

METHOD STATEMENT



Determinand:

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

Matrix:

Groundwaters, Surface Waters, Trade Effluents, and Landfill Leachates.

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 (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 $3\pm 2^{\circ}\text{C}$.

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:

Determinand	L.O.D ug/l	Low Std		High Std	
		% Bias	% RSD	% Bias	% RSD
Total VPH >C5-C10	5.57	0.39	1.49	0.03	1.25
>C5-6 ALI	1.05	-4.73	3.61	-0.98	1.87
>C6-8 ALI	1.98	1.35	2.45	-0.39	2.13
>C8-10 ALI	4.07	9.82	2.80	-1.60	6.35
>C5-C10 ALI	5.35	2.96	2.54	-1.03	3.17
>C5-C7 ARO	0.065	-0.61	5.19	2.22	4.18
>C7-8 ARO	0.258	-5.83	3.99	1.34	2.16
>C8-10 ARO	0.703	-6.11	2.81	2.61	4.74
>C5-10 ARO	0.957	-5.39	2.90	2.40	4.28
Total VPH C4-C12	9.15	1.16	1.44	0.08	1.25
Benzene	0.065	-0.61	5.19	2.22	4.18
Toluene	0.258	-5.83	3.99	1.34	2.16
Ethylbenzene	0.103	-4.12	2.32	1.85	3.32
M/P-Xylene	0.212	-3.34	4.45	3.72	10.1
O-Xylene	0.101	-8.09	3.82	0.74	2.25

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Determinand	Groundwater		Surface Water		Land Leachate		Trade Effluent	
	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
Total VPH >C5-C10	****	2.23	****	2.33	****	1.81	****	1.85
>C5-6 ALI	****	3.19	****	3.94	****	3.22	****	3.67
>C6-8 ALI	****	2.58	****	2.82	****	2.70	****	2.36
>C8-10 ALI	****	18.9	****	24.2	****	15.8	****	10.18
>C5-C10 ALI	****	3.41	****	4.34	****	3.50	****	2.80
>C5-C7 ARO	****	4.63	****	5.07	****	6.38	****	5.51
>C7-8 ARO	****	1.88	****	1.86	****	2.49	****	2.07
>C8-10 ARO	****	2.91	****	2.89	****	2.63	****	2.94
>C5-10 ARO	****	2.38	****	2.37	****	2.42	****	2.44
Total VPH C4-C12	88.0	3.14	88.6	3.58	88.2	2.71	87.6	2.62
Benzene	****	4.63	****	5.07	****	6.38	****	5.51
Toluene	****	1.88	****	1.86	****	2.49	****	2.04
Ethylbenzene	****	4.34	****	4.11	****	4.54	****	4.36
M/P-Xylene	****	3.20	****	2.89	****	2.82	****	2.87
O-Xylene	****	2.09	****	1.93	****	2.01	****	2.14

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 >C4-C12 Gasoline (7.72%) has been used to calculate the UoM for the fractionated bands.

Determinand	Uncertainty of Measurement %
Total VPH >C5-C10	16.57
Aliphatic VPH >C5-C6	17.23
Aliphatic VPH >C6-C8	17.36
Aliphatic VPH >C8-C10	46.15
Aliphatic VPH >C5-C10	17.89
Aromatic VPH >C5-C7	19.01
Aromatic VPH >C7-C8	16.16
Aromatic VPH >C8-C10	16.91
Aromatic VPH >C5-C10	16.36

References:

In house developed method

Chapter Four of the SW-846 Compendium: Organics Analytes. Revision 6 December 2018

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