

METHOD STATEMENT



Determinand:

Volatile Petroleum Hydrocarbons (>C5 to C10) This includes BTEX and MTBE

Matrix:

Groundwaters, Effluents, Leachates and Estuarine Waters.

Principle of Method:

Headspace extraction of the volatile hydrocarbons from aqueous samples using a commercial headspace autosampler followed by gas chromatography (GC). The effluent from the chromatographic column is split between a flame ionisation detector (FID) for the determination of total volatile petroleum hydrocarbons and a mass selective detector (MSD) for the determination of aromatic volatile hydrocarbons.

Sampling and Sample Preparation:

Water samples are to be supplied in 40ml screw top glass vials. They must be taken without any significant headspace. Samples should be preserved with HCl.

Samples are stored prior to analysis in a fridge at 5±3°C.

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

Interferences:

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

Performance of Method:

Reporting Level: 10µg/l

Determinand	L.O.D ug/l	Low Std		High Std	
		% Bias	% RSD	% Bias	% RSD
Total VPH >C5-C10	8.9734	-	6.96	-	4.96
>C5-6 ALI	7.1552	-	8.58	-	6.33
>C6-8 ALI	5.0651	-	13.12	-	13.47
>C8-10 ALI	5.5305	-	12.15	-	18.58
>C5-C10 ALI	7.3017	-	10.14	-	11.12
>C5-C7 ARO	2.3697	-	7.34	-	7.09
>C7-8 ARO	1.5513	-	7.03	-	5.74
>C8-10 ARO	3.4815	-	7.25	-	4.68
>C5-10 ARO	6.4156	-	7.15	-	4.97
Total VPH C4-C12	18.4856	1.97	6.89	1.37	5.50
MTBE	2.3404	-3.78	7.62	-0.50	12.67
Benzene	2.3697	-4.84	7.34	-3.52	7.09
Toluene	1.5513	-6.73	7.03	-3.79	5.74
Ethylbenzene	1.1405	-7.39	8.47	-5.19	5.05
M/P-Xylene	2.0723	-5.58	7.27	-5.56	4.82
O-Xylene	0.9864	-5.43	6.27	-4.41	4.62



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Determinand	Groundwater		Surface Water		Final Effluent	
	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD
Total VPH >C5-C10	-	5.47	-	11.12	-	5.47
>C5-6 ALI	-	4.43	-	11.25	-	5.84
>C6-8 ALI	-	6.47	-	12.03	-	6.49
>C8-10 ALI	-	18.69	-	18.88	-	19.14
>C5-C10 ALI	-	7.06	-	11.99	-	7.63
>C5-C7 ARO	-	7.85	-	12.59	-	7.58
>C7-8 ARO	-	7.60	-	12.14	-	6.85
>C8-10 ARO	-	6.76	-	11.94	-	6.45
>C5-10 ARO	-	6.73	-	11.91	-	6.43
Total VPH C4-C12	91.27	5.10	91.15	11.01	90.29	5.56
MTBE	60.80	11.80	59.66	14.88	58.51	9.25
Benzene	85.97	7.85	84.25	12.59	83.39	7.58
Toluene	104.62	7.60	103.49	12.14	102.78	6.85
Ethylbenzene	89.61	8.03	88.71	12.99	88.44	7.31
M/P-Xylene	87.53	6.02	86.94	11.30	86.52	5.61
O-Xylene	79.74	6.54	79.10	11.82	78.23	6.38

Determinand	Trade Effluent		Soil Leachate		Landfill Leachate	
	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD
Total VPH >C5-C10	-	5.06	-	6.08	-	5.89
>C5-6 ALI	-	6.08	-	7.13	-	7.38
>C6-8 ALI	-	7.34	-	7.54	-	7.49
>C8-10 ALI	-	15.92	-	19.16	-	17.99
>C5-C10 ALI	-	8.11	-	8.45	-	8.38
>C5-C7 ARO	-	7.23	-	8.62	-	7.74
>C7-8 ARO	-	6.16	-	7.66	-	7.14
>C8-10 ARO	-	5.64	-	6.94	-	6.33
>C5-10 ARO	-	5.63	-	7.05	-	6.43
Total VPH C4-C12	90.75	4.51	90.43	5.94	84.98	6.05
MTBE	58.82	9.01	57.82	10.63	58.65	8.45
Benzene	84.56	7.23	83.27	8.62	80.80	7.74
Toluene	104.21	6.16	102.50	7.66	99.88	7.14
Ethylbenzene	88.85	6.44	88.27	8.10	85.69	6.98
M/P-Xylene	87.59	5.41	86.50	6.10	83.78	5.48
O-Xylene	78.77	5.62	78.03	6.44	75.65	5.28



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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%.

The uncertainty contribution for bias from the total VPH >C5-C10 has been used to calculate the UoM for the fractioned bands.

Determinand	Uncertainty of Measurement %
Total VPH >C5-C10	25.21
Aliphatic VPH >C5-C6	25.68
Aliphatic VPH >C6-C8	25.72
Aliphatic VPH >C8-C10	44.54
Aliphatic VPH >C5-C10	26.29
Aromatic VPH >C5-C7	27.31
Aromatic VPH >C7-C8	24.58
Aromatic VPH >C8-C10	24.40
Aromatic VPH >C5-C10	24.34

References:

Based on modified version of EPA 524 for volatiles.

