

# METHOD STATEMENT



## Determinand:

Semi volatile organic compounds (SVOCs)

## Matrix:

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

## Principle of Method:

An automated dispersive liquid/liquid microextraction procedure (DLLME) is used to extract neutral and acidic organic compounds from acidified 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 compounds are determined by Electron Impact GCMS operated in simultaneous SIM/Scan mode.

## 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. Where significant headspace is evident a senior analyst must be informed.

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

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

Samples are stable for 7 days from sampling. This is based upon Chapter Four of the SW-846 Compendium for all compounds apart from 4-Chlorophenol, 3,5-Dimethylphenol and 2,6-Dichlorophenol the stability for which is based upon in-house data.

## Interferences

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

## Performance of Method:

LOD, Precision and Bias

Compound	LOD (ug/L)	MRL (ug/L)	Low Direct		High Direct	
			Bias	RSD	Bias	RSD
Phenol	0.0526	0.5	-0.89%	4.07%	-2.61%	2.28%
Bis(2-chloroethyl)ether	0.0330	1	0.16%	3.32%	-0.48%	4.69%
2-Chlorophenol	0.0252	0.1	2.64%	1.44%	2.05%	3.06%
1,3-Dichlorobenzene	0.0191	0.1	-3.59%	7.38%	-2.75%	2.91%
1,4-Dichlorobenzene	0.0314	0.1	-0.64%	5.60%	2.26%	3.56%
1,2-Dichlorobenzene	0.0228	0.1	-1.48%	5.68%	-0.15%	2.11%
2-Methylphenol	0.0259	0.1	1.14%	4.36%	-1.07%	2.42%
Bis(chloroisopropyl)ether	0.0332	1	0.44%	2.76%	-0.19%	2.76%
3&4-Methylphenol	0.0386	0.1	3.00%	5.91%	0.32%	2.46%
n-Nitroso-di-n-propylamine	0.0331	1.0	2.88%	7.92%	1.45%	2.55%

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Compound	LOD (ug/L)	MRL (ug/L)	Low Direct		High Direct	
			Bias	RSD	Bias	RSD
Hexachloroethane	0.0236	1.0	-1.65%	3.31%	-3.22%	6.06%
Nitrobenzene	0.0316	1.0	0.52%	3.59%	-1.26%	1.23%
Isophorone	0.2190	1.0	2.27%	7.90%	4.30%	8.57%
2-Nitrophenol	0.0526	0.1	4.63%	7.19%	3.28%	2.64%
2,4-Dimethylphenol	0.0186	0.1	3.11%	4.37%	-0.03%	3.36%
Bis(2-chloroethoxy)methane	0.0224	1.0	0.85%	4.32%	-0.09%	4.94%
3,5-Dimethylphenol	0.0325	0.1	4.84%	7.77%	3.98%	6.07%
2,4-Dichlorophenol	0.0199	0.1	6.46%	3.61%	4.31%	4.75%
1,2,4-Trichlorobenzene	0.0189	0.1	-2.77%	4.17%	-2.53%	2.73%
Naphthalene	0.0210	0.1	-1.15%	5.10%	0.99%	2.84%
4-Chlorophenol	0.0545	0.1	4.27%	6.19%	1.42%	5.28%
2,6-Dichlorophenol	0.0226	0.1	-0.06%	3.73%	-2.01%	3.12%
Hexachlorobutadiene	0.0354	0.1	0.05%	6.44%	-3.50%	2.50%
4-Chloro-3-methylphenol	0.0287	0.1	5.61%	5.13%	2.44%	2.28%
2-Methylnaphthalene	0.0221	0.1	-1.24%	4.45%	0.23%	1.09%
1-Methylnaphthalene	0.0220	0.1	-1.36%	4.23%	0.44%	1.31%
2,4,6-Trichlorophenol	0.0247	0.1	-0.03%	2.32%	2.34%	3.01%
2,4,5-Trichlorophenol	0.0597	0.1	3.32%	2.24%	-1.42%	3.03%
2-Chloronaphthalene	0.0236	0.1	-1.67%	4.33%	0.30%	1.11%
Dimethylphthalate	0.0415	1.0	-5.43%	8.09%	1.30%	6.51%
2,6-Dinitrotoluene	0.0725	1.0	4.60%	8.08%	3.05%	2.35%
Acenaphthylene	0.0264	0.1	-2.72%	7.17%	0.27%	2.52%
Acenaphthene	0.0230	0.1	-1.73%	4.39%	-0.33%	1.85%
2,4-Dinitrophenol	0.4517	5.0	3.11%	13.85%	3.23%	7.42%
4-Nitrophenol	3.986	5.0	0.28%	17.07%	3.00%	5.29%
Dibenzofuran	0.0208	0.1	-3.28%	4.91%	-1.48%	3.39%
2,4-Dinitrotoluene	0.1397	1.0	0.88%	8.54%	3.32%	2.42%
2,3,5,6-Tetrachlorophenol	0.0260	0.1	-7.01%	8.39%	-0.19%	2.31%
2,3,4,6-Tetrachlorophenol	0.0250	0.1	-1.75%	2.78%	0.78%	1.90%
Diethylphthalate	0.0311	1.0	3.89%	3.78%	1.79%	2.75%
Fluorene	0.0208	0.1	-2.21%	4.64%	-0.43%	1.37%
4-Chlorophenyl phenyl ether	0.0233	0.1	-3.58%	4.94%	-2.77%	3.91%
4,6-Dinitro-2-Methylphenol	0.2870	2.0	3.05%	11.30%	3.65%	5.43%
Diphenylamine	0.0201	1.0	5.87%	3.86%	2.60%	4.17%
Azobenzene	0.0226	1.0	2.07%	4.98%	3.19%	2.73%
4-Bromophenyl phenyl ether	0.0210	1.0	0.33%	2.99%	0.63%	4.38%
Hexachlorobenzene	0.0237	0.1	1.75%	8.78%	-1.42%	4.59%
Pentachlorophenol	0.0330	1.0	-5.03%	8.78%	0.54%	3.81%
Phenanthrene	0.0634	0.1	6.81%	12.95%	2.72%	7.99%

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Compound	LOD (ug/L)	MRL (ug/L)	Low Direct		High Direct	
			Bias	RSD	Bias	RSD
Anthracene	0.0215	0.1	1.60%	5.02%	0.37%	1.33%
Carbazole	0.0197	1.0	3.40%	2.85%	2.69%	3.50%
Di-n-butylphthalate	0.7063	5.0	5.48%	18.13%	1.80%	7.27%
Fluoranthene	0.0242	0.1	-0.48%	6.56%	-0.89%	2.00%
Pyrene	0.0242	0.1	0.23%	6.34%	-0.47%	2.08%
Benzyl butyl phthalate	0.0313	1.0	2.33%	8.07%	5.05%	4.23%
Benz(a)anthracene	0.0480	0.1	2.51%	9.11%	-1.39%	2.61%
Chrysene	0.0286	0.1	6.47%	8.09%	-1.97%	3.01%
Bis(2-ethylhexyl)phthalate	0.4438	1.0	0.81%	16.23%	4.02%	8.73%
Di-n-octyl phthalate	0.0628	1.0	-1.07%	11.67%	1.72%	6.99%
Benzo(b)fluoranthene	0.0374	0.1	3.65%	7.19%	-5.21%	5.76%
Benzo(k)fluoranthene	0.0241	0.1	0.92%	6.47%	-3.80%	6.91%
Benzo(a)pyrene	0.0390	0.1	4.48%	11.17%	5.33%	6.20%
Indeno(1,2,3-cd)Pyrene	0.0220	0.1	0.17%	10.58%	3.01%	4.51%
Dibenzo(ah)anthracene	0.0226	0.1	2.07%	9.66%	0.81%	6.58%
Benzo(ghi)perylene	0.0259	0.1	3.20%	7.58%	-0.91%	2.86%

## Matrix Spike Recoveries

Compound	Groundwater		Surface water		Treated Effluent	
	Rec	RSD	Rec	RSD	Rec	RSD
Phenol	96.36%	2.13%	96.06%	2.31%	95.69%	1.78%
Bis(2-chloroethyl)ether	98.48%	3.76%	97.97%	3.91%	98.86%	4.28%
2-Chlorophenol	99.51%	3.82%	98.98%	3.67%	98.56%	3.57%
1,3-Dichlorobenzene	97.54%	2.99%	95.71%	3.90%	97.00%	3.72%
1,4-Dichlorobenzene	101.90%	3.24%	98.46%	3.53%	99.97%	2.65%
1,2-Dichlorobenzene	99.77%	2.01%	96.91%	2.99%	98.65%	2.74%
2-Methylphenol	96.94%	2.10%	96.06%	3.15%	95.63%	3.27%
Bis(chloroisopropyl)ether	98.89%	1.82%	98.42%	2.06%	98.92%	2.41%
3&4-Methylphenol	96.77%	3.54%	97.89%	2.68%	97.16%	2.64%
n-Nitroso-di-n-propylamine	98.41%	3.75%	97.34%	6.29%	99.29%	2.52%
Hexachloroethane	97.97%	3.17%	97.70%	4.18%	98.87%	3.03%
Nitrobenzene	101.40%	3.91%	101.21%	4.68%	109.54%	4.97%
Isophorone	103.51%	9.60%	101.91%	9.26%	103.19%	10.92%
2-Nitrophenol	100.47%	4.20%	100.19%	5.85%	101.96%	3.04%
2,4-Dimethylphenol	96.55%	5.17%	100.51%	1.74%	99.44%	4.82%
Bis(2-chloroethoxy)methane	98.50%	4.07%	98.12%	3.92%	98.99%	4.81%
3,5-Dimethylphenol	96.65%	14.15%	102.64%	5.02%	104.89%	6.42%
2,4-Dichlorophenol	101.36%	5.61%	101.32%	5.46%	100.84%	5.39%

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Compound	Groundwater		Surface water		Treated Effluent	
	Rec	RSD	Rec	RSD	Rec	RSD
1,2,4-Trichlorobenzene	96.82%	3.78%	95.00%	3.63%	96.11%	2.81%
Naphthalene	100.63%	4.54%	97.84%	4.02%	99.40%	3.17%
4-Chlorophenol	99.85%	4.98%	100.40%	6.14%	101.21%	5.83%
2,6-Dichlorophenol	98.70%	2.96%	97.34%	3.20%	96.38%	2.63%
Hexachlorobutadiene	94.79%	4.16%	94.43%	3.69%	94.83%	2.95%
4-Chloro-3-methylphenol	101.07%	1.76%	101.02%	1.72%	102.18%	2.11%
2-Methylnaphthalene	99.48%	1.77%	97.75%	2.30%	99.29%	1.65%
1-Methylnaphthalene	99.78%	2.00%	97.82%	2.47%	99.20%	1.77%
2,4,6-Trichlorophenol	102.44%	2.87%	101.19%	3.44%	101.27%	3.13%
2,4,5-Trichlorophenol	99.29%	2.68%	98.26%	2.82%	96.89%	2.17%
2-Chloronaphthalene	99.31%	1.84%	97.72%	2.44%	98.98%	1.81%
Dimethylphthalate	99.35%	5.59%	96.93%	7.07%	99.46%	6.72%
2,6-Dinitrotoluene	101.43%	2.37%	101.08%	3.06%	102.57%	2.60%
Acenaphthylene	91.25%	17.89%	96.88%	3.77%	98.79%	3.21%
Acenaphthene	99.35%	2.63%	97.57%	2.81%	98.45%	2.13%
2,4-Dinitrophenol	100.77%	7.87%	103.97%	9.14%	108.35%	6.93%
4-Nitrophenol	101.15%	5.66%	101.42%	7.10%	103.37%	3.42%
Dibenzofuran	98.18%	4.15%	96.05%	4.28%	95.46%	3.84%
2,4-Dinitrotoluene	101.54%	2.37%	101.18%	3.55%	101.01%	1.87%
2,3,5,6-Tetrachlorophenol	99.61%	1.75%	98.11%	1.98%	99.20%	2.94%
2,3,4,6-Tetrachlorophenol	100.78%	1.89%	98.90%	1.96%	99.82%	1.76%
Diethylphthalate	100.90%	2.60%	100.68%	2.62%	103.37%	3.09%
Fluorene	100.51%	3.80%	97.97%	2.55%	98.86%	2.09%
4-Chlorophenyl phenyl ether	96.82%	4.55%	95.19%	4.89%	94.22%	4.38%
4,6-Dinitro-2-Methylphenol	100.48%	6.28%	102.24%	7.73%	102.60%	5.30%
Diphenylamine	85.82%	17.25%	102.45%	2.03%	103.23%	3.51%
Azobenzene	102.45%	2.04%	102.09%	2.41%	102.55%	2.22%
4-Bromophenyl phenyl ether	100.68%	3.73%	99.36%	4.42%	98.12%	4.01%
Hexachlorobenzene	97.21%	5.98%	97.25%	6.72%	97.82%	6.96%
Pentachlorophenol	99.97%	3.01%	100.47%	2.60%	101.84%	3.33%
Phenanthrene	102.75%	8.43%	100.67%	8.23%	101.12%	7.81%
Anthracene	98.57%	3.01%	98.54%	2.31%	99.63%	1.75%
Carbazole	101.88%	4.40%	102.97%	3.30%	104.37%	3.05%
Di-n-butylphthalate	98.23%	8.05%	106.22%	5.83%	124.30%	4.16%
Fluoranthene	99.31%	2.08%	96.95%	3.29%	95.82%	2.96%
Pyrene	99.66%	2.12%	97.29%	3.34%	96.37%	2.75%
Benzyl butyl phthalate	103.00%	5.66%	106.37%	6.23%	117.24%	6.36%
Benz(a)anthracene	98.90%	3.00%	96.15%	4.98%	93.12%	4.72%
Chrysene	98.33%	3.28%	95.40%	4.72%	95.96%	4.54%
Bis(2-ethylhexyl)phthalate	101.30%	8.51%	89.66%	14.02%	81.33%	9.46%

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Compound	Groundwater		Surface water		Treated Effluent	
	Rec	RSD	Rec	RSD	Rec	RSD
Di-n-octyl phthalate	99.92%	8.04%	84.80%	13.13%	73.53%	5.96%
Benzo(b)fluoranthene	95.92%	3.95%	94.49%	5.35%	95.05%	4.65%
Benzo(k)fluoranthene	97.43%	4.58%	89.90%	7.14%	85.85%	7.99%
Benzo(a)pyrene	104.61%	7.10%	106.04%	8.58%	101.35%	9.25%
Indeno(1,2,3-cd)Pyrene	98.10%	3.12%	98.81%	4.48%	100.45%	2.71%
Dibenzo(ah)anthracene	98.95%	3.25%	90.07%	5.61%	84.48%	3.47%
Benzo(ghi)perylene	99.70%	3.70%	96.93%	4.08%	97.35%	2.72%

Compound	Trade Effluent		Land Leachate	
	Rec	RSD	Rec	RSD
Phenol	95.93%	2.59%	94.99%	2.72%
Bis(2-chloroethyl)ether	98.06%	4.88%	98.40%	3.84%
2-Chlorophenol	98.84%	3.57%	98.53%	4.08%
1,3-Dichlorobenzene	96.57%	3.03%	97.74%	2.74%
1,4-Dichlorobenzene	99.76%	2.83%	101.55%	3.92%
1,2-Dichlorobenzene	98.32%	1.84%	99.77%	2.27%
2-Methylphenol	96.07%	4.26%	95.50%	2.69%
Bis(chloroisopropyl)ether	98.35%	3.09%	98.13%	2.47%
3&4-Methylphenol	96.67%	3.13%	97.57%	3.36%
n-Nitroso-di-n-propylamine	99.07%	4.47%	98.75%	3.45%
Hexachloroethane	96.17%	4.51%	96.39%	6.07%
Nitrobenzene	99.74%	2.42%	146.78%	12.48%
Isophorone	103.61%	10.41%	101.99%	8.78%
2-Nitrophenol	100.81%	4.46%	101.26%	4.09%
2,4-Dimethylphenol	99.50%	4.02%	94.79%	2.83%
Bis(2-chloroethoxy)methane	98.40%	4.97%	98.19%	4.67%
3,5-Dimethylphenol	103.84%	6.00%	104.82%	3.76%
2,4-Dichlorophenol	100.65%	5.23%	101.38%	5.74%
1,2,4-Trichlorobenzene	95.80%	3.30%	96.91%	3.43%
Naphthalene	99.34%	4.22%	99.70%	3.22%
4-Chlorophenol	101.04%	5.81%	103.25%	4.80%
2,6-Dichlorophenol	97.46%	3.88%	93.14%	4.01%
Hexachlorobutadiene	93.74%	3.72%	97.60%	4.32%
4-Chloro-3-methylphenol	101.65%	2.61%	104.57%	2.78%
2-Methylnaphthalene	98.58%	1.23%	101.11%	1.69%
1-Methylnaphthalene	99.09%	1.70%	100.78%	1.97%
2,4,6-Trichlorophenol	101.98%	3.80%	99.20%	3.36%
2,4,5-Trichlorophenol	97.94%	2.89%	95.25%	3.92%
2-Chloronaphthalene	98.70%	1.23%	100.52%	1.83%
Dimethylphthalate	98.79%	5.80%	102.69%	6.48%

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Compound	Trade Effluent		Land Leachate	
	Rec	RSD	Rec	RSD
2,6-Dinitrotoluene	101.31%	3.05%	102.71%	2.93%
Acenaphthylene	98.38%	2.75%	100.12%	2.55%
Acenaphthene	97.86%	2.18%	97.90%	2.36%
2,4-Dinitrophenol	102.27%	9.13%	114.17%	7.32%
4-Nitrophenol	101.48%	6.16%	101.26%	7.31%
Dibenzofuran	96.06%	4.07%	87.75%	4.94%
2,4-Dinitrotoluene	100.92%	2.44%	94.07%	2.46%
2,3,5,6-Tetrachlorophenol	99.09%	1.82%	100.44%	2.93%
2,3,4,6-Tetrachlorophenol	99.81%	2.30%	99.20%	2.38%
Diethylphthalate	101.11%	3.65%	104.32%	4.31%
Fluorene	98.06%	1.51%	99.35%	1.93%
4-Chlorophenyl phenyl ether	94.70%	4.48%	87.33%	6.20%
4,6-Dinitro-2-Methylphenol	101.83%	6.99%	102.84%	6.03%
Diphenylamine	100.09%	3.81%	103.33%	3.50%
Azobenzene	101.13%	3.31%	101.81%	1.87%
4-Bromophenyl phenyl ether	98.03%	4.91%	92.49%	3.81%
Hexachlorobenzene	96.84%	6.59%	103.49%	7.57%
Pentachlorophenol	99.61%	3.11%	109.67%	1.60%
Phenanthrene	101.22%	8.59%	98.19%	9.34%
Anthracene	98.78%	1.26%	98.08%	1.96%
Carbazole	101.81%	4.58%	103.03%	4.00%
Di-n-butylphthalate	97.91%	8.85%	110.39%	7.05%
Fluoranthene	98.80%	1.85%	84.10%	2.28%
Pyrene	99.37%	2.09%	89.65%	2.39%
Benzyl butyl phthalate	101.72%	5.15%	102.51%	7.52%
Benz(a)anthracene	97.87%	3.10%	89.01%	3.09%
Chrysene	97.96%	3.07%	91.24%	7.14%
Bis(2-ethylhexyl)phthalate	101.22%	10.47%	104.61%	6.94%
Di-n-octyl phthalate	100.26%	8.14%	92.53%	9.82%
Benzo(b)fluoranthene	95.78%	4.45%	94.21%	5.77%
Benzo(k)fluoranthene	97.93%	6.03%	86.69%	6.33%
Benzo(a)pyrene	106.32%	7.59%	88.49%	8.22%
Indeno(1,2,3-cd)Pyrene	96.02%	4.20%	102.44%	2.55%
Dibenzo(ah)anthracene	98.37%	3.66%	90.45%	3.25%
Benzo(ghi)perylene	98.70%	3.01%	97.19%	3.36%

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## Uncertainty of Measurement:

Compound	UoM
Phenol	10.45%
Bis(2-chloroethyl)ether	10.172%
2-Chlorophenol	7.912%
1,3-Dichlorobenzene	13.472%
1,4-Dichlorobenzene	9.125%
1,2-Dichlorobenzene	7.331%
2-Methylphenol	10.565%
Bis(chloroisopropyl)ether	7.186%
3&4-Methylphenol	9.04%
n-Nitroso-di-n-propylamine	9.743%
Hexachloroethane	11.263%
Nitrobenzene	11.357%
Isophorone	22.503%
2-Nitrophenol	9.467%
2,4-Dimethylphenol	9.621%
Bis(2-chloroethoxy)methane	11.313%
3,5-Dimethylphenol	17.438%
2,4-Dichlorophenol	10.958%
1,2,4-Trichlorobenzene	10.352%
Naphthalene	7.65%
4-Chlorophenol	16.489%
2,6-Dichlorophenol	12.016%
Hexachlorobutadiene	15.147%
4-Chloro-3-methylphenol	7.685%
2-Methylnaphthalene	5.398%
1-Methylnaphthalene	4.715%
2,4,6-Trichlorophenol	10.174%
2,4,5-Trichlorophenol	9.664%
2-Chloronaphthalene	5.033%
Dimethylphthalate	17.786%
2,6-Dinitrotoluene	8.451%
Acenaphthylene	14.913%
Acenaphthene	5.595%
2,4-Dinitrophenol	23.562%
4-Nitrophenol	13.496%
Dibenzofuran	14.296%
2,4-Dinitrotoluene	8.582%
2,3,5,6-Tetrachlorophenol	7.452%
2,3,4,6-Tetrachlorophenol	6.793%

# METHOD STATEMENT



Compound	UoM
Diethylphthalate	8.496%
Fluorene	5.695%
4-Chlorophenyl phenyl ether	16.135%
4,6-Dinitro-2-Methylphenol	14.178%
Diphenylamine	16.062%
Azobenzene	6.964%
4-Bromophenyl phenyl ether	11.419%
Hexachlorobenzene	19.473%
Pentachlorophenol	12.462%
Phenanthrene	17.818%
Anthracene	9.043%
Carbazole	9.865%
Di-n-butylphthalate	31.125%
Fluoranthene	19.877%
Pyrene	17.206%
Benzyl butyl phthalate	20.706%
Benz(a)anthracene	21.806%
Chrysene	14.741%
Bis(2-ethylhexyl)phthalate	28.08%
Di-n-octyl phthalate	32.257%
Benzo(b)fluoranthene	19.591%
Benzo(k)fluoranthene	26.608%
Benzo(a)pyrene	28.617%
Indeno(1,2,3-cd)Pyrene	16.04%
Dibenzo(ah)anthracene	23.16%
Benzo(ghi)perylene	13.876%

## References:

EPA Method 8270E (SW-846): Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC-MS).

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