



METHOD STATEMENT

Determinand:

This method covers the determination of Nonylphenol, Octylphenol and Nonylphenol Ethoxylates.

Matrix:

Treated and untreated sewage effluents and surface waters

Principle of Method:

Alkylphenols and their ethoxylates are simultaneously converted to their Pentafluorobenzoyl esters and extracted into hexane by shaking 5.5mls of aqueous sample that has been buffered to high pH, with 2mls of a solution of 0.1% Pentafluorobenzoyl chloride in hexane. This process is automated using a robotic sampler. The extract is then analysed by gas chromatography with tandem mass spectrometry (GC-MSMS) using negative chemical ionisation (NCI). Quantitation is by internal standardisation against suitable, isotopically labelled, surrogate compounds.

Sampling and Sample Preparation:

Samples should be collected in 250ml glass bottles with PTFE lined caps and be stored at $3\pm 2^{\circ}\text{C}$. Before analysis, the filled bottles should be buffered to high pH using Sodium Hydroxide. Samples are stable for 5 days from sampling. Stability data for this method has been generated by an external laboratory as part of the CIP2 project.

Interferences:

GC-MS/MS is an extremely selective technique and interferences should only be encountered very rarely, however in theory, any compound that is extracted by the procedure, which has a GC retention time similar to the compound of interest, and which produces both parent and daughter ions similar to that of the compounds in question, may interfere.

Performance of Method:

Range of method: 0.04ug/l – 5ug/l for Nonylphenol and NPEO's. 0.01ug/l – 1ug/l for Octylphenol.

The uncertainty values are reported as P%, which is calculated as %Bias + 2x %RSD, as specified by the CIP2 project technical guidance.

Compound	Standards			
	Low Standard		High Standard	
	% Bias	% RSD	% Bias	% RSD
Nonylphenol	-1.40	7.14	4.95	9.09
NPEO1	-8.88	11.09	-3.46	7.35
NPEO2	-9.35	12.00	-2.84	7.75
NPEO3	0.23	12.89	-1.21	6.24
Octylphenol	1.40	7.70	6.81	8.58



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Compound	Treated Effluent				
	LOD (ug/l)	Low Spike		High Spike	
		% Bias	% RSD	% Bias	% RSD
Nonylphenol	0.021	-2.92	9.01	3.41	4.36
NPEO1	0.039	-6.99	12.05	-1.49	7.38
NPEO2	0.026	-13.14	14.32	-2.54	8.00
NPEO3	0.031	-0.71	10.37	1.52	7.57
Octylphenol	0.004	0.35	7.39	5.61	6.97

Compound	Surface Water		
	LOD (ug/l)	High Spike	
		% Bias	% RSD
Nonylphenol	0.025	2.28	3.57
NPEO1	0.026	-4.55	10.36
NPEO2	0.029	-5.05	8.68
NPEO3	0.031	2.74	6.46
Octylphenol	0.003	5.46	6.66

Compound	Uncertainty of Measurement (P %)
Nonylphenol	12.13
NPEO1	16.25
NPEO2	18.54
NPEO3	16.65
Octylphenol	19.55