

# METHOD STATEMENT



## Determinand:

Methanol, Ethanol, Acetone, Propan-2-ol (IPA), Acetonitrile, Dichloromethane, Propan-1-ol (n-Propanol), Butan-2-one (MEK), Ethyl acetate, Butan-2-ol, Isobutanol, Butan-1-ol, 4 Methyl-2-pentanone (MIBK).

## Matrix:

Treated Sewage Effluent, Untreated Sewage Effluent and Trade effluent.

## Principle of Method:

The sample is placed in a headspace vial and allowed to equilibrate with its headspace vapour at 90°C. A sample of the vapour is injected using an automatic headspace sampler into a capillary column gas chromatograph fitted with a mass spectrum detector.

## Sampling and Sample Preparation:

Water samples are to be supplied in 40ml screw top glass vials. They must be taken without any significant headspace (when vial is inverted, air bubble no more than 6mm diameter).

Samples should be preserved with HCl.

Samples are stored prior to analysis in a fridge at  $3 \pm 2^\circ\text{C}$ .

Acetone and IPA are stable for 30 days (in-house data) from sampling.

## Interferences

Any compound extractable into the headspace, which then elutes chromatographically at a similar retention time to the target compounds and elicits a detector response, may interfere.

Bottles of pure compounds must not be opened in the sample preparation area to avoid contamination.

## Performance of Method:

LOD, Precision and Bias

Compound	LOD	MRL	Low Standard		High Standard	
	mg/L	mg/L	% Bias	% RSD	% Bias	% RSD
Methanol	0.685	5.0	6.48	6.29	-0.20	4.77
Ethanol	0.449	5.0	6.04	6.17	0.69	4.45
Acetone	0.060	1.0	-2.43	3.42	1.93	3.33
Propan-2-ol	0.352	1.0	-3.63	6.04	0.06	3.70
Acetonitrile	0.212	1.0	3.80	4.44	2.12	4.34
Dichloromethane	0.035	0.2	-3.41	6.27	1.69	6.17
Propan-1-ol	0.122	1.0	6.05	6.54	1.28	2.82
Butan-2-one (MEK)	0.068	0.5	-0.36	3.74	1.96	4.72
Ethyl Acetate	0.153	0.5	-6.96	16.07	-4.55	7.88
Butan-2-ol	0.109	1.0	3.88	5.04	2.82	3.60
Isobutanol	0.150	1.0	3.30	4.98	3.13	3.54
Butan-1-ol	0.178	1.0	3.79	4.96	1.60	3.31
4-methyl-2-pentanone (MIBK)	0.067	0.5	-4.10	4.85	3.10	5.67

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## Matrix Spike Recoveries

Compound	Untreated Sewage		Treated Sewage		Trade Effluent	
	% Rec.	% RSD	% Rec.	% RSD	% Rec.	% RSD
Methanol	100.7	4.64	100.7	5.57	99.9	4.34
Ethanol	102.1	4.26	102.1	5.22	101.2	4.44
Acetone	102.3	2.58	102.9	5.02	101.9	3.19
Propan-2-ol	99.0	3.00	99.9	3.33	99.7	4.07
Acetonitrile	100.4	4.04	102.4	5.98	102.4	5.57
Dichloromethane	99.3	6.07	99.3	7.18	100.1	6.33
Propan-1-ol	102.9	3.09	102.9	4.33	102.6	3.84
Butan-2-one (MEK)	101.5	3.78	102.4	4.38	102.9	3.52
Ethyl Acetate	99.5	7.62	103.9	5.32	95.2	8.24
Butan-2-ol	103.8	3.30	103.7	4.17	103.8	3.86
Isobutanol	103.5	3.48	103.8	3.99	103.7	4.25
Butan-1-ol	103.0	2.66	102.5	3.94	102.2	3.92
4-methyl-2-pentanone (MIBK)	102.0	4.23	101.8	4.03	102.6	4.34

## Uncertainty of Measurement:

Compound	Uncertainty of Measurement %
Methanol	11.87
Ethanol	12.19
Acetone	16.39
Propan-2-ol	8.85
Acetonitrile	14.84
Dichloromethane	16.26
Propan-1-ol	12.02
Butan-2-one (MEK)	13.32
Ethyl Acetate	17.22
Butan-2-ol	12.68
Isobutanol	13.62
Butan-1-ol	17.46
4-Methyl-2-Pentanone (MIBK)	12.44

## References:

In-house developed method based on: - Determination of very low concentrations of hydrocarbons and halogenated hydrocarbons in water 1984-5. Methods for the Examination of Waters and Associated Materials, HMSO ISBN 0 11 752004 7.

EPA Method 5021A : Volatile Organic Compounds in Various Sample Matrices Using Equilibrium Headspace Analysis