

METHOD STATEMENT



Determinand:

Substituted Urea, Simazine, Atrazine and Propyzamide

Matrix:

Surface waters, Ground waters, Trade effluents and Landfill leachates

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 stored at $3 \pm 2^\circ\text{C}$.

Samples are stable for 28 days (in-house data) from sampling.

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:

Range of Application: 0.01 - 1 $\mu\text{g/l}$

Determinand	LOD	MRL	Low Std		High Std	
	$\mu\text{g/l}$	$\mu\text{g/l}$	% RSD	% Bias	% RSD	% Bias
Chlorotoluron	0.00191	0.010	2.98	2.46	1.91	1.31
Diuron	0.00569	0.010	2.48	3.21	2.92	2.53
Isoproturon	0.00142	0.010	1.86	2.71	1.08	0.83
Linuron	0.00234	0.010	3.36	3.87	1.57	2.29
Methabenzthiazuron	0.00341	0.010	8.52	3.08	10.12	2.27
Monolinuron	0.00156	0.010	2.24	2.44	1.53	0.38
Monuron	0.00125	0.010	2.00	3.17	1.14	1.27
Atrazine	0.00215	0.010	2.24	3.66	2.32	1.54
Propyzamide	0.00250	0.010	2.03	3.33	1.54	2.49
Simazine	0.00216	0.010	1.97	2.56	1.52	0.80

Spiked Sample Recoveries

Determinand	Ground Water		Surface Water		Trade Effluent		Landfill Leachate	
	%RSD	%Rec.	%RSD	%Rec.	%RSD	%Rec.	%RSD	%Rec.
Chlorotoluron	2.77	104.67	3.75	109.56	1.70	106.01	3.63	106.07
Diuron	2.39	106.32	2.49	109.72	2.03	106.79	3.40	111.61
Isoproturon	2.20	104.46	1.54	103.07	1.60	103.96	2.51	102.07
Linuron	1.72	106.32	2.16	107.44	1.87	106.49	3.37	109.11
Methabenzthiazuron	11.54	102.89	8.65	94.60	8.86	102.61	14.42	109.51
Monolinuron	1.77	104.02	3.35	99.83	2.11	103.59	7.17	104.62
Monuron	1.19	104.67	2.00	106.98	1.22	104.94	4.66	107.46
Atrazine	2.06	105.42	2.47	111.37	2.43	106.57	5.71	110.56
Propyzamide	2.02	106.11	1.92	107.92	1.27	105.41	3.68	108.99
Simazine	1.38	104.69	1.58	103.34	1.31	104.65	1.85	105.79

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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 %
Chlorotoluron	15.03
Diuron	19.11
Isoproturon	9.25
Linuron	15.66
Methabenzthiazuron	24.57
Monolinuron	9.72
Monuron	13.40
Atrazine	19.51
Propyzamide	17.49
Simazine	10.84

References:

In-house developed method