

METHOD STATEMENT



Determinand:

Organotin compounds - di-n-butyltin, tri-n-butyltin and triphenyltin

Matrix:

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

Principle of Method:

Triphenyl and tributyl tin are derivatised by ethylation in the aqueous phase and subsequently extracted into an organic phase using hexane. A portion of this extract is then analysed by gas chromatography with mass spectrometric detection (GC-MS). 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 $3 \pm 2^\circ\text{C}$. 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
Dibutyl Tin	1191-48-6	9.93583	20	107.5%	12.27%	104.7%	16.01%
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
Dibutyl Tin	% Rec.	87.9	83.6	96.0	117.9	106.9	83.6	95.90	100.67
	% RSD	13.74	9.77	14.21	24.02	16.39	9.82	16.93	11.13
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
Dibutyl Tin	% Rec.	87.5	85.2	62.6	62.6	94.4	92.1
	% RSD	13.58	18.36	13.42	13.42	14.21	12.09
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 (%)
Dibutyl Tin	122.38
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