

METHOD STATEMENT



Determinand:

Triclosan and Diclofenac

Matrix:

Untreated Sewage, Treated Sewage Effluent, Surface Water and Groundwaters.

Principle of Method:

An automated dispersive liquid/liquid microextraction procedure (DLLME) is used to extract the compounds of interest from an aqueous matrix. The dispersive solvent is isopropanol and the extraction solvent a mixture of dichloromethane and n-pentane (80:20). The extract is then analysed using MRM transitions on an Agilent 7000C GC MS Triple Quad (GCMSMS) operated in Negative Chemical Ionisation (NCI) mode

Sampling and Sample Preparation:

Water samples are to be supplied in 40ml amber screw top glass vials – STL90 and must be extracted within 7 days after sampling for Triclosan analysis and 15 days after sampling for Diclofenac analysis. They must be taken without any significant headspace – that is filled at least up to the shoulder of the vial - and delivered to the laboratory.

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

Extracts must be stored at room temperature, if they cannot be analysed on the day that they are vialled.

Samples must be allowed to warm to room temperature overnight before extraction.

Interferences

Any co-extracted material with a corresponding GC retention time and similar mass spectrum will interfere.

Performance of Method:

LOD, Precision and Bias

Compound	LOD (ng/L)	Low Standard		High Standard	
		Bias (%)	RSD (%)	Bias (%)	RSD (%)
Triclosan	3.424	11.04	6.25	9.02	8.54
Diclofenac	0.867	4.00	1.39	-1.89	1.32

Matrix Spike Recoveries

Compound	Final Effluent (100ng/L)		Final Effluent (400ng/L)		Surface Water (400ng/L)	
	Rec. (%)	RSD (%)	Rec. (%)	RSD (%)	Rec. (%)	RSD (%)
Triclosan	93.31	11.10	92.61	7.24	95.29	9.45
Diclofenac	98.83	1.18	94.45	1.33	94.88	1.38

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Compound	Crude Sewage (4000ng/L)		Groundwater (400ng/L)	
	Rec. (%)	RSD (%)	Rec. (%)	RSD (%)
Triclosan	112.04	9.77	105.11	6.95
Diclofenac	99.65	1.60	98.13	1.72

Uncertainty of Measurement:

Compound	Uncertainty of Measurement
Triclosan	28.89%
Diclofenac	3.53%

References:

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