

# METHOD STATEMENT



## Determinand:

Chloridazon, Bentazone, Metribuzin, Atrazine, Isoproturon, Diuron, Dichlorprop-p, Mecoprop, Pentachlorophenol, PFOS (Heptadecafluorooctanesulfonic acid), PFOA (Perfluorooctanoic acid) and DEHP (Diethylhexyl phthalate).

## Matrix:

Surface, Ground and Saline waters

## 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 between  $3 \pm 2^\circ\text{C}$  prior to analysis.

Samples are stable for times stated below, from sampling

DEHP	14 Days (in-house data)
PFOA, PFOS and Pentachlorophenol	26 Days (in-house data)
Chloridazon, Bentazone, Metribuzin, Atrazine, Isoproturon, Diuron, Dichlorprop-p and Mecoprop	49 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 5ppm of the analytes' monoisotopic mass, may interfere.

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## Performance of Method:

### Precision, Bias and Limit of Detection

Determinand	LOD µg/l	MRL µg/l	Low Std		High Std	
			%RSD	%Bias	%RSD	%Bias
Atrazine	0.00213	0.02	6.13	-2.51	3.44	1.37
Dichlorprop-p	0.00193	0.02	3.45	-2.82	2.71	1.32
Diuron	0.00199	0.004	6.88	-3.56	3.03	-0.61
Isoproturon	0.00045	0.004	4.45	-1.40	3.58	1.85
Perfluorooctanoic acid	0.000093	0.00013	4.77	-1.68	4.92	-0.15
Perfluorooctanesulfonic acid	0.000042	0.00013	9.61	-3.16	4.77	1.38
DEHP	0.1297	0.2	5.71	-2.27	4.63	1.75
Bentazone	0.00183	0.02	4.49	-3.48	3.08	-0.53
Pentachlorophenol	0.000201	0.02	3.34	-3.56	2.02	-0.27
Chloridazon	0.00052	0.004	4.31	-1.49	3.59	1.35
Metribuzin	0.0019	0.004	3.09	-0.87	3.38	0.21
Mecoprop-p	0.0018	0.02	4.46	-3.04	2.87	-0.30

### Matrix Spike Recoveries

Determinand	Saline Water - Aberdaron		Surface Water - Pocklington River, Upstream		Ground Water - Huncote BH	
	%RSD	%Rec.	%RSD	%Rec.	%RSD	%Rec.
Atrazine	4.95	102.21	7.32	111.94	5.48	100.45
Dichlorprop-p	7.50	95.62	3.23	104.63	2.47	101.76
Diuron	3.47	100.34	4.73	101.74	2.89	99.61
Isoproturon	3.69	101.34	5.03	103.5	3.34	101.84
Perfluorooctanoic acid	5.26	103.59	5.49	106.22	4.02	101.87
Perfluorooctanesulfonic acid	6.99	111.08	8.96	105.74	3.76	100.39
DEHP	4.70	101.63	4.09	99.65	4.64	101.74
Bentazone	3.12	98.59	3.98	102.23	3.42	99.02
Pentachlorophenol	2.64	100.98	2.32	100.75	3.45	101.27
Chloridazon	3.16	103.21	5.97	97.95	3.11	100.74
Metribuzin	4.48	102.33	4.65	105.34	3.41	100.68
Mecoprop-p	8.14	93.34	3.96	107.11	3.72	101.25

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## Uncertainty of Measurement

Determinand	Uncertainty of Measurement %	Minimum UoM (µg/L)
Atrazine	25.96	0.00050
Dichlorprop-p	10.73	0.0016
Diuron	11.29	0.0004
Isoproturon	10.67	0.0004
Perfluorooctanoic acid	17.19	0.00007
Perfluorooctanesulfonic acid	24.42	0.00004
DEHP	22.10	0.030
Bentazone	12.30	0.003
Pentachlorophenol	9.67	0.0005
Chloridazon	22.69	0.0006
Metribuzin	12.28	0.0009
Mecoprop-p	17.84	0.0016

## References:

In-house developed method