

METHOD STATEMENT



Determinand:

BTEX compounds, Trihalomethanes, Chlorinated Solvents, MTBE and Styrene

Matrix:

Surface Waters, Groundwaters, Effluents, Leachates and Estuarine Waters

Principle of Method:

Headspace extraction of the compounds from the sample using a commercial headspace sampler followed by separation and quantitative determination of the compounds using a gas chromatograph with mass spectrometric detection (GCMS).

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±2°C.

Samples are stable for 14 days (US EPA Method 8260) from sampling.

Interferences:

Any compound extractable into the headspace, which then elutes at a similar retention time to the target compounds and also possesses responses to the ions monitored.

Performance of Method:

Range of Application

Reporting Limit to 50µg/l without dilution

Determinand	CAS No.	LOD µg/l	MRL µg/l	Mcerts Accred.	Low Std		High Std	
					%Bias	%RSD	%Bias	%RSD
MTBE	1634-04-4	0.066	0.10		-1.50	2.00	-0.40	1.70
Chloroform	67-66-3	0.097	0.10	✓	-0.20	4.20	-0.40	3.80
1,1,1-TCE	71-55-6	0.082	0.10		1.80	10.90	-1.30	8.70
1,2-DCE	107-06-2	0.096	1.00		0.70	3.20	-2.30	1.40
Carbon Tetrachloride	56-23-5	0.070	0.10		1.00	11.80	-2.80	10.50
Benzene	71-43-2	0.052	0.10	✓	-2.30	5.10	-2.60	5.20
Trichloroethene	79-01-6	0.072	0.10	✓	-4.00	7.40	-3.50	6.90
Bromodichloromethane	75-27-4	0.085	0.10		-3.00	2.20	-1.80	2.30
Toluene	108-88-3	0.072	0.10		-5.50	5.50	-3.10	5.70
1,1,2-Trichloroethane	79-00-5	0.092	0.50		-1.90	2.10	-1.80	1.60
Tetrachloroethylene	127-18-4	0.099	0.10		-2.70	9.40	-4.40	8.40
Dibromochloromethane	124-48-1	0.097	0.10		-5.30	2.70	-2.40	2.20
Ethyl Benzene	100-41-4	0.095	0.10		-6.40	6.10	-4.10	6.50
m&p-Xylene	1330-20-7	0.195	0.20		-5.30	6.20	-3.70	6.60
o-Xylene	95-47-6	0.090	0.10		-6.20	4.60	-2.60	4.80
Styrene	100-42-5	0.097	0.10		-11.40	3.40	-2.50	4.00
Bromoform	75-25-2	0.096	0.10		-7.50	2.60	-2.30	2.10
1,1,2,2-Tetrachloroethane	79-34-5	0.086	0.10		-3.50	3.40	-1.50	2.70

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20% Spiked Samples

Determinand	Finham FE		Trade Effluent		Wolston FE	
	%Rec.	%RSD	%Rec.	%RSD	%Rec.	%RSD
MTBE	96.70	3.90	97.20	2.10	99.50	4.00
Chloroform	95.90	9.40	96.00	4.60	100.60	6.20
1,1,1-Trichloroethane	99.10	14.40	96.90	12.90	92.00	13.70
1,2-Dichloroethane	97.50	2.50	99.50	3.30	104.40	6.00
Carbon Tetrachloride	98.20	14.40	95.40	13.70	88.70	10.20
Benzene	96.80	9.60	98.60	6.80	98.80	6.50
Trichloroethene	97.90	11.00	95.70	8.70	96.30	6.30
Bromodichloromethane	96.30	4.40	97.20	4.30	101.40	6.30
Toluene	94.10	9.70	96.60	7.40	89.30	7.00
1,1,2-Trichloroethane	98.40	2.70	101.90	3.40	104.40	5.00
Tetrachloroethene	98.10	11.90	95.60	11.80	94.10	9.70
Dibromochloromethane	94.90	2.90	97.10	4.30	103.30	4.30
Ethyl Benzene	94.00	10.00	96.40	8.20	90.20	7.40
m & p-Xylene	94.90	9.90	97.40	8.40	99.70	5.10
o-Xylene	93.10	8.70	94.60	6.40	85.80	7.90
Styrene	88.50	7.20	90.90	4.70	94.80	4.40
Bromoform	92.70	3.00	95.80	4.30	108.70	4.90
1,1,2,2-Tetrachloroethane	93.10	3.80	100.80	3.30	99.20	6.10

Determinand	Groundwater		Landfill Leachate		Soil Leachate		Surface Water	
	%Rec.	%RSD	%Rec.	%RSD	%Rec.	%RSD	%Rec.	%RSD
MTBE	97.60	1.50	97.50	1.80	104.00	4.00	98.06	2.59
Chloroform	98.20	2.80	97.30	3.50	99.90	3.40	98.32	4.63
1,1,1-Trichloroethane	101.50	9.90	99.50	8.10	92.40	6.30	101.99	12.13
1,2-Dichloroethane	98.00	2.40	98.00	3.20	107.10	7.50	97.96	3.68
Carbon Tetrachloride	99.90	10.40	98.50	8.70	93.70	9.90	100.60	12.70
Benzene	98.70	3.50	97.90	5.40	104.40	4.60	98.43	5.71
Trichloroethene	97.00	5.30	98.20	6.50	100.10	4.70	98.62	7.25
Bromodichloromethane	97.00	2.10	97.00	2.80	105.70	5.10	97.04	3.27
Toluene	95.90	3.80	95.00	5.40	102.30	5.90	95.94	5.79
1,1,2-Trichloroethane	99.80	1.70	98.80	2.80	108.90	6.90	98.91	3.30
Tetrachloroethene	98.90	9.00	98.10	7.30	94.70	5.70	100.50	10.04
Dibromochloromethane	95.20	2.10	95.10	3.20	105.10	7.20	95.40	4.02
Ethyl Benzene	95.60	4.10	94.90	6.20	102.20	5.80	95.70	6.22
m & p-Xylene	96.40	5.00	95.80	6.60	102.00	6.50	96.90	6.99
o-Xylene	94.70	3.40	93.80	4.90	101.50	5.50	94.85	4.94
Styrene	89.50	2.90	88.80	3.90	99.10	7.40	89.65	3.99
Bromoform	92.50	2.50	92.50	3.40	102.30	7.20	92.73	4.44
1,1,2,2-Tetrachloroethane	96.30	3.10	93.90	4.50	106.10	7.60	94.82	4.18

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80% Spiked Samples

Determinand	Finham FE		Trade Effluent		Wolston FE	
	%Rec.	%RSD	%Rec.	%RSD	%Rec.	%RSD
MTBE	99.60	2.00	99.60	1.70	100.40	2.90
Chloroform	98.50	3.20	98.20	3.20	96.40	3.50
1,1,1-Trichloroethane	96.30	6.70	95.40	7.20	92.30	8.30
1,2-Dichloroethane	98.00	1.90	100.20	3.00	101.50	5.40
Carbon Tetrachloride	95.20	7.70	95.10	6.80	95.10	9.00
Benzene	97.70	4.20	100.40	3.80	100.20	6.50
Trichloroethene	99.20	5.00	98.10	5.10	100.20	6.40
Bromodichloromethane	99.00	2.40	100.00	3.10	101.60	5.60
Toluene	96.90	4.70	100.50	4.60	97.10	4.80
1,1,2-Trichloroethane	99.30	1.40	103.10	3.40	100.60	4.40
Tetrachloroethene	95.20	6.80	95.30	5.80	93.40	6.00
Dibromochloromethane	99.00	2.00	101.60	3.80	99.40	4.20
Ethyl Benzene	95.80	5.00	100.20	5.10	96.60	4.60
m & p-Xylene	96.30	5.50	100.20	5.60	98.40	4.30
o-Xylene	97.10	4.00	99.60	4.30	96.80	4.10
Styrene	97.80	3.40	101.20	3.90	98.60	4.70
Bromoform	98.70	2.10	102.60	3.90	99.20	4.00
1,1,2,2-Tetrachloroethane	95.40	3.00	104.80	4.10	97.80	5.80

Determinand	Groundwater		Landfill Leachate		Soil Leachate		Surface Water	
	%Rec.	%RSD	%Rec.	%RSD	%Rec.	%RSD	%Rec.	%RSD
MTBE	98.80	1.40	99.20	2.00	99.20	2.80	99.64	1.43
Chloroform	97.30	2.60	97.10	4.80	95.10	5.00	99.03	2.59
1,1,1-Trichloroethane	94.30	7.40	93.20	10.80	89.10	10.50	98.00	6.03
1,2-Dichloroethane	98.00	1.90	98.40	1.80	99.80	6.60	98.48	1.61
Carbon Tetrachloride	93.60	9.00	92.30	11.80	89.60	14.30	97.77	6.61
Benzene	97.00	4.00	96.60	5.80	97.80	5.90	98.84	3.14
Trichloroethene	95.60	5.50	96.80	6.90	94.30	7.40	99.68	4.43
Bromodichloromethane	98.90	2.00	99.20	2.40	100.70	4.80	99.63	1.86
Toluene	96.50	4.50	95.50	7.00	97.50	6.20	98.38	3.90
1,1,2-Trichloroethane	100.00	2.30	99.90	2.00	103.50	4.80	99.95	1.72
Tetrachloroethene	93.80	7.60	92.70	10.00	91.00	8.70	97.90	5.60
Dibromochloromethane	99.30	3.00	99.30	2.60	102.10	5.20	99.76	2.38
Ethyl Benzene	95.60	5.00	94.70	7.60	97.10	6.10	97.49	4.00
m & p-Xylene	95.90	5.70	94.70	7.80	97.10	7.00	98.61	4.83
o-Xylene	96.90	3.90	96.10	5.70	97.40	5.20	98.74	3.49
Styrene	97.70	3.40	97.20	4.50	98.70	4.80	99.38	2.97
Bromoform	98.90	3.00	99.30	2.50	100.90	4.40	99.63	2.49
1,1,2,2-Tetrachloroethane	98.90	3.40	96.60	3.40	102.00	4.40	97.76	2.98

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Uncertainty of Measurement

The reported uncertainty is an expanded uncertainty calculated using a coverage factor of 2, which gives a level of confidence of approximately 95%.

Determinand	Uncertainty of Measurement %	Determinand	Uncertainty of Measurement %
1,1,1-Trichloroethane	23.22	Dibromochloromethane	10.83
1,1,2,2-Tetrachloroethane	9.64	Ethyl Benzene	12.50
1,1,2-Trichloroethane	9.57	m & p-Xylene	14.95
1,2-Dichloroethane	10.28	MTBE	7.24
Benzene	11.43	o-Xylene	11.94
Bromodichloromethane	10.30	Styrene	18.49
Bromoform	11.11	Tetrachloroethene	22.58
Carbon Tetrachloride	27.40	Toluene	11.78
Chloroform	13.01	Trichloroethene	16.85

References:

Based on modified version of EPA 524 for volatiles.

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