

# METHOD STATEMENT



## Determinand:

Kjeldahl Nitrogen and Total Phosphorus

## Matrix:

Sample Type: soils and sludges samples

## Principle of Method:

Samples are digested with concentrated sulphuric acid in the presence of sodium sulphate and a copper catalyst to convert nitrogen compounds present to ammonium sulphate. After digestion, the samples are pH adjusted whilst making up to 250ml and the ammonia present is determined colorimetrically by discrete autoanalysis according to waste anions method.

## Sampling and Sample Preparation:

Samples are normally received in sludge or soil pots, or re-sealable plastic bags.

Note: Ground samples are stored at room temperature. Sludge samples and wet soils are refrigerated at  $3\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ .

Soil samples are air dried and ground according to method WSC15 prior to analysis. Sludge samples are mixed to obtain as near a homogeneous sample as possible.

Samples are stable for 30 days (BS ISO 18512: 2007) from sampling.

## Interferences

The presence of nitrate and/or nitrite may lead to erratic results but this interference is ill-defined. At the levels of nitrate/nitrite generally encountered in sewage sludge it is unlikely that any important interference will occur.

## Performance of Method:

### Range of Application:

The range of application is 3.1 - 60 mg/l in the extract.

This range may be extended by sample dilution.

Normal reporting limit: 11 mg/kg

### Limit of Detection:

Method LOD: 10.07mg/kg

## Recoveries of Compounds, Bias and Uncertainty of measurement:

Sample type	Mean sample result ( $\mu\text{g/l}$ )	Bias (%)	RSD (%)	Spike recovery (%)	% Uncertainty
Low Standard	14.490	-0.40	2.18	-	4.75
High Standard	58.940	1.77	3.83	-	9.44
Sand	2803.107	-	4.46	107.40	16.33
Clay	2665.515	-	4.26	105.36	13.89
Loam	1642.201	-	6.35	106.64	19.33
Sludge	3.610	-	4.91	98.10	11.72

## References:

"Total nitrogen and total phosphorus in sewage sludges (1985)" Method C - Methods for the examination of waters and associated materials HMSO. ISBN: 011 7518883.

BS ISO 18512: 2007- Soil quality - Guidance on long and short term storage of soil samples.