

METHOD STATEMENT



Determinand:

Taste and odour compounds

<u>Compounds</u>	<u>CAS Number</u>
2-Bromophenol	95-56-7
2,6-Dibromophenol	608-33-3
2,4-Dibromophenol	615-58-7
2,4,6-Tribromophenol	118-79-6
2-Isopropyl-3-methoxypyrazine	25773-40-4
3-Chloroanisole	2845-89-8
4-Chloroanisole	623-12-1
2-Chloroanisole	766-51-8
2-Isobutyl-3-methoxypyrazine	24683-00-9
2-Methylisoborneol	2371-42-8
2,4,6-Trichloroanisole	87-40-1
Geosmin	19700-21-1 (or 16423-19-1)
2,3,4-Trichloroanisole	54135-80-7
2,4,6-Tribromoanisole	607-99-8

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:

Samples are taken in 500 mL amber or green glass bottles containing 0.50 mL of sodium thiosulfate solution (1.8%, non-hazardous substance at this concentration) as preservative, designated as "T&O", and are stored at 1-5°C on receipt at the laboratory. Samples should be extracted within the number of days from sampling as stated below.

<u>Determinand</u>	<u>Maximum period of analyte stability prior to any extraction step (days)</u>	<u>Maximum period of analyte stability after to any extraction step (days)</u>	<u>Data is quoted from BS EN ISO 5667-3: 2003 ["ISO"] or ALS in-house data ["ALS-AS IHD"]</u>
2-Bromophenol	21	N/A	ALS IHD
2,6-Dibromophenol	21	N/A	ALS IHD
2,4-Dibromophenol	21	N/A	ALS IHD
2,4,6-Tribromophenol	21	N/A	ALS IHD
2-Isopropyl-3-methoxypyrazine	21	N/A	ALS IHD
3-Chloroanisole	21	N/A	ALS IHD
4-Chloroanisole	21	N/A	ALS IHD
2-Chloroanisole	21	N/A	ALS IHD
2-Isobutyl-3-methoxypyrazine	21	N/A	ALS IHD
2-Methylisoborneol	21	N/A	ALS IHD

METHOD STATEMENT



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"]
2,4,6-Trichloroanisole	21	N/A	ALS IHD
Geosmin	21	N/A	ALS IHD
2,3,4-Trichloroanisole	21	N/A	ALS IHD
2,4,6-Tribromoanisole	21	N/A	ALS IHD

Selected distribution/final treated water samples should be tested, at random, for levels of residual chlorine in order to confirm that bottles are continuing to be received with sodium thiosulfate having been present prior to sampling, according to WOP56.

Interferences

Any compound, which passes through the extraction procedure and that co-elutes with any of the analytes and produces a significant response to the relevant ions being monitored.

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:

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%

References:

In house Method