

# METHOD STATEMENT



## Determinand:

Trace level Pesticides and selected non-polar organic compounds

## Matrix:

Surface, Ground and Saline waters

## Principle of Method:

Approximately 400mls of sample is extracted with 20mls of iso-hexane. The extract is analysed by GC-MSMS with negative chemical ionisation (NCI)

## Sampling and Sample Preparation:

Samples should be taken in a 500ml amber or green glass bottle with 25mls of methanol as a preservative.

Samples are stored at  $3 \pm 2^{\circ}\text{C}$  and analysed within 14 days of sampling for all compounds except those listed below. (In-house stability data)

Cypermethrin

8 days (in-house data)

## Interferences:

MS/MS is an extremely selective technique and interferences should only be encountered very rarely, however in theory, any compound which is extracted by the procedure, which has a chromatographic retention time similar to the compound of interest and which produces both precursor and product ions similar to that of the compounds in question, may interfere.

## Performance of Method:

Precision, Bias and Limit of Detection

Compound	LOD (ng/L)	Calibration Range	MRL ( $\mu\text{g/l}$ )	Low Standard		High Standard	
				Bias	RSD	Bias	RSD
alpha-Endosulphan	0.0509	0.00005 - 0.01000	0.00008	1.87%	7.39%	2.75%	5.79%
beta-Endosulphan	0.0291	0.00005 - 0.01000	0.000075	-3.84%	7.66%	-0.89%	7.04%
Endosulphan Sulphate	0.0119	0.00005 - 0.01000	0.000050	-4.49%	8.52%	-1.88%	7.66%
Endosulphan - Total	0.0770	0.00015 - 0.03000	0.00016	-2.15%	7.25%	-0.01%	6.58%
Bifenox	0.0344	0.00005 - 0.01000	0.000070	-2.84%	7.79%	0.88%	7.20%
Cypermethrin	0.0067	0.00001 - 0.00200	0.000015	-3.24%	5.01%	-1.59%	3.52%

# METHOD STATEMENT



## Matrix Spike Recoveries

Compound	Surface Water		Ground Water		Saline Water	
	Recovery	RSD	Recovery	RSD	Recovery	RSD
alpha-Endosulphan	102.08%	5.85%	104.76%	4.69%	105.55%	6.25%
beta-Endosulphan	98.59%	6.14%	101.03%	4.95%	103.40%	5.08%
Endosulphan Sulphate	99.69%	5.94%	100.38%	5.62%	102.59%	5.75%
Endosulphan - Total	100.12%	5.19%	102.05%	4.69%	103.85%	5.31%
BifenoX	100.10%	7.72%	101.97%	5.92%	101.18%	7.07%
Cypermethrin	98.50%	4.13%	100.04%	4.59%	99.92%	4.33%

## 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%.

Compound	Relative UoM (%)	Minimum UoM ( $\mu\text{g/L}$ )
BifenoX	16.7	0.000026
Cypermethrin	9.8	0.000005
Endosulphan - Total WFD	15.7	0.00005
alpha-Endosulphan	16.1	0.00003
beta-Endosulphan	20.2	0.000017
Endosulphan Sulphate	16.7	0.000021

## References:

EU Priority Substances Directive 2013  
Directive 2013/39/EC

EQS Substance Datasheet - Priority Substance No. 18. 2005

EQS Substance Datasheet - Priority Substance No. 14. 2005