METHOD STATEMENT

Determinand:
Determination of nitrogen present in water, in the form of free ammonia, ammonium, nitrite, nitrate and organic nitrogen compounds capable of conversion to nitrate

Matrix:
Sample Type: final effluents, trade discharges, crude sewage and other related waste waters.

Principle of Method:
Oxidation of ammonia, nitrite and many organic nitrogen-containing compounds to nitrate using persulphate (peroxodisulphate) in a buffered alkaline system by boiling at elevated pressure in a closed container.
The absorbance due to nitrate is then measured by a spectrophotometer at 210nm and converted to a Total Nitrogen value by interpolation from a nitrate calibration curve.
For Kjeldahl analysis, the TON (generated by waste water anions method by discrete analyser) is subtracted from the Total Nitrogen to give Kjeldahl Nitrogen.

Sampling and Sample Preparation:
Samples are taken in STL650 bottles (125 ml azlons containing 1 ml of 15% sulphuric acid).
Samples are stored at 3±2 °C until ready for analysis.
Acidified samples are stable for 1 month (ISO 5667 – 3:2012)
Samples not taken in containers with preservative have compromised stability; an appropriate sample comment should be added.

Interferences
Interference will be caused by suspended solids and any non-nitrate species scattering or absorbing UV at the frequency used for quantitation. Particulate matter in the digested sample must be removed by filtration before the ultra-violet absorbance measurement is carried out. Interference from nitrite is eliminated by the addition of sulphamic acid, and carbonate and hydroxyl interference is eliminated by acidification.
High concentrations of dissolved organic matter: samples should not be analysed undiluted if the COD of the test sample exceeds 120 mg/l O. Samples must be diluted appropriately based on their COD, regardless of their TN content therefore a raised reporting limit may be necessary in those cases.
As the interfering absorbance will most likely be across a range of wavelengths, the absorbance at 275nm is used to establish the presence of interference. Any sample for which the absorbance at 275nm exceeds 10% of the absorbance at 210nm must be re-prepped on a higher dilution (if unreacted compounds are believed to be the interference) or the reporting limit raised.

Performance of Method:

Range of Application:
MRV to 5mg/l N. This range may be extended by sample dilution.

Limit of Detection:
0.7317 mg/l

Recoveries of Compounds:

	Standards		Final Effluent		Trade Discharge		Crude Sewage	
	Low Std	High Std	Low Spike	High Spike	Low Spike	High Spike	Low Spike	High Spike
%Recovery	100.85	101.28	100.35	100.32	103.14	103.59	99.97	100.09
%RSD	2.38	1.24	2.59	1.24	4.25	1.41	3.67	1.13

References:

Water Quality – Determination of Nitrogen Using Oxidative Digestion with Peroxodisulphate, ISO/TC 147/SC2/WG1 N146.

HMSO Methods for the Examination of Waters and Associated Materials, Oxidised Nitrogen in Waters 1981, ISBN 0 11 7515930.

Water Quality – Determination of nitrogen – Part 1: Method using oxidative digestion with peroxodisulphate, ISO 11905-1: 1997