

METHOD STATEMENT



Determinand:

TCEP and TCPP

Matrix:

Treated sewage effluents.

Principle of Method:

TCEP and TCEP are extracted from aqueous solution using a mixture of ethylacetate and hexane solvent. The extract is quantified by gas chromatography with mass spectrometry detection (GCMSMS). The mass spectrometer is operated in electron impact positive ion mode with multiple reaction monitoring (MRM).

Sampling and Sample Preparation:

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

Samples are stable for 14 days (in-house data) from sampling

Interferences:

GC-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 GC retention time similar to the compound of interest and which produces both parent and daughter ions similar to that of the compounds in question, may interfere.

Performance of Method:

Range of Method: $1\mu\text{g/l}$ - $10\mu\text{g/l}$ without dilution

Determinand	MCERTS Accred.	LOD $\mu\text{g/l}$	MRL $\mu\text{g/l}$	Low Std		High Std	
				%RSD	%Bias	%RSD	%Bias
TCEP	✓	0.3532	1	8.63	-2.83	5.90	-0.60
TCPP	✓	0.3924	1	10.70	-1.29	6.51	-0.38

Determinand		Derby STW FE		Warwick STW FE		Finham STW FE	
		Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk
TCEP	%Rec.	98.26	97.42	93.33	98.70	95.69	99.64
	%RSD	11.94	8.47	11.23	6.63	11.73	6.76
TCEP	%Rec.	95.20	94.73	87.45	97.27	93.09	97.07
	%RSD	11.74	6.99	7.78	7.38	11.13	4.95

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 %
TCEP	17.19
TCPP	20.48

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References:

Who_Ehc_209 - Flame Retardants:Tris(Chloropropyl)Phosphate And Tris(2-Chloroethyl) Phosphate

HP 6890 Series Gas Chromatograph Operating Manual Vol. 1. General Information G1530-90440.

Environmental GCMSD Instrument and Chemstation Operation Vols. 1 and 2. G 1701 BA.

MCERTS (Waters) Standard Version 3