METHOD STATEMENT

Determinand:
Analysis of Emerging Organic Contaminants for the EU water framework directive in Saline, Ground and Surface Waters.

Matrix:
Saline, Ground and Surface Waters

Principle of Method:
The compounds of interest are extracted from an aqueous matrix via Agilent's online SPE system, equipped with a Hypersil Gold aQ pre-concentration column. The compounds are then flushed from the pre-concentration column via a gradient run and are separated on an Agilent Eclipse Plus C18 RRHD 3.0x55mm, 1.8um analytical column, before being ionised by Agilent's Jetstream Electrospray technology, and analysed using MRM transitions on an Agilent 6470 LCMSMS

Sampling and Sample Preparation:
Samples should be taken in an STL33 bottle containing 250ul of 2.51M Citric acid, containing 480mg/L of Silver citrate (**Error! Reference source not found.**)

All determinands are stable for 14 days from sampling with the exception of Dichlorvos and Metsulfuron-methyl* which have been shown to be stable for 10 days.

(*Metsulfuron-methyl suitable for analysis in groundwater and surface water only due to low stability in saline matrix).

Interferences
Interfering compounds would have to display the identical MRM transition at the same retention time. However, any compound, which passes through the extraction procedure, and has a similar liquid chromatographic retention time and mass spectrometric properties to the compound of interest, will cause interference.

Performance of Method:

Determinand	LOD µg/l	LOQ µg/l	MRL µg/l	Low Std		High Std	
				%RSD	%Bias	%RSD	%Bias
Diclofenac	0.000391	0.000781	0.002	10.33	-0.51	6.33	-1.66
Dichlorvos	0.0000261	0.0000522	0.0001	13.18	1.02	8.45	-0.10
Cybutryne	0.0000272	0.0000544	0.0002	2.43	-1.82	2.78	-0.49
MCPA	0.00161	0.00323	0.01	1.52	-1.35	2.80	0.86
Metsulfuron-methyl	0.00199	0.00399	0.004	10.57	1.11	8.65	-1.58
Pirimicarb	0.000819	0.00164	0.002	12.18	-0.31	9.89	1.64
Simazine	0.000194	0.000387	0.002	2.10	-1.22	3.04	-0.13
Triclosan	0.00115	0.00229	0.003	5.69	-0.82	5.41	0.74
Sulfosulfuron	0.00399	0.00797	0.01	6.89	-12.14	7.99	-10.00

Determinand	Saline Water - Aberdaron		Surface Water – Draycote Reservoir		Ground Water - Huncote	
	%RSD	%Rec.	%RSD	%Rec.	%RSD	%Rec.
Diclofenac	9.86	103.54	7.69	89.34	8.08	93.44
Dichlorvos	8.78	94.60	10.21	96.71	9.64	96.87
Cybutryne	2.47	98.43	2.46	98.21	2.53	97.09
MCPA	2.66	101.44	2.53	99.94	3.07	99.56
Metsulfuron-methyl	--- *	--- *	5.21	99.11	8.87	97.31
Pirimicarb	5.33	98.15	5.88	98.85	3.99	98.25
Simazine	2.05	99.79	2.14	99.71	2.65	99.05
Triclosan	5.77	99.92	5.27	99.27	5.29	97.89
Sulfosulfuron	7.47	94.07	8.61	92.56	8.39	92.42

Uncertainty of Measurement

Determinand	Uncertainty of Measurement %
Diclofenac	20.64
Dichlorvos	18.22
Cybutryne	8.04
MCPA	11.12
Metsulfuron-methyl	19.47
Pirimicarb	20.94
Simazine	9.53
Triclosan	13.41
Sulfosulfuron	22.60