

METHOD STATEMENT

Determinand:

The analysis of Trichloromethane, Bromodichloromethane, Dibromochloromethane, Tribromomethane, Tetrachloromethane, Trichloroethene, Tetrachloroethene, 1,1,1-Trichloroethane, 1,2-Dichloroethane, 1,2-Dibromoethane, MethylTertiary Butyl Ether (MTBE), TertAmylMethylEther (TAME), 1,1-Dichloroethane, Dichloromethane, 1,4-DichloroBenzene, Benzene, Toluene, Ethylbenzene and Xylenes (BTEX compounds).

Matrix:

Sample Type: Raw and Potable waters

Principle of Method:

The water sample is placed in a septum vial and allowed to equilibrate with its headspace vapour at 60°C. A sample of the vapour is injected using an automatic headspace sampler into a capillary column gas chromatograph (GC), the volatile organic compounds are separated and then identified and quantified with mass spectrometric detection (MSD) in selected ion monitoring (SIM) mode.

Interferences:

Any compound, which passes through the extraction procedure, and has similar Gas Chromatographic and mass spectrometric properties to the analyte will cause interference. No bottles of reference materials are to be opened, and no stock, intermediate or spiking solution preparation is to be carried out in the Wakefield VOC Laboratory.

Performance of Method:

Range of Application:

Headspace Twister instruments Determinands	Operational Range
Trichloromethane	LOD - 200 µg/l
Bromodichloromethane	LOD - 100 µg/l
Dibromochloromethane	LOD - 100 µg/l
Tribromomethane	LOD - 100 µg/l
Tetrachloromethane	LOD - 10 µg/l
Trichloroethene	LOD - 50 µg/l
Tetrachloroethene	LOD - 20 µg/l
1,1,1 - Trichloroethane	LOD - 20 µg/l
1,2 - Dichloroethane	LOD - 20 µg/l
1,2 - Dibromoethane	LOD - 50 µg/l
MethylTertiary Butyl Ether (MTBE)	LOD - 50 µg/l
TertAmylMethylEther (TAME)	LOD - 50 µg/l
Benzene	LOD - 5 µg/l
Ethylbenzene	LOD - 20 µg/l
Toluene	LOD - 20 µg/l
Para-Xylene	N/A
Meta-Xylene	N/A
Sum of Meta-Xylene + Para-Xylene	LOD - 40 µg/l
Ortho-Xylene	LOD - 20 µg/l
1,1-Dichloroethane	LOD - 20 µg/l
Dichloromethane	LOD - 20 µg/l
1,4-DichloroBenzene	LOD - 20 µg/l



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Reporting Limits are the same as the LOD

Limit of Detection:

Headspace instrument

<u>Compound</u>	<u>Limit of Detection µg/l</u>
Trichloromethane	0.30 µg/l
Bromodichloromethane	0.06 µg/l
Dibromochloromethane	0.09 µg/l
Tribromomethane	0.15 µg/l
Tetrachloromethane	0.02 µg/l
Trichloroethene	0.05 µg/l
Tetrachloroethene	0.05 µg/l
1,1,1 - Trichloroethane	0.04 µg/l
1,2 - Dichloroethane	0.07 µg/l
1,2 - Dibromoethane	0.09 µg/l
MethylTertiary Butyl Ether (MTBE)	0.06 µg/l
TertAmylMethylEther (TAME)	0.05 µg/l
Benzene	0.02 µg/l
Ethylbenzene	0.03 µg/l
Toluene	0.03 µg/l
Meta-Xylene + Para-Xylene	0.05 µg/l
Ortho-Xylene	0.04 µg/l
1,1-Dichloroethane	0.03 µg/l
Dichloromethane	0.58 µg/l
1,4-DichloroBenzene	0.12 µg/l

Twister instrument

<u>Compound</u>	<u>Limit of Detection µg/l</u>
Trichloromethane	0.29 µg/l
Bromodichloromethane	0.27 µg/l
Dibromochloromethane	0.25 µg/l
Tribromomethane	0.30 µg/l
Tetrachloromethane	0.14 µg/l
Trichloroethene	0.34 µg/l
Tetrachloroethene	0.39 µg/l
1,1,1 - Trichloroethane	0.18 µg/l
1,2 - Dichloroethane	0.10 µg/l
1,2 - Dibromoethane	0.21 µg/l
MethylTertiary Butyl Ether (MTBE)	0.18 µg/l
TertAmylMethylEther (TAME)	0.31 µg/l
Benzene	0.07 µg/l
Ethylbenzene	0.16 µg/l
Toluene	0.16 µg/l
Meta-Xylene + Para-Xylene	0.32 µg/l
Ortho-Xylene	0.15 µg/l
1,1-Dichloroethane	0.20 µg/l



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<u>Compound</u>	<u>Limit of Detection µg/l</u>
Dichloromethane	0.02 µg/l
1,4-DichloroBenzene	0.04 µg/l

Recoveries of Compounds

Recoveries from spiked solutions of Elvington water at AQC level for the Headspace instrument

<u>Compound</u>	<u>% Recovery</u>
Trichloromethane	92.91
Bromodichloromethane	100.34
Dibromochloromethane	98.75
Tribromomethane	108.63
Tetrachloromethane	103.31
Trichloroethene	96.97
Tetrachloroethene	98.16
1,1,1 - Trichloroethane	93.90
1,2 - Dichloroethane	105.50
1,2 - Dibromoethane	99.13
MethylTertiary Butyl Ether (MTBE)	98.15
TertAmylMethylEther (TAME)	96.15
Benzene	102.49
Ethylbenzene	101.56
Toluene	99.31
Meta-Xylene + Para-Xylene	99.19
Ortho-Xylene	101.15
1,1-Dichloroethane	94.69
Dichloromethane	89.05
1,4-DichloroBenzene	100.81

Recoveries from spiked solutions of Elvington water at AQC level for the Twister instrument

<u>Compound</u>	<u>% Recovery</u>
Trichloromethane	99.0
Bromodichloromethane	94.2
Dibromochloromethane	90.0
Tribromomethane	96.6
Tetrachloromethane	95.8
Trichloroethene	97.9
Tetrachloroethene	94.9
1,1,1 - Trichloroethane	96.3
1,2 - Dichloroethane	103.6
1,2 - Dibromoethane	98.2
MethylTertiary Butyl Ether (MTBE)	104.5
TertAmylMethylEther (TAME)	99.2
Benzene	97.2
Ethylbenzene	96.2
Toluene	98.6
Meta-Xylene + Para-Xylene	95.6
Ortho-Xylene	96.4



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<u>Compound</u>	<u>% Recovery</u>
1,1-Dichloroethane	103.8
Dichloromethane	100.5
1,4-DichloroBenzene	102.7

Uncertainty of Measurement

Uncertainty of measurement of spiked solutions of Elvington water at AQC level for the Headspace instrument.

<u>Compound</u>	<u>Uncertainty %</u>
Trichloromethane	± 11.99%
Bromodichloromethane	± 6.60%
Dibromochloromethane	± 8.39%
Tribromomethane	± 17.62%
Tetrachloromethane	± 11.96%
Trichloroethene	± 10.93%
Tetrachloroethene	± 5.90%
1,1,1 - Trichloroethane	± 13.31%
1,2 - Dichloroethane	± 9.68%
1,2 - Dibromoethane	± 7.09%
MethylTertiary Butyl Ether (MTBE)	± 6.95%
TertAmylMethylEther (TAME)	± 8.40%
Benzene	± 7.52%
Ethylbenzene	± 3.84%
Toluene	± 2.66%
Meta-Xylene + Para-Xylene	± 3.05%
Ortho-Xylene	± 5.27%
1,1-Dichloroethane	± 12.61%
Dichloromethane	± 17.09%
1,4-DichloroBenzene	± 10.97%

Uncertainty of measurement of spiked solutions of Elvington water at AQC level for the Twister instrument.

<u>Compound</u>	<u>Uncertainty %</u>
Trichloromethane	± 12.21%
Bromodichloromethane	± 17.76%
Dibromochloromethane	± 22.35%
Tribromomethane	± 21.88%
Tetrachloromethane	± 25.91%
Trichloroethene	± 21.17%
Tetrachloroethene	± 24.53%
1,1,1 - Trichloroethane	± 24.25%
1,2 - Dichloroethane	± 9.46%
1,2 - Dibromoethane	± 13.48%
MethylTertiary Butyl Ether (MTBE)	± 8.75%
TertAmylMethylEther (TAME)	± 13.33%
Benzene	± 14.54%



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<u>Compound</u>	<u>Uncertainty %</u>
Ethylbenzene	± 14.75%
Toluene	± 11.31%
Meta-Xylene + Para-Xylene	± 15.39%
Ortho-Xylene	± 14.50%
1,1-Dichloroethane	± 12.10%
Dichloromethane	± 7.72%
1,4-DichloroBenzene	± 11.60%

References:

In house method.

