METHOD STATEMENT

Determinand:
Pyridine

Matrix:
Trade and sewage effluents, crude sewage and final effluents

Principle of Method:
5ml of sample is extracted with 5ml of diethyl ether. Analysis of the extract is by capillary gas chromatography, followed by mass spectrometric detection in selected ion mode (GCMS-SIM). The internal standard method of quantitation is used.

Sampling and Sample Preparation:
Samples are provided in amber 40ml vials (STL79)
Samples are stored in the refrigerator at 3 ± 2°C.
Samples are stable for 22 days (In-House Data) from sampling.

Interferences:
Any substance with an equivalent GC retention time, and the same ions as those being monitored, which elicit a response from the Mass Selective Detector will interfere.

Performance of Method:
Range of Method: 0.1mg/l – 10 mg/l without dilution

Determinand	MCERTS Accred.	LOD	MRL	Low Std		High Std	
		mg/L	mg/L	%Bias	%RSD	%Bias	%RSD
Pyridine	✓	0.0469	0.1	-1.36	5.6	-0.36	3.49

Determinand	Finham Final Effluent		Huddersfield Crude		Coopers Bridge Final Effluent		Syngenta Trade Effluent	
	%Rec.	%RSD	%Rec.	%RSD	%Rec.	%RSD	%Rec.	%RSD
Pyridine Low Spike 2mg/l	99.32	5.27	98.43	4.70	99.07	5.60	101.66	10.45
Pyridine High Spike 8mg/l	98.16	2.58	98.86	2.79	98.65	3.86	97.67	4.35

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 %
Pyridine	14.37

References:
HP 6890 Series Gas Chromatograph Operating Manual Vol. 1. General Information G1530-90440.

Agilent 5975 Series MSD Operation Manual (G3170-90036)

MCERTS (Waters) Standard Version 3