METHOD STATEMENT

Determinand:

Determination of Ammoniacal Nitrogen, Total Oxidised Nitrogen, Nitrite Nitrogen, Chloride, Orthophosphate and Nitrate by calculation using Gallery Plus discrete analyser.

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 540 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 660 nm by automated discrete colorimetric analysis.

Chloride

Chloride ions are mixed with acid-chloride colour reagent containing mercury (II) thiocyanate and iron (III) nitrate. The released thiocyanate ions react with the iron (III) nitrate to give a reddish-brown coloured iron (III) thiocyanate complex which is measured spectrophotometrically at 480nm.

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 880 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, high TON/nitrite and silicon

Performance of Method:

High Level Range of Application:

Ammoniacal Nitrogen	LOD-60mg/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.

The Low Level ranges are a 1/10th of the above ranges.

Limit of Detection and Recoveries of Compounds:

High Level

					Final Effluent		Final Effluent Filtered		Trade Effluent		Crude Sewage	
Analyte	**LOD mg/l**	**MRV mg/l**	**Maximum Allowable Blank**		**Low Spike**	**High Spike**	**Low Spike**	**High Spike**	**Low Spike**	**High Spike**	**Low Spike**	**High Spike**
Ammonia	0.2585	0.5	0.5	%Recovery	100.97	105.01	103.13	104.73	100.20	103.25	104.76	103.76
				%RSD	5.57	0.80	0.99	2.70	7.39	3.83	1.42	0.96
TON	0.2329	0.40	0.4	%Recovery	103.92	98.62	105.8	102.31	109.11	106.77	105.77	97.08
				%RSD	4.33	4.42	3.93	1.56	3.70	1.78	3.67	4.61
Phosphate	0.1100	0.20	0.2	%Recovery	93.37	93.5	92.04	95.61	90.6	99.4	97.97	97.55
				%RSD	4.78	4.96	4.82	4.94	4.90	3.80	2.86	4.09
Nitrite	0.0147	0.020	0.02	%Recovery	99.46	94.89	99.67	98.65	104.17	98.91	104.19	101.66
				%RSD	4.24	4.77	3.99	4.95	2.58	0.87	4.97	4.74
Chloride	4.4185	10	10	%Recovery	106.53	100.54	109.35	101.90	102.56	102.92	108.2	103.25
				%RSD	4.53	2.81	3.82	3.84	3.74	2.87	4.10	3.22
Nitrate	0.3633	0.40	0.4	%Recovery	107.98	103.16	109.91	105.7	109.72	107.75	105.96	96.51
				%RSD	4.59	4.90	4.38	1.96	4.12	1.95	4.07	5.03

Low Level

					Final Effluent		Final Effluent Filtered		Trade Effluent		Crude Sewage	
Analyte	**LOD mg/l**	**MRV mg/l**	**Maximum Allowable Blank**		**Low Spike**	**High Spike**	**Low Spike**	**High Spike**	**Low Spike**	**High Spike**	**Low Spike**	**High Spike**
Ammonia	0.0316	0.075	0.075	%Recovery	95.7	96.77	98.48	99.32	102.76	102.19	103.13	104.33
				%RSD	2.37	3.05	4.24	3.01	4.08	2.04	3.62	1.63
TON	0.0590	0.075	0.075	%Recovery	96.89	103.35	96.68	103.27	105.6	94.46	-	-
				%RSD	4.18	2.26	4.32	4.06	4.33	4.40	-	-
Phosphate	0.0245	0.025	0.025	%Recovery	92.72	96.75	91.54	96.2	98.33	99.87	99.95	102.07
				%RSD	4.89	4.50	4.76	4.03	4.76	2.97	3.39	4.47
Nitrite	0.00481	0.005	0.005	%Recovery	99.19	95.68	101.33	98.43	104.81	101.76	91.91	94.38
				%RSD	4.17	4.84	3.66	2.47	3.76	1.93	3.78	3.45
Chloride	0.337	0.50	0.5	%Recovery	103.57	95.24	105.18	95.68	104.83	95.61	105.4	96.07
				%RSD	2.76	2.27	1.94	2.56	2.80	2.10	2.78	1.92
Nitrate	0.0612	0.075	0.075	%Recovery	102.75	107.7	102.74	107.45	105.71	94.39	-	-
				%RSD	4.95	4.56	4.88	4.77	4.63	5.01	-	-

References:

In House method. Chemistries based on SCA blue books.
Gallery Plus Operation & Reference Manual 5.3A