

METHOD STATEMENT



Determinand:

4-Tert-Butylphenol

Matrix:

Surface Waters, Untreated Sewage and Treated Sewage Effluent

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 7010 GC MS Triple Quad (GCMSMS). A sandwich injection technique is employed to co-inject an analyte protectant solution (3-Ethoxy-1,2-propanediol) together with the sample extract onto the GCMSMS, this improves the chromatographic performance of the target analyte

Sampling and Sample Preparation:

Water samples are to be supplied in 40ml amber screw top glass vials.

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. Where significant headspace is evident a senior analyst must be informed.

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	Low Standard		High Standard	
	Bias (%)	RSD (%)	Bias (%)	RSD (%)
4-tert-Butylphenol	-4.18	6.66	0.26	5.04

Matrix Spike Recoveries

Compound	Final Effluent (100ng/L)		Final Effluent (800ng/L)		Surface Water (800ng/L)		Crude Sewage (8000ng/L)	
	Rec. (%)	RSD (%)	Rec. (%)	RSD (%)	Rec. (%)	RSD (%)	Rec. (%)	RSD (%)
4-tert-Butylphenol	89.8	7.17	96.7	5.69	96.7	4.46	98.2	3.95

Limits of Detection

Compound	Final Effluent (ng/L)		Surface Water (ng/L)		Crude Sewage (ng/L)	
	LOD	MRL	LOD	MRL	LOD	MRL
4-tert-Butylphenol	6.0	10	5.7	10	55.8	100

Uncertainty of Measurement:

Compound	Uncertainty of Measurement
4-tert-Butylphenol	24.57%

References:

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