xylem

Method Abstract
OI Analytical, a Xylem brand • 4178-01

Flow Solution™ FS 3700 Automated Chemistry Analyzer Total Dissolved Nitrogen by Segmented Flow Analysis (SFA), USEPA 353.2

WATER & WASTEWATER SERIES

Cartridge Part Number: 330955CT
Channel Part Number: 330954

Scope and Application

This method is used for the determination of total dissolved nitrogen in drinking water, surface water, and domestic and industrial wastes, according to USEPA Method 353.2.[1, 2, 3]

Method Performance

Range	0.01 – 20 mg/L
Rate	20 samples/hour
Precision	≤2% RSD at mid-point range
Method Detection Limit (MDL)	0.006 mg/L N as NO_3^-

The range may be extended to analyze other concentrations by changing the size of the sample loop.

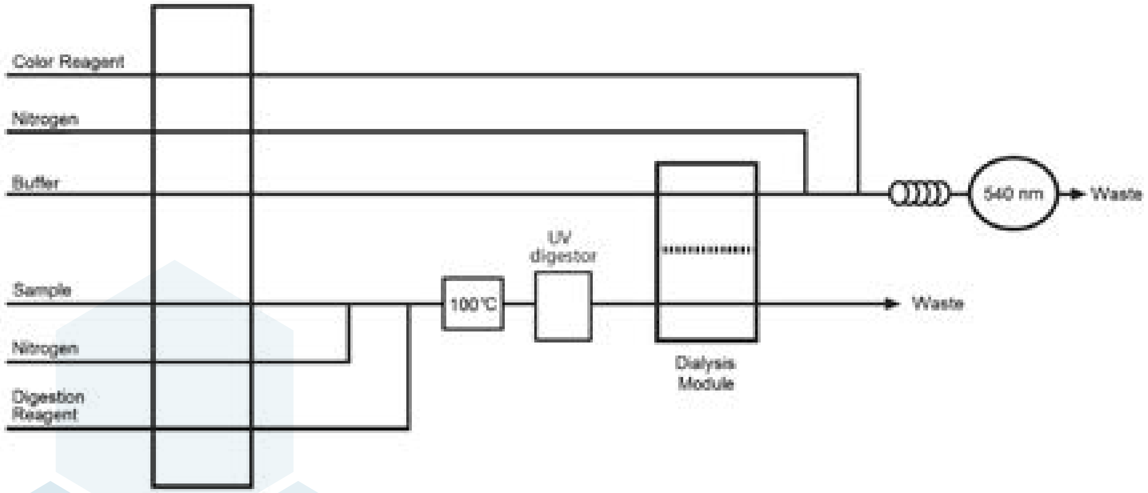

Figure 1. General Flow Diagram for Total Dissolved Nitrogen by USEPA 353.2

O·I·Analytical
a xylem brand

Reagents and Calibrants

Chemical Name	CAS #	Chemical Formula	Part Number
Ammonium sulfate	7783-20-2	$(NH_4)_2SO_4$	
Ascorbic acid	50-81-7	$C_6H_8O_6$	
Boric acid	10043-35-3	H_3BO_4	
Brij®-35	9002-92-0	$(C_2H_4O)_nC_{12}H_{26}O$	326126
Cupric sulfate pentahydrate	7758-99-8	$CuSO_4 \bullet 5H_2O$	
Ethylenediaminetetraacetic acid, disodium salt dihydrate (EDTA)	6381-92-6	$C_{10}H_{16}N_2Na_2O_8 \bullet 2H_2O$	
Glycine	56-40-6	NH_2CH_2COOH	
Hydrochloric acid	7647-01-0	HCl	
Imidazole	288-32-4	$C_3H_4N_2$	
N-(1-Naphthyl)ethylenediamine dihydrochloride	1465-25-4	$C_{12}H_{14}N_2 \bullet 2HCl$	
Phosphoric acid	7664-38-2	H_3PO_4	
Potassium nitrate	7757-79-1	KNO_3	
Potassium nitrite	7758-09-0	KNO_2	
Potassium persulfate	7727-21-1	$K_2S_2O_8$	
Sodium hydroxide	1310-73-2	$NaOH$	A001103
Sulfanilamide	63-74-1	$C_6H_8N_2O_2S$	
Water, deionized		H_2O	
Urea	57-13-6	H_2NCONH_2	
Additionally, the following chemicals may be needed for sample preservation or treatment			
Chloroform	67-66-3	$CHCl_3$	

Summary of USEPA Methods 353.2[1]

Method

- Dissolved nitrogen compounds are converted to nitrate by persulfate oxidation and subsequent UV digestion in a caustic solution of boric acid (pH 14). Nitrate is reduced quantitatively to nitrite by a cadmium-copper reductor in the form of an open tube cadmium reactor. The nitrite formed is diazotized with sulfanilamide and subsequently coupled with N-(1-naphthyl)ethylenediamine dihydrochloride. This reaction takes place in an acidic solution (pH 2).
The resulting highly colored azo dye is colorimetrically detected at 540 nm.[1]

Interferences

- Turbid samples may interfere with the photometric detector's ability to measure the true absorbance of the sample. Filter turbid samples prior to analysis.
- Iron, copper, and other metals may interfere with the analysis by binding with the nitrate and/or nitrite in the sample, thus blocking the color formation reaction. Use of EDTA or other complexing agents in the buffering solution manages this interference.

NOTE: The alternative imidazole buffer solution is particularly adept at eliminating these interferences, and should be utilized whenever possible.

- Samples that are outside the functional pH range of the buffering solution may affect the results obtained from this method. Adjust the pH of these samples to pH 5-9 using either concentrated hydrochloric acid (HCl) or sodium hydroxide (NaOH).

- Oil and grease will coat the cadmium surface, thus reducing its reduction efficiency. Extract samples containing large concentrations of oil and grease with an appropriate organic solvent.
- Sulfide in the presence of cadmium will form cadmium sulfide (CdS), which will inhibit nitrate reduction. Samples containing sulfide cannot be determined using this method without first removing the sulfide by precipitation with cadmium salts.
- Degas all reagents prior to analysis. Dissolved oxygen and carbonate can react with the cadmium to form cadmium hydroxide ($Cd(OH)_2$) and cadmium carbonate ($CdCO_3$) precipitants. Additionally, dissolved oxygen competitively inhibits the reduction of nitrate to nitrite.
- Care must be taken to ensure that the pH never exceeds 8.5.
- Chlorine may reduce the reduction efficiency of the cadmium reactor. Samples that may contain residual chlorine should be tested for reduction efficiency. When necessary, dechlorinate samples with sodium thiosulfate ($Na_2S_2O_3$).
- Norwitz and Keliher, as well as Nydahl have compiled a comprehensive study of interferences in the spectrophotometric analysis of nitrite.[4,5,6]
- Other compatible procedures for removing or suppressing interferences may be used, provided they do not adversely affect overall method performance.
- Method interferences can be caused by contaminants in the reagents, reagent water, and glassware, which may bias the results. Take care to keep all such items free of contaminants.

Safety

1. The toxicity or carcinogenicity of each compound or reagent used in this method has not been fully established. Each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level.
2. For reference purposes, a file of Safety Data Sheets (SDS) for each chemical used in this method should be available to all personnel involved in this chemical analysis. The preparation of a formal safety plan is advisable.
3. Chemicals used in this method may be highly toxic or hazardous and should be handled with extreme caution at all times. Consult the appropriate SDS before handling.
4. Unknown samples may be potentially hazardous and should be handled with extreme caution at all times.
5. Proper personal protective equipment (PPE) should be used when handling or working in the presence of chemicals.
6. This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

Sample Handling and Preservation

1. Collect samples in plastic or glass bottles with minimal headspace that have been thoroughly cleaned and rinsed with reagent water.
2. Ensure the volume of sample collected is sufficient to obtain a representative sample, analyze replicates, and minimize waste disposal.
3. Preserve samples by adding concentrated sulfuric acid (H_2SO_4) to a pH <2 and refrigerating the samples at 2-6 °C from the time of collection. Do not preserve samples with mercuric chloride.
4. Perform sample analysis as soon as possible to eliminate analyte loss. If storage is required, hold preserved samples that have been refrigerated at 2-6 °C for a maximum of 28 days from the time of collection.

References

1. Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Cadmium Reduction). Methods for the Determination of Inorganic Substances in Environmental Samples; EPA/600/R-93/100; U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1993; Method 353.2.

2. Standard Method for the Examination of Water and Wastewater, 21st ed.; American Public Health Association: Washington, D.C., 2005.

3. Water Quality–Determination of Nitrite Nitrogen and Nitrate Nitrogen and the Sum of Both by Flow Analysis (CFA and FIA) and Spectrophotometric Detection. International Standard; ISO 13395:1996(E); 1st ed.; Geneva, Switzerland, 1996.

4. Nydahl, F. Talanta 1976, 23, 349–357.

5. Norwitz, G.; Keliher, P.N. Study of Interferences in the Spectrophotometric Determination of Nitrite Using Composite Diazotization-Coupling Reagents. Analyst 1985, 110, 689–694.

6. Norwitz, G.; Keliher, P.N. Study of Interferences in the Spectrophotometric Determination of Nitrite Using Composite Diazotization-Coupling Reagents. Analyst 1986, 111, 1033–1037.

7. Flow Solution™ 3700 Operator's Manual (PN 329998). Available from OI Analytical, P.O. Box 9010, College Station, TX, 77842–9010.

8. Fox, J.B. J. Anal. Chem. 1979, 51, 1493.

OI Analytical, a Xylem brand
PO Box 9010
College Station, TX 77842-9010

☎ +1.979.690.1711
✉ xylem-lab@xyleminc.com
oico.com

oico.com/fs3700