

METHOD STATEMENT

Determinand:

<u>Compound</u>	<u>CAS No.</u>
Phenol	108-95-2
o-Cresol	95-48-7
m-Cresol	108-39-4
p-Cresol	106-44-5
2-Chlorophenol	95-57-8
3-Chlorophenol	108-43-0
4-Chlorophenol	106-48-9
2,5-Xylenol	95-87-4
2,4-Xylenol	105-67-9
3,5-Xylenol	108-68-9
2,6-Xylenol	576-26-1
3,4-Xylenol	95-65-8
2,3-Xylenol	526-75-0
4-Chloro-2-Methylphenol*	1570-64-5
2-Chloro-5-Methylphenol*	615-74-7
2-Chloro-4-Methylphenol	6640-27-3
3-Chloro-4-Methylphenol	59-50-7
2,3-Dichlorophenol	576-24-9
2,4-Dichlorophenol	120-83-2
2,6-Dichlorophenol	87-65-0
2,4,5-Trichlorophenol	95-95-4
2,4,6 Trichlorophenol	88-06-2

*-These compounds co-elute and are reported as a pair.

Matrix:

Sample Type: Potable, Surface and groundwaters

Principle of Method:

The samples are made alkaline and pre-extracted with hexane to reduce levels of interfering compounds. The sample is then buffered using sodium bicarbonate and reacted with a solution of pentafluorobenzoyl chloride in hexane, by shaking. The phenol pentafluorobenzoyl esters partition into the hexane layer, excess derivatising reagent is removed by rinsing with sodium hydroxide solution and the esters are determined using gas chromatography with mass spectrometric detection.

Interferences:

Any compound which passes through the extraction and derivatisation procedure, and has similar gas chromatographic and mass spectrometric properties to the analyte.

Performance of Method:

Range of Application:

The analysis is quadratic with an allowed intercept for all parameters from the limit of detection to 1.2µg/l.



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Limit of Detection, Recoveries and Uncertainty of measurement of Compounds

An LOD re-evaluation exercise for this method was carried out on 07/08/18 to 31/08/18, the new LODs for the reportable compounds is listed below.

Determinand	Compound	Medium Water @800ng/L		L.O.D	
		Rec.	RSD	Uncert.	ng/l
A1Z	Phenol	94.6%	7.2%	± 19.8%	37
A28	o-Cresol	94.3%	7.1%	± 19.9%	10
A27	m-Cresol	98.0%	5.5%	± 13.1%	13
A29	p-Cresol	97.0%	5.6%	± 14.2%	11
A20	2-Chlorophenol	99.3%	3.4%	± 7.5%	25
A21	3-Chlorophenol	100.9%	6.4%	± 13.7%	9
A22	4-Chlorophenol	100.7%	7.0%	± 14.7%	10
AWS	4-Chloro-2-Methyl & 2-Chloro-5-Methylphenol	97.7%	4.5%	± 11.3%	8
A23	2-Chloro-4-Methylphenol	97.8%	4.2%	± 10.5%	5
A26	3-Chloro-4-Methylphenol	98.1%	5.1%	± 12.1%	8
A2A	2,4-Dichlorophenol	98.7%	4.2%	± 9.6%	12
A2C	2,4,5-Trichlorophenol	100.2%	5.1%	± 10.4%	6

The following compounds listed below are temporarily suspended from the suite due to their very poor sensitivity and chromatography. The data that was produced during the LOD exercise for these compounds does not meet our quality requirements for DWTS accredited results hence the decision to suspend.

Determinand	Compound	Medium Water @800ng/L		L.O.D	
		Rec.	RSD	Uncert.	ng/l
A2B	2,6-Dichlorophenol	98.5%	7.6%	± 16.8%	25
A2L	2,3-Dichlorophenol	99.6%	4.8%	± 10.0%	25
A2D	2,4,6-Trichlorophenol	96.9%	9.4%	± 21.9%	25
A2G	2,6-Xylenol	100.0%	4.3%	± 8.6%	25
A2F	2,5-Xylenol	99.2%	4.2%	± 9.2%	25
A2K	2,4-Xylenol	98.5%	4.4%	± 10.3%	25
A2J	3,5-Xylenol	102.3%	6.6%	± 15.4%	25
A2E	2,3-Xylenol	98.5%	4.0%	± 9.5%	25
A2H	3,4-Xylenol	102.9%	6.8%	± 16.5%	25

References:

"The Determination of Microgram and Submicrogram Amounts of Individual Phenols In River and Potable Waters" –HMSO publication Method 4 ISBN: 011 7520993.

