

METHOD STATEMENT

Determinand:

This method covers the determination selected dissolved phenols, chlorophenols and nitrophenols.

Matrix:

Surface Waters, Groundwaters, Effluents and Leachates

Principle of Method:

The compounds are extracted from aqueous solution using solid phase extraction cartridges. The compounds are eluted from the cartridges with ethyl acetate and are quantified by gas chromatography with mass spectrometry detection (GCMS). The mass spectrometer is operated in electron impact mode with simultaneous full scan and specific ion monitoring.

Sampling and Sample Preparation:

Samples should be collected in 250ml glass bottles with either glass or polypropylene tops and stored between 5 ± 3°C.

Samples are stable for 7 days (US EPA Soil Analysis QA QC Manual 2012 Table 3-1) from sampling.

Interferences:

Any co-extracted substance with a corresponding GC retention time and with the same ions as those being monitored will interfere.

Performance of Method:

Range of Method: Low Range 2µg/l - 8µg/l without dilution.
High Range 8µg/l - 80µg/l without dilution.

Low range calibration

Low Standard = 2µg/l
High Standard = 8µg/l

Determinand	LOD µg/l	MRL (µg/l)	MCERTS Accred.	Low Standard		High Standard	
				% Rec.	% RSD	% Rec.	% RSD
Phenol	0.093	0.5	✓	102.3	9.4	97.8	5.0
2-Chlorophenol	0.092	0.1		103.5	8.7	105.0	8.0
o-Cresol	0.063	0.1		99.7	8.5	103.4	6.7
m/p-Cresol	0.089	0.1		98.2	8.5	101.9	5.2
2-Nitrophenol	0.089	0.1		108.0	12.1	107.0	9.0
2,4-Dimethylphenol	0.092	0.1		102.3	16.2	107.5	8.3
3,5-Dimethylphenol	0.057	0.1		98.7	8.6	101.5	5.2
2,4-Dichlorophenol	0.096	0.1		97.4	6.7	101.8	4.3
4-Chlorophenol	0.075	0.1		99.3	7.2	100.3	4.1
2,6-Dichlorophenol	0.062	0.1		100.1	7.2	102.9	4.7
4-Chloro-3-methylphenol	0.072	0.1		95.7	9.5	101.3	4.5
2,4,6-Trichlorophenol	0.085	0.1		98.3	18.3	105.6	10.2
2,4,5-Trichlorophenol	0.079	0.1		92.8	13.0	102.6	7.9



METHOD STATEMENT



Determinand		Finham Final Effluent		Trade Effluent		Wolston Final Effluent	
		Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk
Phenol	%Rec.	85.3	97.7	87.3	98.6	84.0	94.9
	%RSD	14.1	5.4	12.8	4.3	13.4	14.0
2-Chlorophenol	%Rec.	95.1	102.2	98.1	104.4	89.6	100.6
	%RSD	8.1	7.0	8.5	5.4	5.5	6.4
o-Cresol	%Rec.	93.9	101.3	95.3	101.8	92.1	101.2
	%RSD	8.1	5.6	9.5	5.5	5.4	5.8
m/p-Cresol	%Rec.	91.8	98.3	92.1	101.5	95.1	102.4
	%RSD	10.4	12.2	14.6	4.6	6.1	6.9
2-Nitrophenol	%Rec.	106.7	110.4	102.5	105.3	104.3	107.1
	%RSD	12.4	9.2	12.6	9.0	5.7	4.1
2,4-Dimethylphenol	%Rec.	103.2	104.2	98.9	99.4	104.7	107.6
	%RSD	14.0	6.9	4.94	11.3	12.1	6.8
3,5-Dimethylphenol	%Rec.	93.7	100.3	95.3	98.6	96.7	101.9
	%RSD	10.3	4.2	12.2	3.3	5.3	5.4
2,4-Dichlorophenol	%Rec.	94.0	100.5	93.1	99.9	92.1	100.6
	%RSD	7.0	3.5	5.9	2.9	5.3	5.7
4-Chlorophenol	%Rec.	91.6	100.0	92.9	98.2	89.1	100.8
	%RSD	11.9	4.5	7.2	3.0	6.7	5.7
2,6-Dichlorophenol	%Rec.	97.1	102.2	96.0	101.4	94.0	103.3
	%RSD	7.6	3.4	7.0	3.0	5.3	4.6
4-Chloro-3-methylphenol	%Rec.	95.8	103.4	94.9	98.3	98.4	104.6
	%RSD	9.2	5.3	9.6	4.0	6.0	5.2
2,4,6-Trichlorophenol	%Rec.	111.6	110.7	97.3	101.9	109.3	115.4
	%RSD	12.0	8.3	20.4	11.5	5.3	4.3
2,4,5-Trichlorophenol	%Rec.	97.7	104.6	84.3	100.4	98.1	109.1
	%RSD	14.7	8.2	18.3	7.3	4.3	7.2

Determinand		Groundwater		Landfill Leachate		Soil Leachate		Surface Water	
		Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk
Phenol	%Rec.	88.9	96.4	84.4	96.6	90.1	96.4	88.09	97.00
	%RSD	18.8	5.2	15.3	4.6	23.0	5.2	10.22	4.96
2-Chlorophenol	%Rec.	94.2	103.0	92.8	103.1	93.1	96.9	94.57	103.19
	%RSD	9.7	9.3	9.4	7.4	6.3	4.6	8.28	7.96
o-Cresol	%Rec.	91.5	100.5	89.8	100.1	93.9	98.1	94.4	101.9
	%RSD	8.3	6.7	8.2	5.6	5.2	4.4	8.5	6.7
m/p-Cresol	%Rec.	92.2	100.3	87.7	99.5	93.6	98.1	93.8	100.6
	%RSD	8.6	5.2	14.6	5.2	6.5	4.8	10.2	5.2
2-Nitrophenol	%Rec.	106.8	112.5	107.6	111.2	103.9	106.1	104.8	104.8
	%RSD	10.8	8.2	9.0	8.0	7.5	6.9	8.5	9.0
2,4-Dimethylphenol	%Rec.	83.5	88.4	87.4	94.3	99.6	103.3	100.0	106.0
	%RSD	20.9	20.7	26.4	23.1	3.8	4.4	9.7	8.3
3,5-Dimethylphenol	%Rec.	92.8	99.1	89.9	98.5	97.5	101.2	92.1	99.8
	%RSD	10.1	4.9	9.4	3.7	3.2	3.4	7.1	5.2
2,4-Dichlorophenol	%Rec.	92.9	100.4	93.3	100.5	94.1	99.2	92.3	100.2
	%RSD	6.6	4.4	5.2	3.6	3.0	2.8	6.7	4.3
4-Chlorophenol	%Rec.	93.9	100.7	93.0	99.7	96.7	102.2	91.5	98.8
	%RSD	9.4	4.3	7.4	3.8	4.3	5.1	7.7	4.1
2,6-Dichlorophenol	%Rec.	96.3	101.9	95.6	102.0	95.5	99.6	95.8	101.5
	%RSD	7.0	5.1	5.1	3.4	4.9	4.1	5.9	4.7
4-Chloro-3-methylphenol	%Rec.	98.0	102.9	97.2	103.3	103.9	107.7	93.0	100.3
	%RSD	9.9	5.0	7.7	5.5	5.3	5.5	9.5	4.5
2,4,6-Trichlorophenol	%Rec.	113.2	112.6	117.3	112.2	91.5	98.1	93.8	104.0
	%RSD	12.5	7.6	11.2	7.5	8.8	5.9	13.4	10.2
2,4,5-Trichlorophenol	%Rec.	93.6	105.8	99.8	107.3	87.3	94.9	87.2	101.4
	%RSD	13.9	6.1	10.8	7.1	17.3	12.2	9.0	7.9



METHOD STATEMENT



High Range calibration

Low Standard = 8µg/l
High Standard = 80µg/l

Determinand	LOD µg/l	MRL (µg/l)	MCERTS Accred.	Low Standard		High Standard	
				% Rec.	% RSD	% Rec.	% RSD
Phenol	0.697	2	✓	110.7	14.4	103.3	4.4
2-Chlorophenol	1.283	2		110.6	7.7	107.9	7.1
o-Cresol	0.642	2		105.5	8.3	106.4	6.5
m/p-Cresol	1.462	2		105.1	7.7	105.4	5.6
2-Nitrophenol	1.618	2		95.1	12.4	107.9	8.3
2,4-Dimethylphenol	1.436	2		103.8	7.4	106.0	4.5
3,5-Dimethylphenol	0.998	2		103.6	7.3	104.8	4.0
2,4-Dichlorophenol	0.856	2		104.8	5.7	104.4	3.3
4-Chlorophenol	1.276	2		99.1	7.2	104.8	3.5
2,6-Dichlorophenol	0.686	2		104.3	6.6	104.5	3.6
4-Chloro-3-methylphenol	1.44	2		95.9	7.2	103.5	3.4
2,4,6-Trichlorophenol	2.382	3		94.6	13.6	106.1	4.3
2,4,5-Trichlorophenol	2.03	3		95.1	9.5	99.5	7.6

Determinand		Finham Final Effluent		Trade Effluent		Wolston Final Effluent	
		Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk
Phenol	%Rec.	92.5	102.4	97.6	101.9	92.6	91.8
	%RSD	4.5	4.0	4.6	4.0	16.3	11.8
2-Chlorophenol	%Rec.	107.9	107.8	109.7	106.8	104.8	99.3
	%RSD	6.7	6.1	4.3	6.1	7.1	6.9
o-Cresol	%Rec.	102.7	105.9	103.2	104.4	104.9	100.5
	%RSD	6.4	5.7	5.8	5.6	6.4	6.2
m/p-Cresol	%Rec.	102.4	104.9	102.9	103.9	106.0	100.3
	%RSD	5.1	5.0	5.0	4.7	7.2	6.7
2-Nitrophenol	%Rec.	97.5	109.6	92.0	106.3	97.5	104.2
	%RSD	13.0	8.4	11.3	7.6	6.2	4.5
2,4-Dimethylphenol	%Rec.	102.0	103.3	96.5	100.9	108.0	103.4
	%RSD	6.1	5.0	9.9	8.3	6.7	5.6
3,5-Dimethylphenol	%Rec.	101.4	104.5	99.5	102.7	104.1	102.1
	%RSD	4.4	4.2	3.8	2.6	6.2	5.4
2,4-Dichlorophenol	%Rec.	102.4	104.4	101.9	102.8	103.5	100.9
	%RSD	4.2	2.7	3.6	2.1	5.8	5.3
4-Chlorophenol	%Rec.	97.3	104.2	95.5	102.8	101.5	102.4
	%RSD	5.4	3.0	4.0	2.2	5.9	4.7
2,6-Dichlorophenol	%Rec.	102.5	104.5	102.0	103.0	104.5	100.3
	%RSD	3.6	2.9	2.6	3.1	5.7	5.3
4-Chloro-3-methylphenol	%Rec.	98.6	103.4	93.1	102.2	103.4	104.3
	%RSD	5.7	3.2	4.8	2.5	5.8	4.1
2,4,6-Trichlorophenol	%Rec.	103.2	108.0	92.7	105.6	109.2	106.7
	%RSD	10.9	4.1	13.7	4.1	5.3	4.2
2,4,5-Trichlorophenol	%Rec.	99.5	102.5	94.6	100.0	112.5	104.6
	%RSD	10.7	8.5	8.2	8.9	5.3	9.6



METHOD STATEMENT



Determinand		Groundwater		Landfill Leachate		Soil Leachate		Surface Water	
		Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk
Phenol	%Rec.	101.2	100.0	96.6	100.9	96.3	96.6	98.13	102.76
	%RSD	12.6	7.1	4.7	5.7	9.5	6.6	4.76	4.44
2-Chlorophenol	%Rec.	108.8	105.0	107.8	105.7	89.6	95.0	109.10	107.70
	%RSD	5.8	9.9	6.8	8.7	14.1	12.0	5.95	7.12
o-Cresol	%Rec.	102.9	102.4	101.2	103.9	94.8	94.9	103.6	106.3
	%RSD	5.7	8.8	5.4	8.4	16.1	11.6	5.6	6.5
m/p-Cresol	%Rec.	103.0	101.7	101.6	103.1	94.3	97.1	103.1	105.3
	%RSD	5.1	8.1	4.8	7.7	12.9	11.6	5.0	5.6
2-Nitrophenol	%Rec.	99.3	108.5	98.7	108.6	89.3	97.9	91.9	107.7
	%RSD	12.7	9.2	12.6	8.7	13.8	13.1	12.8	8.3
2,4-Dimethylphenol	%Rec.	86.8	93.0	92.4	95.8	91.0	98.0	103.8	105.9
	%RSD	21.4	10.7	22.0	19.9	14.2	11.3	5.3	4.5
3,5-Dimethylphenol	%Rec.	101.3	101.8	100.0	102.4	93.7	99.1	102.3	104.6
	%RSD	5.3	7.2	4.7	6.9	13.3	11.4	7.2	4.0
2,4-Dichlorophenol	%Rec.	103.6	101.6	102.7	102.5	95.4	99.4	102.9	104.2
	%RSD	4.5	5.5	5.0	5.4	11.0	11.4	5.5	3.3
4-Chlorophenol	%Rec.	98.7	102.4	97.8	102.4	101.7	103.5	96.4	104.6
	%RSD	5.7	5.7	5.6	6.1	11.2	11.2	6.9	3.5
2,6-Dichlorophenol	%Rec.	102.9	101.6	102.4	102.6	91.5	98.7	102.1	104.4
	%RSD	3.8	5.8	3.6	6.1	13.5	12.0	4.1	3.6
4-Chloro-3-methylphenol	%Rec.	98.6	101.8	99.3	102.8	100.7	104.9	95.2	103.4
	%RSD	5.9	5.8	5.9	5.9	14.9	11.2	6.0	3.4
2,4,6-Trichlorophenol	%Rec.	104.1	107.1	105.3	106.8	96.6	102.2	95.6	106.1
	%RSD	11.7	6.1	8.9	6.8	18.8%	12.1	13.6	4.3
2,4,5-Trichlorophenol	%Rec.	100.1	101.3	101.2	105.3	93.5	106.5	95.8	99.3
	%RSD	8.0	8.9	7.8	9.1	16.0	11.7	9.6	7.6

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 (%)	
	Low Level	High Level
Phenol	22.95	23.10
2-Chlorophenol	20.88	29.96
o-Cresol	19.91	24.33
m/p-Cresol	27.07	25.63
2-Nitrophenol	17.50	20.51
2,4-Dimethylphenol	19.69	27.53
3,5-Dimethylphenol	20.94	24.28
2,4-Dichlorophenol	15.06	25.38
4-Chlorophenol	19.84	20.39
2,6-Dichlorophenol	15.93	13.85
4-Chloro-3-methylphenol	18.44	17.01
2,4,6-Trichlorophenol	17.85	29.06
2,4,5-Trichlorophenol	24.46	22.53

References:

“Extraction of Priority Pollutant Phenols from Aqueous Samples using A Styrenedivinybenzene Adsorbent”, by Lydia Nolan, Supelco Inc., Bellefonte, PA 16823-0048 USA. Reference available from Supelchem Limited, Shire Hill, Saffron Walden, Essex, CB11 3AZ.

