METHOD STATEMENT

Determinand:
pH value

Matrix:
Sample Type: sludge and soil samples at 20°C

Principle of Method:
The pH of the sample is equal to Log10 1/[H+] and is measured directly on the sludge or on an aqueous extract of soil using a pH probe that has been previously calibrated using solutions of a known pH.
The pH value of liquid digested sludge is used to monitor digester performance. The pH value of sludges and soils is generally used to assess the application rate of sludge for disposal to land.

Sampling and Sample Preparation:
Sludge samples are normally received in pots and soil sample in bags. No preservation is required for pH.
Sludge, cake and unground soils are stored at 3±2 °C; if the sample is ground then this is stored at room temperature.
Soil samples are dried and ground according to method WSC15 prior to analysis. Sludge samples should be kept in sealed and preferably full containers with limited air space in order to minimise the possibility of gas exchange with the atmosphere e.g. ammonia and carbon dioxide.
Wet samples are stable for 7 days from sampling, once dried soil samples are stable for 3 years (BS ISO 18512: 2007). Samples should be measured as soon as possible in order to minimise possible effects from the above problems.

Interferences
Gross suspended matter, oil or grease may cause interference by masking part of the electrode surface. Solids are dispersed in the sample prior to taking the pH reading.
Above a pH of 12 the electrode response may not be linear for pH values. Also, if high sodium concentrations are present, the response for pH may not be perfectly linear above pH 10. It is unlikely that either of these cases will be encountered for the samples analysed by this method.

Performance of Method:

Range of Application:
The pH probe is calibrated using buffers at pH4, pH7 and pH10, which restricts the calibrated range of application to pH 4-10. However, due to the linear response of the pH probe within a larger range, results may be reported for pH values in the range pH 1-13 provided additional checks at these pH values are carried out. Normal Reporting Limit: To the nearest 0.1 pH units

Limit of Detection:
Not applicable

Recoveries of Compounds, Precision, Bias and Uncertainty of Measurement:
Spiking Recovery: Spiking is not applicable for pH

pH	Low Standard	High Standard	Liquid AQC	Soil AQC	Sand	Loam	Clay	Sludge
pH Value, pH Units	4.02	10.05	7.06	7.32	7.48	6.47	6.10	7.49
Total Standard Deviation, pH Units	0.02	0.02	0.02	0.03	0.04	0.06	0.04	0.10

pH	Low Standard	High Standard	Liquid AQC	Soil AQC	Sand	Loam	Clay	Sludge
Bias, %	0.021	0.045	0.059	0.005	-	-	-	-
Uncertainty, %	±0.061	±0.082	±0.094	±0.061	-	-	-	-

References:

Determination of the pH value of sludge soil, mud and sediment; and lime requirement of soil (1977). Methods for the examination of waters and associated materials HMSO - ISBN 0117512524.
WOP132 - PROCEDURE FOR MIXING OF WAKEFIELD SOIL AQCS PRIOR TO USE IN ANALYSIS