Flow Solution FS3700 automated chemistry analyzer total dissolved nitrogen by Segmented Flow Analysis (SFA), USEPA 353.2

Segmented Flow Analysis (SFA) 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]

Table 1. 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.

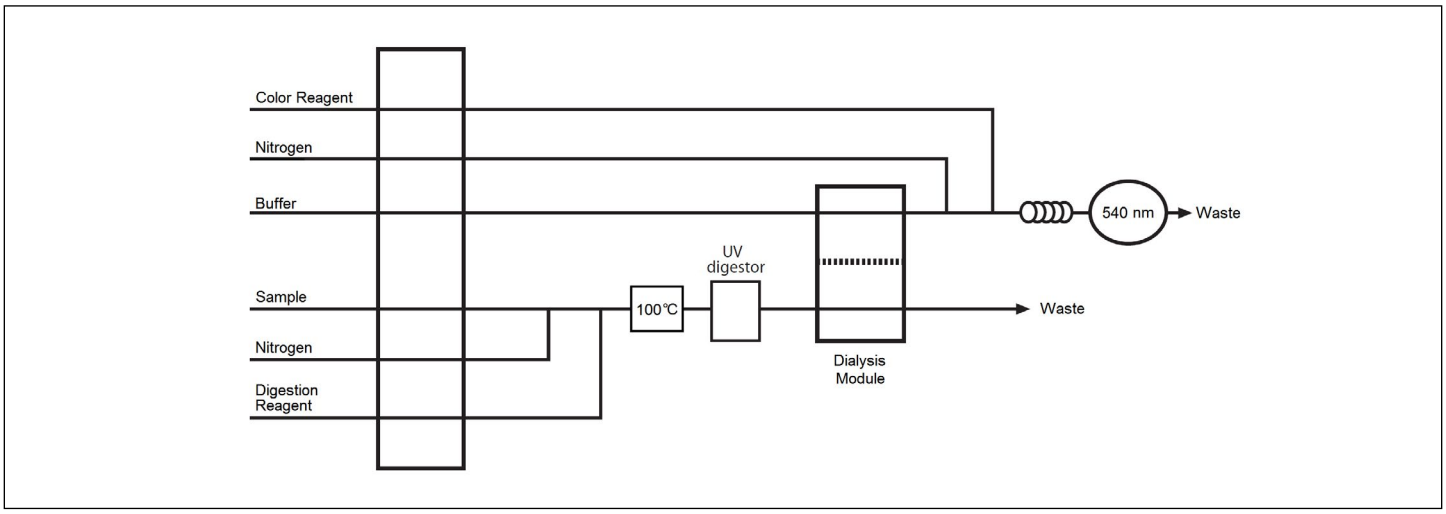


Figure 1. General flow diagram for Total Dissolved Nitrogen by USEPA 353.2

Table 2. Reagents and calibrants

Chemical name	Cas #	Chemical formulas	Part number
ammonium sulfate	7783-20-2	$(NH_4)_2SO_4$	n/a
i-ascorbic acid	50-81-7	$C_6H_8O_6$	n/a
boric acid	10043-35-3	H_3BO_3	n/a
Brij®-35	9002-92-0	$(C_2H_4O)_nC_{12}H_{26}O$	326126
cupric sulfate pentahydrate	7758-99-8	$CuSO_4 \cdot 5H_2O$	n/a
ethylenediaminetetraacetic acid, disodium salt dihydrate (EDTA)	6381-92-6	$C_{10}H_{16}N_2Na_2O_8 \cdot 2H_2O$	n/a
glycine	56-40-6	$C_2H_5NO_2$	n/a
hydrochloric acid	7647-01-0	HCl	n/a
imidazole	288-32-4	$C_3H_4N_2$	n/a
N-(1-naphthyl) ethylenediamine dihydrochloride	1465-25-4	$C_{12}H_{14}N_2 \cdot 2HCl$	n/a
phosphoric acid	7664-38-2	H_3PO_4	n/a
potassium nitrate	7757-79-1	KNO_3	n/a
potassium nitrite	7758-09-0	KNO_2	n/a
potassium persulfate	7727-21-1	$K_2S_2O_8$	n/a
sodium hydroxide	1310-73-2	NaOH	A001103
sulfanilamide	63-74-1	$C_6H_8N_2O_2S$	n/a
water, deionized	7732-18-5	H_2O	n/a
urea	57-13-6	CH_4N_2O	n/a
Additionally, the following chemicals may be needed for sample preservation or treatment			
chloroform	67-66-3	$CHCl_3$	n/a

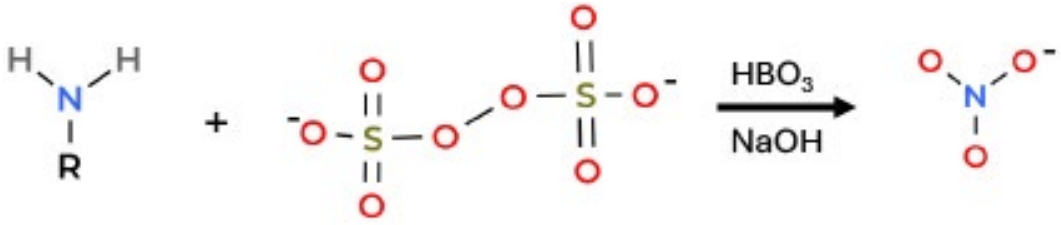
Summary of USEPA methods 353.2[1]

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]

Example reactions

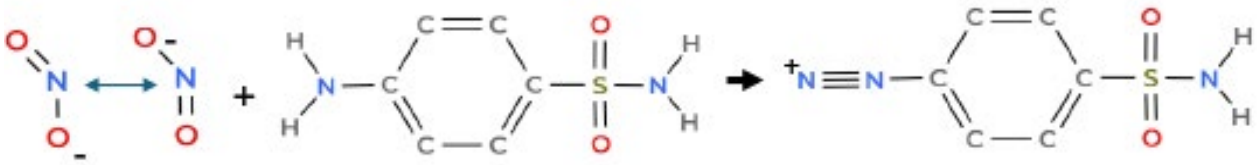
First, the organic nitrogen species are oxidized by sodium persulfate at high, buffered pH to form nitrate ion.
The pH drops as the persulfate is consumed and converts to bisulfate ions.



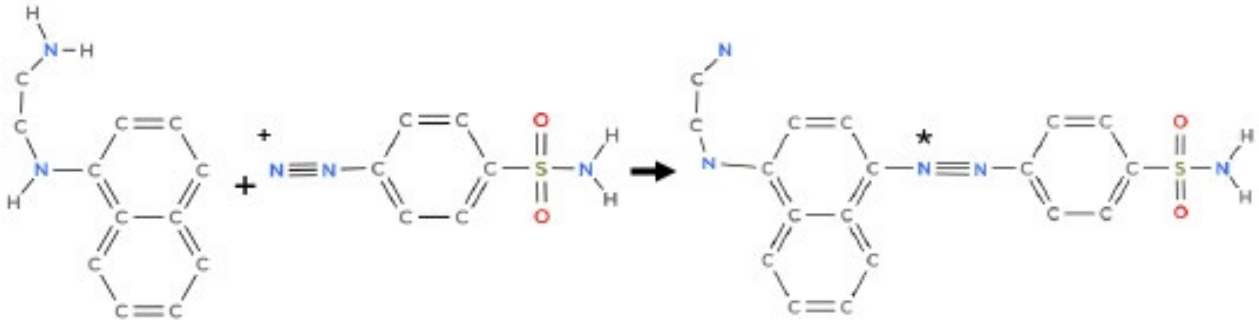
Second, in the open-tube cadmium reactor, nitrate ions are reduced to nitrite ions.



Third, sulfanilamide is used to diazotize the nitrite ion, forming a diazonium ion.



Fourth, the diazonium ion is treated with N-(1-naphthyl)ethylenediamine to form a strongly-colored azo dye, that absorbs light at 540 nm. This is the species detected by the photometric detector and the nitrogen atom from the original sample is marked with an asterisk.



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 ($Na_2S_2O_3$).

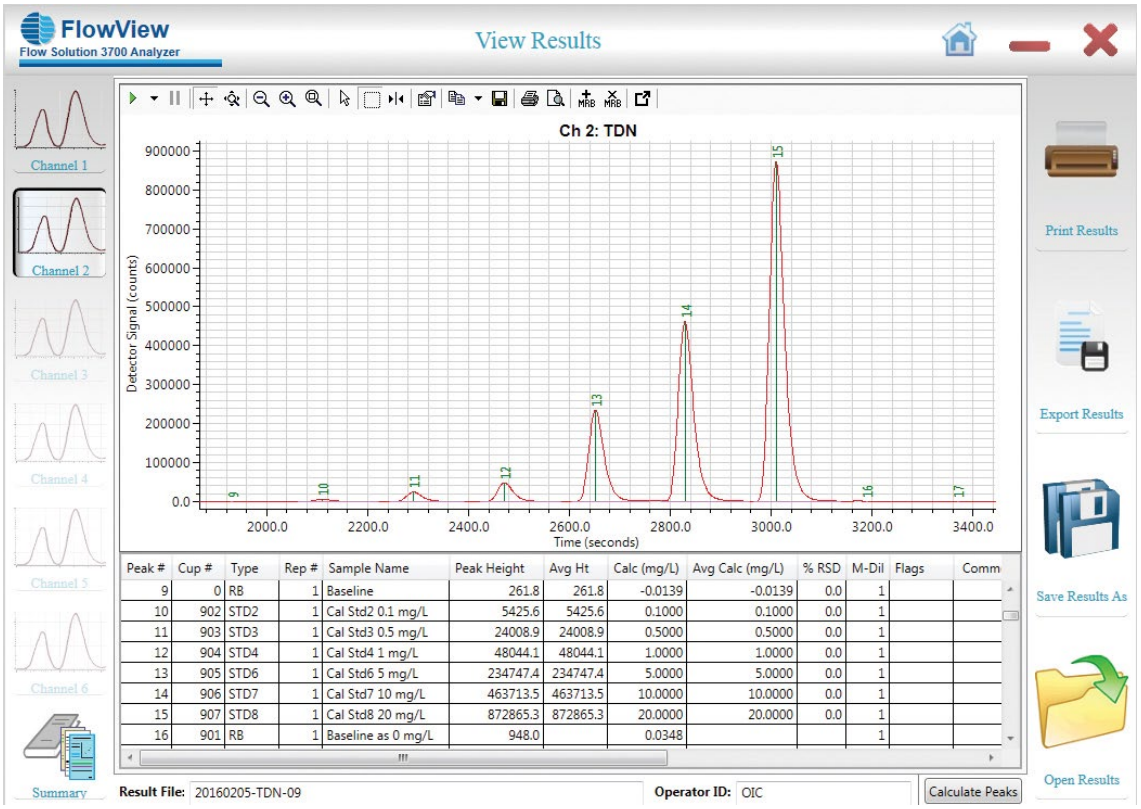
- Norwitz and Keliher, as well as Nydahl have compiled a comprehensive study of interferences in the spectrophotometric analysis of nitrite. (Reference 4,5,6)

- 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.

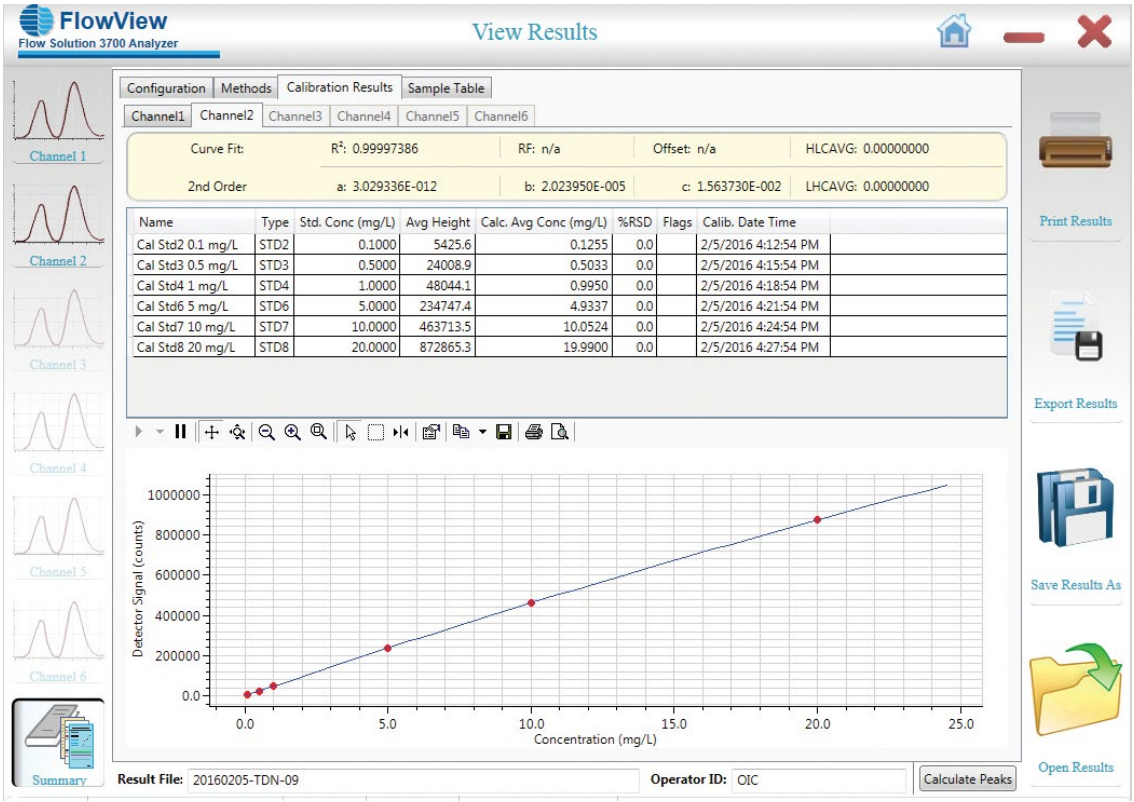
Performance and data quality

Figure 3. Total nitrogen calibration series



Peak #	Cup #	Type	Rep #	Sample Name	Peak Height	Avg Ht	Calc (mg/L)	Avg Calc (mg/L)	% RSD	M-Dil	Flags	Comm
9	0	RB	1	Baseline	261.8	261.8	-0.0139	-0.0139	0.0	1		
10	902	STD2	1	Cal Std2 0.1 mg/L	5425.6	5425.6	0.1000	0.1000	0.0	1		
11	903	STD3	1	Cal Std3 0.5 mg/L	24008.9	24008.9	0.5000	0.5000	0.0	1		
12	904	STD4	1	Cal Std4 1 mg/L	48044.1	48044.1	1.0000	1.0000	0.0	1		
13	905	STD6	1	Cal Std6 5 mg/L	234747.4	234747.4	5.0000	5.0000	0.0	1		
14	906	STD7	1	Cal Std7 10 mg/L	463713.5	463713.5	10.0000	10.0000	0.0	1		
15	907	STD8	1	Cal Std8 20 mg/L	872865.3	872865.3	20.0000	20.0000	0.0	1		
16	901	RB	1	Baseline as 0 mg/L	948.0		0.0348			1		

Figure 4. Calibration curve and statistics



Name	Type	Std. Conc (mg/L)	Avg Height	Calc. Avg Conc (mg/L)	%RSD	Flags	Calib. Date Time
Cal Std2 0.1 mg/L	STD2	0.1000	5425.6	0.1255	0.0		2/5/2016 4:12:54 PM
Cal Std3 0.5 mg/L	STD3	0.5000	24008.9	0.5033	0.0		2/5/2016 4:15:54 PM
Cal Std4 1 mg/L	STD4	1.0000	48044.1	0.9950	0.0		2/5/2016 4:18:54 PM
Cal Std6 5 mg/L	STD6	5.0000	234747.4	4.9337	0.0		2/5/2016 4:21:54 PM
Cal Std7 10 mg/L	STD7	10.0000	463713.5	10.0524	0.0		2/5/2016 4:24:54 PM
Cal Std8 20 mg/L	STD8	20.0000	872865.3	19.9900	0.0		2/5/2016 4:27:54 PM

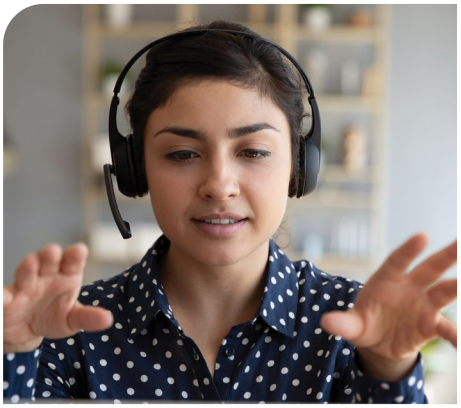
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1) the tissue in plants that brings water and nutrients upward from the roots.
2) a leading provider of unique solutions for your lab.

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