

METHOD STATEMENT



Determinand:

PAH compounds (EPA16) in environmental waters

Matrix:

Surface Water, Land Leachate, Groundwater, Untreated Sewage, Treated Sewage and Trade Effluent.

Principle of Method:

An automated dispersive liquid/liquid microextraction procedure (DLLME) is used to extract the PAH compounds from aqueous matrix. The dispersive solvent is a 1% solution of toluene in isopropanol and the extraction solvent a mixture of dichloromethane and n-pentane (80:20). The extract is then quantified by gas chromatography with mass spectrometric detection (GCMS).

Sampling and Sample Preparation:

Water samples are to be supplied in 40ml amber screw top glass vials.

They must be taken without any significant headspace - that is filled at least up to the shoulder of the vial - and delivered to the laboratory.

Samples should be stored at $3 \pm 2^\circ\text{C}$.

Extracts must be stored at room temperature, if they cannot be analysed on the day that they are vialled.

Samples must be allowed to warm to room temperature overnight before extraction.

Samples are stable for 7 days (Ref - US EPA SW-846 Chapter four) from sampling

Interferences:

Any co-extracted material with a corresponding GC retention time and similar mass spectrum will interfere.

Performance of Method:

Precision, Bias and Limit of Detection

Compound	LOD ($\mu\text{g/L}$)	Low Standard		High Standard	
		Bias	RSD	Bias	RSD
Naphthalene	0.00670	2.46%	0.91%	-3.06%	0.40%
Acenaphthylene	0.00120	-3.52%	0.56%	3.12%	0.85%
Acenaphthene	0.00390	3.84%	1.92%	-2.67%	1.01%
Fluorene	0.00212	4.91%	1.36%	-2.32%	0.93%
Phenanthrene	0.00064	2.59%	1.02%	-3.39%	0.90%
Anthracene	0.00899	3.78%	3.98%	-3.70%	2.98%
Fluoranthene	0.00095	3.29%	0.85%	-3.07%	1.06%
Pyrene	0.00101	3.49%	0.93%	-3.48%	1.10%
Benzo(a)anthracene	0.00137	4.63%	3.80%	-3.74%	5.04%
Chrysene	0.00162	8.36%	3.18%	-1.24%	2.02%
Benzo(b)fluoranthene	0.00474	-1.28%	3.99%	-7.05%	3.63%
Benzo(k)fluoranthene	0.00429	4.60%	3.95%	-7.09%	2.65%
Benzo(a)pyrene	0.00268	1.75%	3.67%	-3.29%	3.60%
Indeno(123cd)pyrene	0.00333	4.85%	6.50%	-3.49%	3.96%
Dibenzo(ah)anthracene	0.00522	4.57%	4.79%	-4.03%	2.73%
Benzo(ghi)perylene	0.00160	2.36%	1.28%	-3.30%	1.10%

METHOD STATEMENT



Matrix Spike Recoveries

Compound	Final Effluent		Crude Sewage		Trade Effluent	
	Recovery	RSD	Recovery	RSD	Recovery	RSD
Naphthalene	100.4%	2.68%	90.0%	7.30%	101.0%	1.54%
Acenaphthylene	101.8%	2.33%	102.8%	2.59%	103.1%	1.75%
Acenaphthene	96.0%	2.97%	95.0%	5.15%	98.2%	2.53%
Fluorene	101.1%	2.60%	100.0%	4.25%	102.3%	2.14%
Phenanthrene	101.8%	2.75%	101.1%	3.29%	102.6%	1.51%
Anthracene	106.3%	3.42%	109.6%	5.28%	106.2%	3.28%
Fluoranthene	102.1%	2.93%	98.1%	4.68%	105.8%	2.49%
Pyrene	102.1%	3.06%	98.2%	5.86%	105.5%	2.50%
Benzo(a)anthracene	107.3%	5.68%	112.2%	9.55%	104.7%	5.19%
Chrysene	99.9%	3.10%	97.8%	3.72%	101.7%	2.44%
Benzo(b)fluoranthene	92.8%	6.00%	92.0%	4.56%	98.3%	4.73%
Benzo(k)fluoranthene	91.4%	4.81%	89.7%	3.35%	98.0%	5.42%
Benzo(a)pyrene	99.3%	4.73%	96.4%	5.14%	105.0%	3.36%
Indeno(123cd)pyrene	100.4%	5.08%	105.8%	6.02%	105.1%	6.09%
Dibenzo(ah)anthracene	93.2%	4.23%	91.1%	4.07%	100.8%	5.08%
Benzo(ghi)perylene	100.1%	3.39%	96.3%	3.52%	103.6%	2.34%

Compound	Surface Water		Landfill Leachate		Groundwater	
	Recovery	RSD	Recovery	RSD	Recovery	RSD
Naphthalene	101.7%	2.44%	101.6%	1.82%	101.1%	2.52%
Acenaphthylene	104.9%	1.91%	104.3%	2.12%	104.4%	2.68%
Acenaphthene	97.9%	3.09%	98.8%	3.39%	100.4%	1.71%
Fluorene	102.8%	2.54%	103.4%	2.65%	103.4%	2.38%
Phenanthrene	104.2%	2.20%	103.8%	2.22%	103.8%	2.92%
Anthracene	108.5%	3.14%	108.2%	4.01%	105.9%	4.62%
Fluoranthene	105.8%	2.78%	103.4%	2.95%	105.0%	3.16%
Pyrene	105.1%	2.88%	104.3%	3.06%	104.7%	3.14%
Benzo(a)anthracene	106.0%	5.92%	107.3%	6.04%	104.8%	4.21%
Chrysene	101.1%	2.63%	101.4%	3.68%	101.9%	2.19%
Benzo(b)fluoranthene	98.1%	5.93%	97.5%	6.00%	97.4%	5.72%
Benzo(k)fluoranthene	96.8%	5.29%	97.0%	5.39%	96.6%	3.91%
Benzo(a)pyrene	104.3%	3.70%	104.9%	4.41%	104.6%	3.55%
Indeno(123cd)pyrene	103.5%	4.00%	108.6%	5.00%	102.2%	6.58%
Dibenzo(ah)anthracene	98.5%	3.68%	100.2%	6.52%	96.6%	5.14%
Benzo(ghi)perylene	101.9%	2.13%	104.2%	3.77%	102.8%	2.52%

METHOD STATEMENT



Uncertainty of Measurement:

Compound	UoM (%)
Naphthalene	9.44
Acenaphthylene	8.67
Acenaphthene	9.36
Fluorene	9.17
Phenanthrene	7.98
Anthracene	17.11
Fluoranthene	11.07
Pyrene	10.65
Benzo(a)anthracene	19.79
Chrysene	7.59
Benzo(b)fluoranthene	14.23
Benzo(k)fluoranthene	14.83
Benzo(a)pyrene	12.24
Indeno(123cd)pyrene	16.06
Dibenzo(ah)anthracene	16.49
Benzo(ghi)perylene	9.81

References:

Methods for the Examination of Waters and Associated Materials. The determination of 6 specific polynuclear aromatic hydrocarbons in waters (with notes on the determination of other PAH) 1986. HMSO, London. ISBN.0117520322.