

## METHOD STATEMENT

### **Determinand:**

Determination of Ammoniacal Nitrogen, Total Oxidised Nitrogen, Nitrite Nitrogen, Chloride and Orthophosphate.

### **Matrix:**

Sample Type: Effluents and Wastewaters

### **Principle of Method:**

Certain analytes, when reacted with specific reagents, will form coloured complexes. The intensity of the colour formed is proportional to the original concentration of the analyte.

The intensity of colour within samples can be evaluated by comparison with standards containing known concentrations of the analyte, thus allowing the concentration within the sample to be assessed.

The sample volumes, reagent volumes, mixing speeds, colour development time and development temperature are all controlled automatically using a discrete autoanalyser, which significantly improves method performance over manual colour assessment.

Specific details of colorimetric reactions are given below.

#### **Nitrite**

Nitrite ions react with sulphanilamide and N-1-naphthylethylene diamine in acidic media to form a pink azo dye, the intensity of which is proportional to the concentration of nitrite present and which is measured at 520 nm by automated discrete colorimetric analysis.

#### **TON**

Nitrate is reduced to Nitrite using a copper/hydrazine reducing agent. The nitrite produced is then reacted as described above to give a value for the total oxidised nitrogen (nitrite plus nitrate) within the sample. Nitrate can then be calculated by difference.

#### **Ammonia**

In the presence of a Sodium Nitroprusside catalyst, ammonia reacts with salicylate ions to produce a blue indophenol dye, whose intensity is proportional to the concentration of ammonia present and which is measured at 656 nm by automated discrete colorimetric analysis.

#### **Chloride**

Chloride reacts with mercury TPTZ (2,4,6-Tripyridyl-1,3,5-triazine) to form the stable mercuric chloride and free TPTZ. Free TPTZ reacts with ferrous ions to produce a blue complex, which is measured at 480 nm by automated discrete colorimetric analysis.

#### **Orthophosphate**

In an acid medium, ammonium molybdate and antimony potassium tartrate react with orthophosphate to form an antimony-phosphor-molybdate complex. This is then reduced by ascorbic acid to form a blue complex, the intensity of which is proportional to the original concentration of orthophosphate ions present. The intensity of the colour formed is measured at a wavelength of 724 nm using automated discrete colorimetric analysis

### **Interferences:**

Interference may be caused to any determinand depending on the colour and nature of the sample

#### **Ammonia**

Interferences include aromatic amines, chloramines, ketones, aldehydes and alcohols

#### **Nitrate / Nitrite**

Amines, strong reducing agents and strong oxidising agents are the only substances known to interfere

#### **Chloride**

Interferences include thiocyanate, thiosulphate, cyanide, sulphide and sulphite. Iron in excess will mask the end point

#### **Phosphate**

Interferences include sulphide, arsenic and silicon



# METHOD STATEMENT



## Performance of Method:

### Range of Application:

Ammoniacal Nitrogen	LOD-75mg/l as N
Total Oxidised Nitrogen	LOD-40 mg/l as N
Nitrite Nitrogen	LOD-5 mg/l as N
Chloride	LOD-500 mg/l as Cl
Orthophosphate	LOD-15 mg/l as P

These ranges may be extended by sample dilution.

### Limit of Detection, Recoveries of Compounds and Uncertainty of measurement:

Analyte	LOD mg/l	MRV mg/l	Maximum Allowable Blank		Final Effluent		Final Effluent Filtered		Trade Discharge		Crude Sewage	
					Low Spike	High Spike	Low Spike	High Spike	Low Spike	High Spike	Low Spike	High Spike
Ammonia	0.2072	0.5	0.5	%Recovery	101.39	102.4	101.77	102.39	104.03	97.78	104.15	97.78
				%RSD	1.73	0.9	1.7	1.14	1.6	1.91	1.69	1.91
TON	0.3776	0.40	0.4	%Recovery	101.12	103.46	101.89	104.65	98.79	96.59	99.05	97.64
				%RSD	4.3	2.05	3.47	1.95	1.7	1.78	2.41	2.77
Phosphate	0.1037	0.20	0.2	%Recovery	93.65	92.67	96.07	94.06	98.13	91.88	98.28	91
				%RSD	2.2	1.78	2.32	2.23	2.95	2.22	1.83	1.84
Nitrite	0.0314	0.032	0.032	%Recovery	101.21	101.23	101.51	101.89	100.17	100.81	103.26	101.54
				%RSD	0.71	0.56	1.03	0.88	1.2	3.01	1.55	0.96
Chloride	4.448	10	10	%Recovery	98.83	92.54	99.35	92.92	100.66	104.73	100.01	104.93
				%RSD	0.87	0.57	2.17	0.78	0.83	0.86	0.99	0.86
Nitrate	0.384	0.4	0.4	%Recovery	100.89	103.9	101.66	103.82	98.62	96.06	98.53	97.15
				%RSD	4.33	2.32	3.86	2.2	1.96	2.19	2.72	3.11

## References:

In House method. Chemistries based on SCA blue books.  
Aquakem 600 user's guide

