METHOD STATEMENT

Determinand:
Selection of Phenols and chlorophenols

Matrix:
Sample Type: treated and raw water, i.e. waters that are abstracted for potable supply and potable waters

Principle of Method:
Sample is buffered using di-sodium tetraborate and reacted with a solution of pentafluorobenzoyl chloride in hexane. Phenol pentafluorobenzoyl esters partition into the hexane layer, excess derivatising reagent is removed by washing the hexane sample extract with sodium hydroxide solution. Phenol pentafluorobenzoyl esters are determined using gas chromatography with mass spectrometric detection.

Sampling and Sample Preparation:
Samples are taken in 100mL amber or green glass bottles 0.100mL of – ascorbic acid solution 3% w/v as preservative and are stored at 3±2°C on receipt at the laboratory. Samples should be extracted within 28 days from sampling. Selected distribution/final treated water samples should be tested, at random, for levels of residual chlorine in order to confirm that bottles are continuing to be received with sodium thiosulfate having been present prior to sampling, according to our procedure.

Sampling, samples should be collected in 100mL coloured glass which has been proven to be suitable for this analysis, with PTFE lined screw caps and contain 0.100mL of – ascorbic acid solution 3% w/v.

Storage – samples should be analysed as soon as possible after collection. When this is not possible they should be stored under refrigeration at 3±2°C in the dark, until analysis can begin. The maximum permissible storage time prior to analysis is given below.

Interferences
GC-MS is a selective technique and interferences should only be encountered rarely. Any interfering compounds would have to display the identical SIM ion at the same retention time, this is unlikely in potable water samples. However, any compound which passes through the extraction procedure and has a similar gas chromatographic retention time and mass spectrometric properties to the compound of interest will cause interference.

Performance of Method:

Range of Application:
The analysis is quadratic with an allowed intercept for all parameters from the limit of Quantification to 1250ng/l

Limit of Quantification, Recoveries of Compounds and Uncertainty of measurement:

DETERMINAND	LOQ ng L^{-1}	UoM	DIRECT STANDARDS				ELVINGTON TREATED (HARD HARDNESS) 80% SPIKE (1µg L^{-1})	
			LOW STANDARD		HIGH STANDARD			
			RECOVERY	RSD	RECOVERY	RSD	RECOVERY	RSD
Phenol	89	± 7.67 %	99.1%	4.1%	99.5%	1.7%	100.0%	2.2%
2-Methylphenol	7	± 7.22 %	98.7%	2.4%	100.3%	2.2%	99.7%	1.9%
3-Methylphenol	29	± 7.97 %	100.7%	2.4%	100.6%	1.0%	101.9%	1.3%
4-Methylphenol	10	± 8.60 %	99.1%	2.7%	101.1%	2.6%	101.9%	2.3%
2-Chlorophenol	10	± 6.65 %	98.5%	1.5%	100.3%	1.4%	100.7%	1.2%
3-Chlorophenol	8	± 6.33 %	97.8%	1.5%	100.0%	1.6%	100.5%	2.0%
4-Chlorophenol	9	± 6.44 %	98.1%	1.7%	100.1%	1.7%	100.1%	2.0%
2,6-Methylphenol	8	± 6.90 %	98.9%	1.8%	100.2%	1.7%	100.5%	1.2%
2,5-Methylphenol	9	± 7.51 %	98.9%	1.3%	101.2%	1.7%	101.8%	1.8%

METHOD STATEMENT

DETERMINAND	LOQ ng L⁻¹	UoM	DIRECT STANDARDS				ELVINGTON TREATED (HARD HARDNESS) 80% SPIKE (1µg L⁻¹)	
			LOW STANDARD		HIGH STANDARD			
			RECOVERY	RSD	RECOVERY	RSD	RECOVERY	RSD
2,4-Methylphenol	8	± 7.55 %	99.0%	1.9%	100.5%	2.0%	100.9%	1.4%
3,5-Methylphenol	9	± 7.97 %	98.4%	1.9%	100.5%	2.5%	101.7%	1.8%
2,3-Methylphenol	10	± 7.49 %	98.5%	2.0%	101.1%	1.8%	101.6%	2.0%
3,4-Methylphenol	8	± 11.74 %	100.1%	4.5%	101.9%	3.6%	104.3%	3.7%
2-Chloro-5-methylphenol + 4-Chloro-2-methylphenol	16	± 7.55 %	100.9%	2.0%	100.5%	1.8%	101.2%	1.5%
2-Chloro-4-methylphenol	9	± 8.11 %	101.0%	2.2%	101.4%	1.6%	102.0%	1.5%
3-Chloro-4-methylphenol	7	± 8.05 %	101.5%	2.4%	101.2%	1.8%	102.1%	1.8%
2,6-Dichlorophenol	13	± 8.05 %	100.2%	2.4%	102.5%	3.8%	101.1%	2.4%
2,4-Dichlorophenol	11	± 7.15 %	101.3%	3.7%	100.9%	2.1%	101.2%	1.7%
2,3-Dichlorophenol	15	± 8.45 %	99.3%	2.0%	101.2%	2.4%	102.2%	1.9%
2,4,6-Trichlorophenol	14	± 8.90 %	101.2%	3.1%	103.3%	4.6%	100.6%	2.4%
2,4,5-Trichlorophenol	9	± 12.40 %	99.1%	4.7%	101.6%	4.4%	102.4%	3.6%

References:
In house Method