

METHOD STATEMENT



Determinand:

Siloxanes

Matrix:

Surface Water, Untreated Sewage and Treated Sewage Effluent

Principle of Method:

Headspace extraction of the Siloxane compounds from the sample using a commercial headspace sampler followed by separation and quantitative determination of the compounds using GCMS.

Sampling and Sample Preparation:

Water samples are to be supplied in 40ml screw top glass vials. They must be taken without any significant headspace and delivered to the laboratory. Where significant headspace is evident (see appendix IV), analysis will be undertaken but the results must be accompanied by an analyst comment informing the client of the potential for losses to have occurred.

Samples should be stored at $3 \pm 2^\circ\text{C}$ and analysed within 14 days of sampling

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	Final Effluent ($\mu\text{g/L}$)		Surface Water ($\mu\text{g/L}$)		Crude Sewage ($\mu\text{g/L}$)	
	LOD	MRL	LOD	MRL	LOD	MRL
Hexamethyldisiloxane	0.0030	0.010	0.0048	0.010	0.051	0.10
1,1,1,3,5,5,5-heptamethyltrisiloxane	0.0030	0.010	0.0024	0.010	0.027	0.10
Octamethyltrisiloxane	0.0028	0.010	0.0016	0.010	0.010	0.10
Octamethylcyclotetrasiloxane	0.0048	0.007	0.0066	0.007	0.018	0.07
1,1,1,3,5,5,5-heptamethyl-3-((trimethylsilyl)oxy)trisiloxane	0.0020	0.010	0.0020	0.010	0.024	0.10
Decamethyltetrasiloxane	0.0013	0.010	0.0019	0.010	0.015	0.10
Decamethylcyclopentasiloxane	0.0032	0.020	0.0063	0.020	0.025	0.20
Dodecamethylpentasiloxane	0.0030	0.010	0.0032	0.010	0.032	0.10
Dodecamethylcyclohexasiloxane	0.0099	0.010	0.0173	0.010	0.097	0.10

Compound	Low Standard		High Standard	
	Bias (%)	RSD (%)	Bias (%)	RSD (%)
Hexamethyldisiloxane	11.82	9.08	7.85	6.44
1,1,1,3,5,5,5-heptamethyltrisiloxane	4.04	10.19	1.59	7.94
Octamethyltrisiloxane	9.28	8.12	7.23	6.74
Octamethylcyclotetrasiloxane	11.28	5.75	7.47	4.29
1,1,1,3,5,5,5-heptamethyl-3-((trimethylsilyl)oxy)trisiloxane	0.19	5.15	1.63	3.73
Decamethyltetrasiloxane	1.28	4.88	1.74	3.85
Decamethylcyclopentasiloxane	4.86	4.47	4.37	2.24
Dodecamethylpentasiloxane	1.49	6.10	-0.11	5.19
Dodecamethylcyclohexasiloxane	1.43	5.29	1.70	2.61

METHOD STATEMENT



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 (%)
Hexamethyldisiloxane	113.9	6.15	109.2	5.99	108.5	5.24	102.3	13.48
1,1,1,3,5,5,5-heptamethyltrisiloxane	79.8	11.37	84.0	11.61	74.2	15.83	104.6	7.09
Octamethyltrisiloxane	109.1	3.91	107.1	4.51	104.2	6.87	102.6	11.00
Octamethylcyclotetrasiloxane	107.7	3.79	105.8	6.82	101.6	3.09	101.7	5.93
1,1,1,3,5,5,5-heptamethyl-3-((trimethylsilyl)oxy)trisiloxane	101.0	3.17	102.3	3.25	95.5	5.78	99.8	3.26
Decamethyltetrasiloxane	101.8	3.28	102.4	3.28	97.8	4.19	98.8	3.02
Decamethylcyclopentasiloxane	109.4	4.04	106.9	5.34	104.8	5.25	104.5	5.71
Dodecamethylpentasiloxane	99.6	9.30	99.9	5.16	94.2	6.18	95.8	5.49
Dodecamethylcyclohexasiloxane	98.9	5.93	100.6	6.30	98.3	2.90	98.0	6.11

Uncertainty of Measurement:

Compound	Uncertainty of Measurement (%)
Hexamethyldisiloxane	26.20
1,1,1,3,5,5,5-heptamethyltrisiloxane	42.94
Octamethyltrisiloxane	16.92
Octamethylcyclotetrasiloxane	15.28
1,1,1,3,5,5,5-heptamethyl-3-((trimethylsilyl)oxy)trisiloxane	7.34
Decamethyltetrasiloxane	8.36
Decamethylcyclopentasiloxane	17.48
Dodecamethylpentasiloxane	19.00
Dodecamethylcyclohexasiloxane	12.96

References:

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