

# 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). They must be taken without any significant headspace (when vial is inverted, air bubble no more than 6mm diameter).

Samples are stored in the refrigerator at  $3 \pm 2^\circ\text{C}$ .

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: 0.1 mg/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)

# METHOD STATEMENT



MCERTS (Waters) Standard Version 3  
EPA Method 5021A : Volatile Organic Compounds in Various Sample Matrices Using Equilibrium  
Headspace Analysis