

METHOD STATEMENT



Determinand:

Taste and odour compounds

Matrix:

Sample Type: treated and raw water, i.e. waters that are abstracted for potable supply and potable waters.

Principle of Method:

Approximately 200mL of sample is extracted with 20mL of hexane. 6mL of the hexane solvent layer is transferred to a glass test tube and internal standard '2' is added. The sample extract is then concentrated to approximately 0.3mL and transferred to a 2mL auto-sampler vial containing a vial insert. The sample extract is further concentrated to 0.1mL and is then derivatized by adding 10µL of N-Methyl-N-(trimethylsilyl)trifluoroacetamide to convert the compounds to their corresponding trimethylsilyl (TMS) derivatives. The sample extract vial is capped ready for analysis.

Sampling and Sample Preparation:

Sampling, samples should be collected in 500 mL coloured glass which has been proven to be suitable for this analysis, with PTFE lined screw caps and contain 0.5 mL of sample preservative – sodium thiosulfate 1.8% w/v. Storage – samples should be analysed as soon as possible after collection. When this is not possible, they should be stored under refrigeration at 1-5°C in the dark, until analysis can begin. The maximum permissible storage time prior to analysis is given below.

Determinand	Maximum period of analyte stability prior to any extraction step (days)	Maximum period of analyte stability after to any extraction step (days)	Data is quoted from BS EN ISO 5667-3: 2003 ["ISO"] or ALS in-house data ["ALS-AS IHD"]
2-Bromophenol	28	N/A	ALS IHD
2,6-Dibromophenol	28	N/A	ALS IHD
2,4-Dibromophenol	28	N/A	ALS IHD
2,4,6-Tribromophenol	28	N/A	ALS IHD
2-Isopropyl-3-methoxypyrazine	28	N/A	ALS IHD
3-Chloroanisole	28	N/A	ALS IHD
4-Chloroanisole	28	N/A	ALS IHD
2-Chloroanisole	28	N/A	ALS IHD
2-Isobutyl-3-methoxypyrazine	28	N/A	ALS IHD
2-Methylisoborneol	28	N/A	ALS IHD
2,4,6-Trichloroanisole	28	N/A	ALS IHD
Geosmin	28	N/A	ALS IHD
2,3,4-Trichloroanisole	28	N/A	ALS IHD
2,4,6-Tribromoanisole	28	N/A	ALS IHD

Interferences

GC-MS/MS is an extremely selective technique and interferences should only be encountered very rarely. Any interfering compounds would have to display the identical MRM transition at the same retention time, this is extremely unlikely in potable water samples. However, any compound which passes through the extraction procedure and has a similar gas chromatographic retention time and mass spectrometric properties to the compound of interest will cause interference..

METHOD STATEMENT



Performance of Method:

Range of Application:

The operational range for each Taste and Odour compound is from the limit of detection Quantification to 75ng/l. Samples producing results above this range should be diluted and re-extracted.

Limit of Quantification, Recoveries of Compounds and Uncertainty of measurement:

AG1

Determinand	LOQ ng L ⁻¹	UoM	Direct Standards				Elvington Treated Water	
			Low Standard, 20%		High Standard, 80%		Spike, 80%	
			Recovery	RSD	Recovery	RSD	Recovery	RSD
2-Bromophenol	1	± 18.99 %	102.7%	5.7%	102.2%	5.5%	105.8%	6.6%
2,6-Dibromophenol	1	± 5.69 %	99.9%	1.5%	100.1%	0.8%	100.5%	1.0%
2,4-Dibromophenol	1	± 5.74 %	98.9%	1.4%	99.9%	0.7%	100.0%	1.1%
2,4,6-Tribromophenol	2	± 6.03 %	97.2%	1.9%	100.0%	1.5%	99.7%	1.1%
2-Isopropyl-3-methoxypyrazine	2	± 7.34 %	98.7%	3.0%	100.8%	3.2%	100.3%	2.6%
3-Chloroanisole	1	± 6.31 %	99.5%	1.4%	100.1%	0.9%	99.8%	1.5%
4-Chloroanisole	2	± 5.84 %	99.9%	1.2%	100.3%	1.0%	100.4%	1.5%
2-Chloroanisole	1	± 5.76 %	101.0%	2.5%	100.1%	0.8%	99.9%	1.0%
2-Isobutyl-3-methoxypyrazine	2	± 6.71 %	99.3%	1.5%	100.8%	1.6%	100.8%	2.1%
2-Methylisoborneol	2	± 8.49 %	100.1%	4.3%	100.7%	3.5%	101.4%	4.5%
2,4,6-Trichloroanisole	2	± 5.76 %	99.2%	1.3%	100.0%	0.9%	100.3%	1.1%
Geosmin	2	± 8.07 %	99.9%	3.2%	99.4%	2.5%	100.6%	4.8%
2,3,4-Trichloroanisole	1	± 12.71 %	99.9%	4.4%	100.4%	4.0%	98.1%	4.1%
2,4,6-Tribromoanisole	2	± 6.42 %	99.1%	1.7%	99.5%	1.9%	99.4%	1.6%

AG7

Determinand	LOQ (ng L ⁻¹)	UoM	Direct Standards				Elvington Treated Water	
			Low Standard, 20%		High Standard, 80%		Spike, 80%	
			Recovery	RSD	Recovery	RSD	Recovery	RSD
2-Bromophenol	1	±10.72%	102.82%±1.49%	2.86%	102.61%±1.50%	3.24%	100.69%±2.07%	4.32%
2,6-Dibromophenol	1	±15.43%	99.70%±0.47%	1.05%	99.69%±0.40%	0.84%	99.82%±0.89%	4.09%
2,4-Dibromophenol	1	±13.63%	98.70%±0.43%	0.71%	99.47%±0.45%	0.86%	99.20%±0.78%	3.72%
2,4,6-Tribromophenol	2	±17.24%	99.75%±0.42%	1.06%	100.07%±0.62%	1.24%	98.03%±2.03%	8.73%
2-Isopropyl-3-methoxypyrazine	3	±15.38%	98.30%±1.43%	4.97%	99.74%±1.50%	3.77%	100.43%±2.46%	5.17%
3-Chloroanisole	2	±9.07%	98.85%±0.60%	2.22%	99.72%±0.80%	1.77%	100.18%±0.92%	2.13%
4-Chloroanisole	1	±6.40%	99.87%±0.36%	0.96%	99.69%±0.43%	0.95%	99.53%±0.49%	1.09%
2-Chloroanisole	1	±6.08%	99.68%±0.42%	0.98%	99.92%±0.40%	0.92%	99.53%±0.51%	1.01%

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Determinand	LOQ (ng L ⁻¹)	UoM	Direct Standards				Elvington Treated Water	
			Low Standard, 20%		High Standard, 80%		Spike, 80%	
			Recovery	RSD	Recovery	RSD	Recovery	RSD
2-Isobutyl-3-methoxypyrazine	2	±9.94%	101.36%± 1.03%	2.14%	101.16%± 0.83%	1.63%	100.66%± 0.62%	1.56%
2-Methylisoborneol	2	±7.80%	100.80%± 0.62%	2.19%	100.86%± 0.63%	1.46%	99.95%± 0.85%	1.92%
2,4,6-Trichloroanisole	1	±6.96%	99.50%± 0.49%	1.12%	99.64%± 0.69%	1.32%	98.87%± 0.71%	1.41%
Geosmin	1	±6.54%	101.66%± 0.45%	1.16%	99.77%± 0.45%	0.99%	100.08%± 0.96%	2.17%
2,3,4-Trichloroanisole	1	±7.05%	100.08%± 0.96%	1.92%	99.33%± 1.11%	2.21%	99.17%± 1.18%	2.26%
2,4,6-Tribromoanisole	1	±6.95%	98.98%± 0.45%	1.15%	99.82%± 0.67%	1.24%	99.21%± 0.67%	1.34%

Determinand	AG1 Limit of Quantification (ng L ⁻¹)	AG7 Limit of Quantification (ng L ⁻¹)	METHOD (Standardised) Limit of Quantification (ng L ⁻¹)
2-Bromophenol	1	1	1
2,6-Dibromophenol	1	1	1
2,4-Dibromophenol	1	1	1
2,4,6-Tribromophenol	2	2	2
2-Isopropyl-3-methoxypyrazine	2	3	3
3-Chloroanisole	1	2	2
4-Chloroanisole	2	1	2
2-Chloroanisole	1	1	1
2-Isobutyl-3-methoxypyrazine	2	2	2
2-Methylisoborneol	2	2	2
2,4,6-Trichloroanisole	2	1	2
Geosmin	2	1	2
2,3,4-Trichloroanisole	1	1	1
2,4,6-Tribromoanisole	2	1	2

References:

In house Method