

METHOD STATEMENT



Determinand:

Organotin compounds - tri-n-butyltin and triphenyltin

Matrix:

Groundwaters, surface waters, crude sewage, effluents & leachates.

Principle of Method:

The named compounds are ethylated in the aqueous phase using sodium tetraethylborate. The ethylated derivatives are extracted into hexane and quantified by gas chromatography with mass spectrometry detection (GCMS). The mass spectrometer is operated in electron impact mode with specific ion monitoring.

Sampling and Sample Preparation:

Samples should be collected in glass bottles with PTFE-lined screw caps and stored at 3±2°C. Samples are stable for 7 days (ISO 5667:3) from sampling.

Interferences:

Any substance, or substance yielding an ethyl derivative, with a corresponding GC retention time and with the same ions as those being monitored, will interfere.

Performance of Method:

Ranges of Method: 20ng/l - 400ng/l without dilution

Determinand	CAS Number	LOD ng/l	Reporting Limit (ng/l)	Low Standard		High Standard	
				% Rec.	% RSD	% Rec.	% RSD
Tributyl Tin	56573-85-4	2.8653	20	103.9%	10.79%	100.7%	5.79%
Triphenyl Tin	668-34-8	4.86546	20	99.1%	10.76%	100.2%	6.33%

Determinand		Finham FE		Trade		Barston FE		Groundwater	
		Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk
Tributyl Tin	% Rec.	103.5	92.2	108.7	106.4	97.8	90.2	82.94	97.18
	% RSD	6.41	7.06	5.72	9.00	6.53	5.30	9.99	5.99
Triphenyl Tin	% Rec.	118.7	105.9	97.5	115.8	106.6	88.9	93.46	107.46
	% RSD	12.75	6.34	7.46	12.53	11.65	8.54	19.5	15.17

Determinand		Landfill Leachate		Soil Leachate		Crude	
		Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk
Tributyl Tin	% Rec.	97.6	87.2	92.4	92.4	108.8	99.2
	% RSD	7.18	10.92	8.41	8.41	5.72	6.88
Triphenyl Tin	% Rec.	102.6	92.6	105.4	105.4	100.6	98.3
	% RSD	6.79	7.98	7.63	7.63	7.46	7.61

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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 (%)
Tributyl Tin	32.88
Triphenyl Tin	39.44

References:

Determination of Organotin Compounds in Environmental samples - Analytical Sciences, April 2000, Vol.16, pp. 349-359 (The Japan Society for Analytical Chemistry).

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.

ISO 5667-3 2018 - Water quality Sampling Part 3: Guidance on the preservation and handling of water samples