

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:

200 mL of sample is extracted with 20 mL of hexane. 6 mL of the hexane extraction solvent is transferred to a glass test tube and internal standard '2' is added. The sample extract is then concentrated to approximately 0.3 mL and transferred to a 2 mL auto-sampler vial containing a vial insert. The sample extract is further concentrated to 0.1 mL. 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.

<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-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

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

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.

Performance of Method:

Range of Application:

METHOD STATEMENT



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,

Determinand	AG1 (WPC66) Limit of Quantification (µg L⁻¹)	AG2 (WPC77) Limit of Quantification (µg L⁻¹)	Standardised Limit of Quantification (µg L⁻¹)
2-Isopropyl-3-methoxypyrazine	0.002	0.001	0.002
3-Chloroanisole	0.001	0.001	0.001
4-Chloroanisole	0.002	0.001	0.002
2-Chloroanisole	0.001	0.001	0.001
2-Isobutyl-3-methoxypyrazine	0.002	0.001	0.002
2-Methylisoborneol	0.002	0.002	0.002
2,4,6-Trichloroanisole	0.002	0.001	0.002
Geosmin	0.002	0.002	0.002
2,3,4-Trichloroanisole	0.001	0.001	0.001
2,3,4-Trichloroanisole	0.002	0.001	0.002

Recoveries of Compounds and Uncertainty of measurement:

Determinand	UoM	Direct Standards				Elvington Treated	
		Low Standard, 20%		High Standard, 80%		Spike, 80%	
		Recovery	RSD	Recovery	RSD	Recovery	RSD
2-Isopropyl-3-methoxypyrazine	8.181%	99.3%	3.53%	99.1%	2.45%	99.9%	2.56%
3-Chloroanisole	8.948%	97.6%	2.49%	98.4%	1.65%	99.4%	1.95%
4-Chloroanisole	8.593%	97.9%	2.76%	98.5%	1.65%	99.3%	1.96%
2-Chloroanisole	8.032%	97.9%	3.04%	98.5%	1.80%	99.0%	2.02%
2-Isobutyl-3-methoxypyrazine	7.733%	98.3%	2.83%	98.6%	1.81%	99.5%	2.08%
2-Methylisoborneol	9.549%	100.1%	3.71%	98.7%	2.10%	100.8%	3.34%
2,4,6-Trichloroanisole	9.821%	98.2%	3.10%	98.1%	2.33%	99.0%	2.64%
Geosmin	14.876%	97.4%	5.49%	98.7%	5.65%	98.6%	3.66%
2,3,4-Trichloroanisole	12.016%	98.2%	5.31%	99.1%	3.17%	99.6%	6.05%
2,4,6-Tribromoanisole	10.466%	95.7%	3.16%	98.1%	1.88%	98.3%	2.76%

References:

In house Method