPeA	12.9
Climbazole	9.18
PFBS	9.06
PFHxA	7.56
GenX	13.7
PFHpA	8.88
PFHxS	12.1
6:2FTAB	42.5
6:2FTS	9.90
PFOA	7.06*
PFOS	6.84*
Fipronil	9.6
Triclocarban	14.4

References:

15.1 UKWIR (2019) Final CIP3 Technical Specification and Guidance (03/03/2020)