METHOD STATEMENT

Determinand:
Ammoniacal nitrogen

Matrix:
Effluents, leachates, waste water, industrial discharge and ground water

Principle of Method:
The ammonia-selective electrode uses a hydrophobic gas-permeable membrane to separate the sample solution from an electrode internal filling solution of ammonium chloride. Dissolved ammonium is converted to ammonia by raising the pH of the sample above 11 with sodium hydroxide. Ammonia then diffuses through the membrane and changes the internal solution pH, which is sensed by a pH electrode.

A chloride ion-selective electrode that serves as the reference electrode senses the fixed level of chloride in the internal solution. Potentiometric measurements are made with a dedicated ion meter. Samples are compared directly against a fresh calibration in order to calculate individual sample concentrations. The entire system is automated to ensure identical measurement conditions for all standards and samples.

Sampling and Sample Preparation:
Samples should be stored at room temperature and analysed as soon as possible. The addition of acid as a preservative should be avoided. Refrigeration of samples may delay the analysis until a stable room temperature is attained.
Excessive handling or filtering of the sample should be avoided.
Samples are stable for 6 days (In-House Data) from sampling.

Interferences:
False results may be obtained if the samples, standards and associated quality control solutions are not at the same temperature. Low results may be obtained if the samples are very acidic or have a buffering capacity sufficiently large to prevent the pH of the sample reaching 11 or higher. Amines give a positive interference.

Performance of Method:
Range of Application: 50 to 1000mg/l as N without dilution.
Limit of detection: 4.5645 mg/l as N
Normal reporting Level: 50 mg/l as N

Determinand	Low standard		High standard	
	Tot. RSD %	Bias %	Tot. RSD %	Bias %
Ammoniacal Nitrogen as N	3.52	5.33	4.19	2.46

Samples and Spiked Samples

Determinand		Final Effluent	Trade Effluent	Ground Water	Landfill Leachate
Ammoniacal Nitrogen as N	%RSD	4.35	3.52	4.29	3.62
	Rec. %	101.51	101.64	102.94	99.81

Determinand		Surface Water	Soil Leachate	Crude
Ammoniacal Nitrogen as N	%RSD	3.97	4.12	3.27
	Rec. %	102.40	102.32	102.66

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 (%)
Ammoniacal Nitrogen as N	7.74

Triggers:
There are three methods that run ammonia and they all have different ranges. To enable accurate results the dets get swapped depending on the result.
For example det 04Q limit of detection is <50mg/l so if the result was 7mg/l this will swap to det 017 as this limit of detection is <0.41mg/l and therefore we can report a result rather than a less than.

The ranges of each method/det are:
WAS036 det 017 is 0.41 - 75mg/l
WAS067 det 817 is 0.06 - 2mg/l
WAS055 det 04Q is 50 – 1000mg/l (without dilution)

The trigger parameters are as follows:
817 det – 04Q det when sample concentration is >15mg/l
017 det – 04Q det when sample concentration is >150mg/l
04Q det – 017 det when sample concentration is <50mg/l

References:
Standard Methods for the Examination of Water and Wastewater 20th Edition. ISBN 0-87553-235-7.

TitreFast software manual.

Orion Thermo Fisher Ammonia Electrode Instruction Manual, Rev A.