

Flow Solution™ FS3700 Automated Chemistry Analyzer

Nitrate plus Nitrite Nitrogen or Nitrite Nitrogen by Segmented Flow Analysis (SFA) or Flow Injection Analysis (FIA), USEPA 353.2

Cartridge Part Numbers 331376CT (SFA) and 330093CT (FIA)

Scope and Application

This method is used for the determination of nitrate (NO_3^-) plus nitrite (NO_2^-) or nitrite singly in drinking water, groundwater, surface water, and domestic and industrial wastes according to **USEPA Method 353.2, Standard Methods 4500- NO_3^- I and 4500- NO_3^- F** and **ISO Method 13395**.^{1,2,3} This method includes information for both FIA and SFA.

Method Performance	SFA	FIA
Range	0.005 – 10 mg/L	0.005 – 10 mg/L
Rate	40 samples/hour	48 samples/hour
Precision	1% RSD at mid-point of range	1% RSD at mid-point of range
Method Detection Limit (MDL)	0.001 mg/L	0.001 mg/L

For SFA analysis, the range may be extended to analyze other concentrations by modifying the **Load Time** (see page 5). For FIA analysis, this may be accomplished by changing the size of the sample loop.

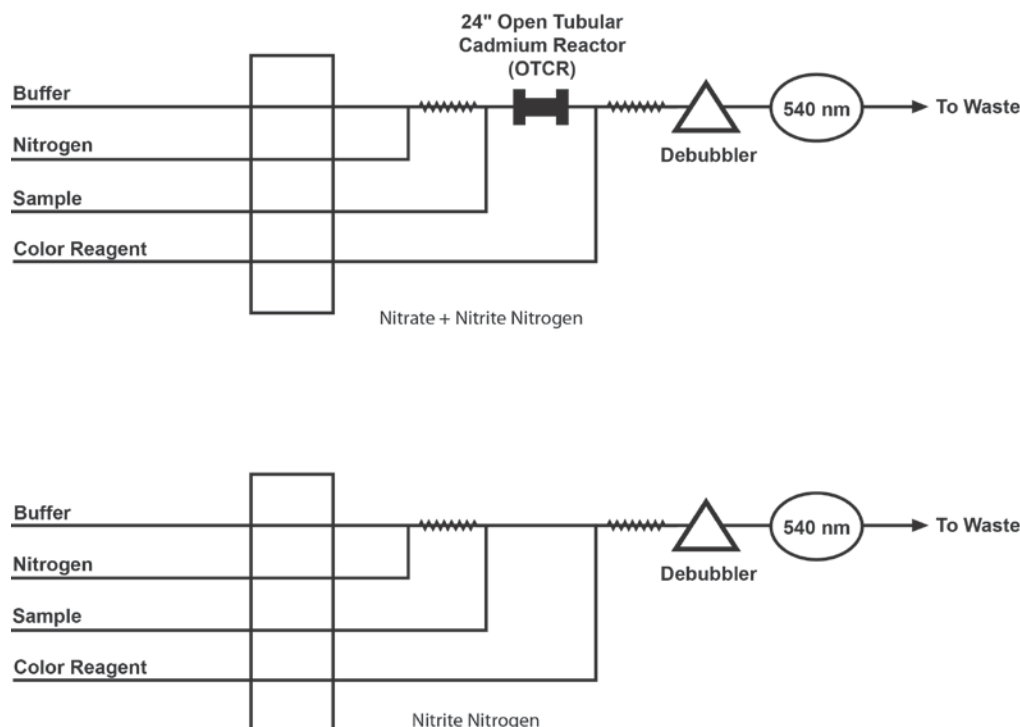


Figure 1. General SFA Flow Diagram for Nitrate plus Nitrite Nitrogen and Nitrite Nitrogen by USEPA 353.2

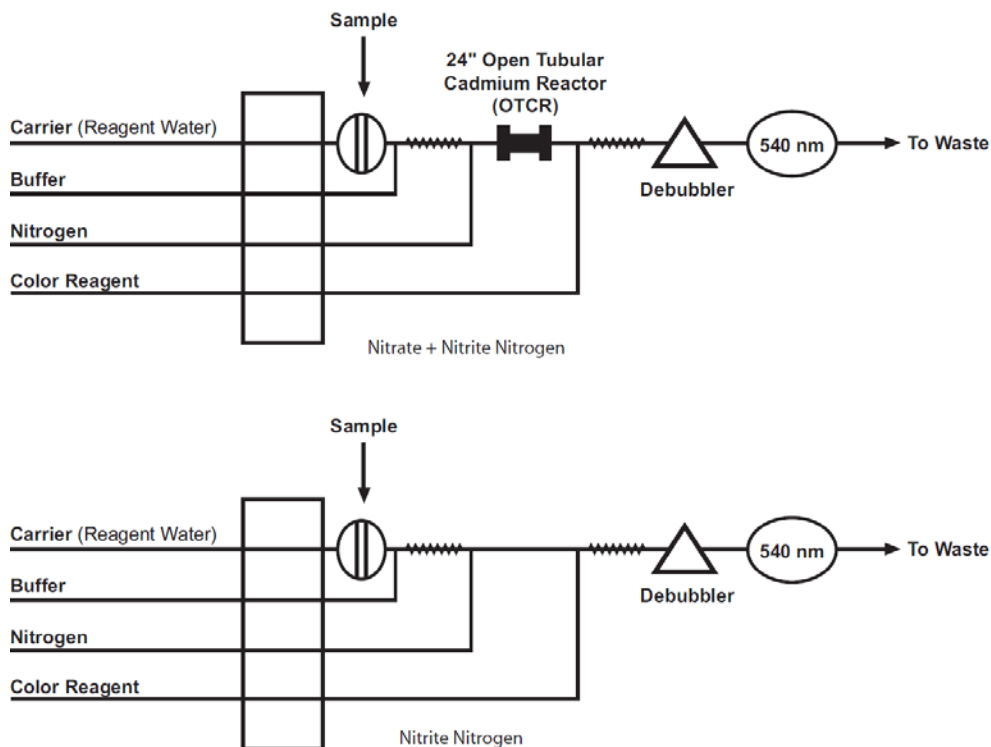


Figure 2. General FIA Flow Diagram for Nitrate plus Nitrite Nitrogen and Nitrite Nitrogen by USEPA 353.2

Reagents and Calibrants

Chemical Name	CAS #	Chemical Formula	Part Number
Ammonium chloride	12125-02-9	NH ₄ Cl	
Ammonium hydroxide	1336-21-6	NH ₄ OH	
Brij [®] -35 (21% solution)	9002-92-0	(C ₂ H ₄ O) _n C ₁₂ H ₂₆ O	326126
Cupric sulfate pentahydrate	7758-99-8	CuSO ₄ • 5H ₂ O	
Ethylenediaminetetraacetic acid, disodium salt dihydrate (EDTA)	6381-92-6	C ₁₀ H ₁₆ N ₂ Na ₂ O ₈ • 2H ₂ O	
Hydrochloric acid	7647-01-0	HCl	
Imidazole	288-32-4	C ₃ H ₄ N ₂	
N-(1-Naphthyl)ethylenediamine dihydrochloride	1465-25-4	C ₁₂ H ₁₄ N ₂ • 2HCl	
Phosphoric acid	7664-38-2	H ₃ PO ₄	
Potassium nitrate	7757-79-1	KNO ₃	
Potassium nitrite	7758-09-0	KNO ₂	
Sulfanilamide	63-74-1	C ₆ H ₈ N ₂ O ₂ S	
Water, deionized		H ₂ O	
Additionally, the following chemicals may be needed for sample preservation or treatment			
Chloroform	67-66-3	CHCl ₃	

Summary of USEPA Method 353.2

Method

- A filtered sample is passed through a column to quantitatively reduce nitrate to nitrite using cadmium metal. Nydahl provides a good discussion of nitrate reduction by cadmium metal.⁴ The nitrite (that was originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and subsequently coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye that is measured colorimetrically at 540 nm.
- Measure nitrite singly by performing the same analysis, but without the cadmium reduction coil. Nitrate is not reduced to nitrite without cadmium and is not detected because only nitrite forms the azo dye.
- The value for nitrate may be calculated by subtracting the nitrite value from the combined nitrate plus nitrite value.

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 ($\text{Cd}(\text{OH})_2$) 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 ($\text{Na}_2\text{S}_2\text{O}_3$).
- 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.

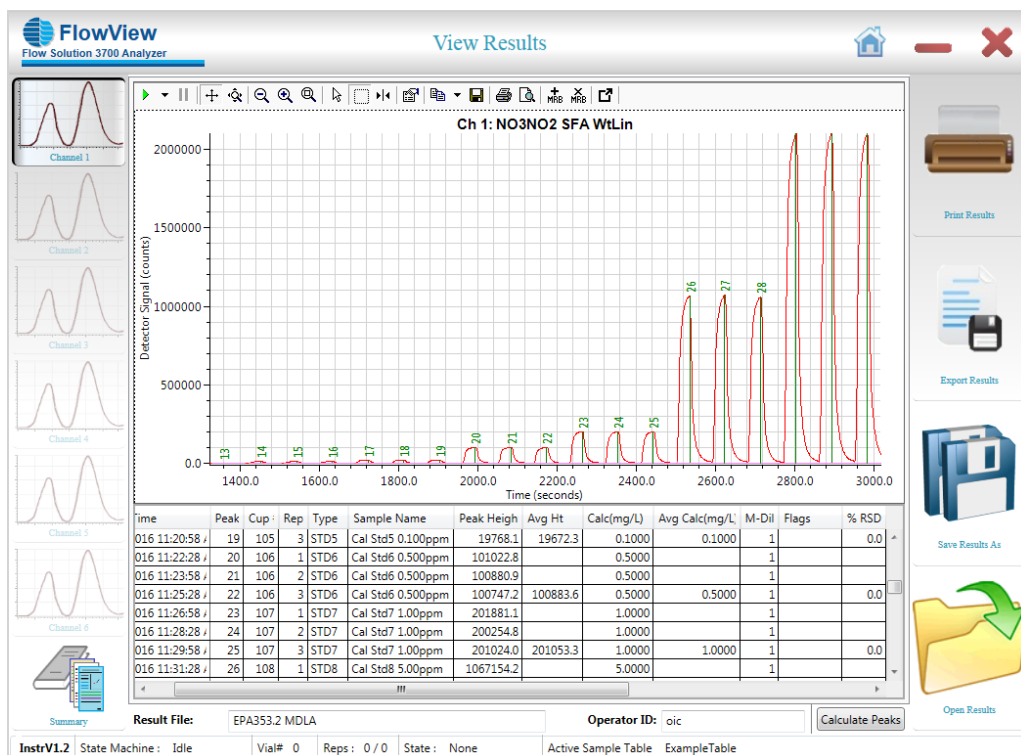


Figure 5. Nitrate Plus Nitrite Calibration Series (SFA)

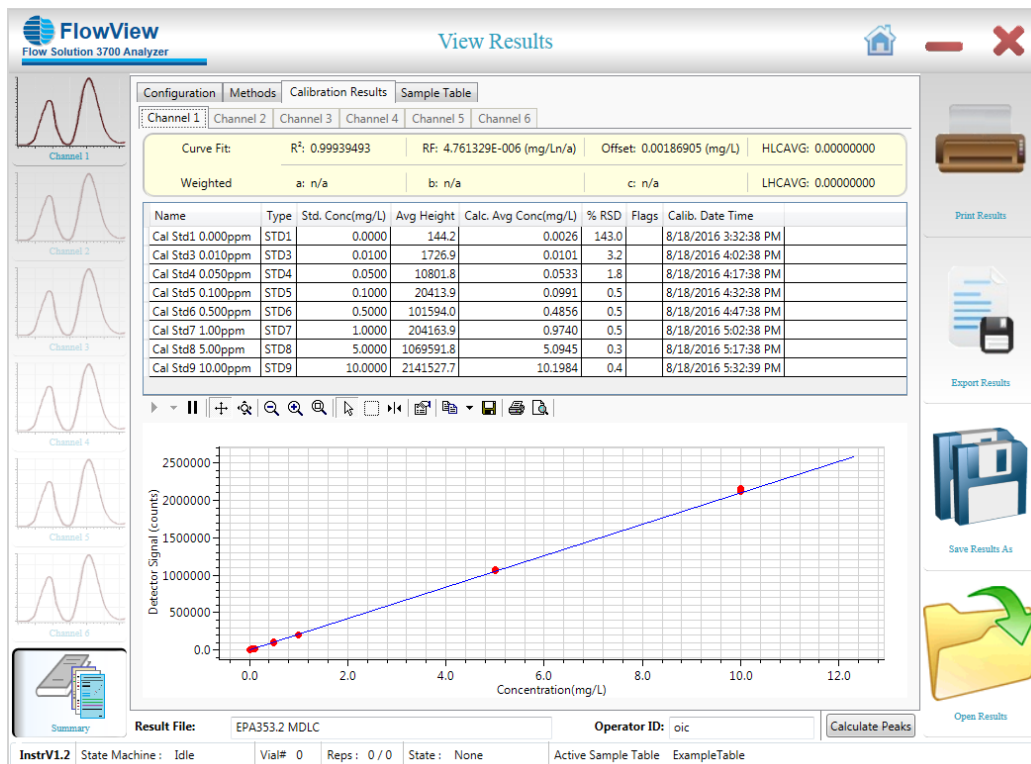


Figure 6. Calibration Curve and Statistics (SFA)

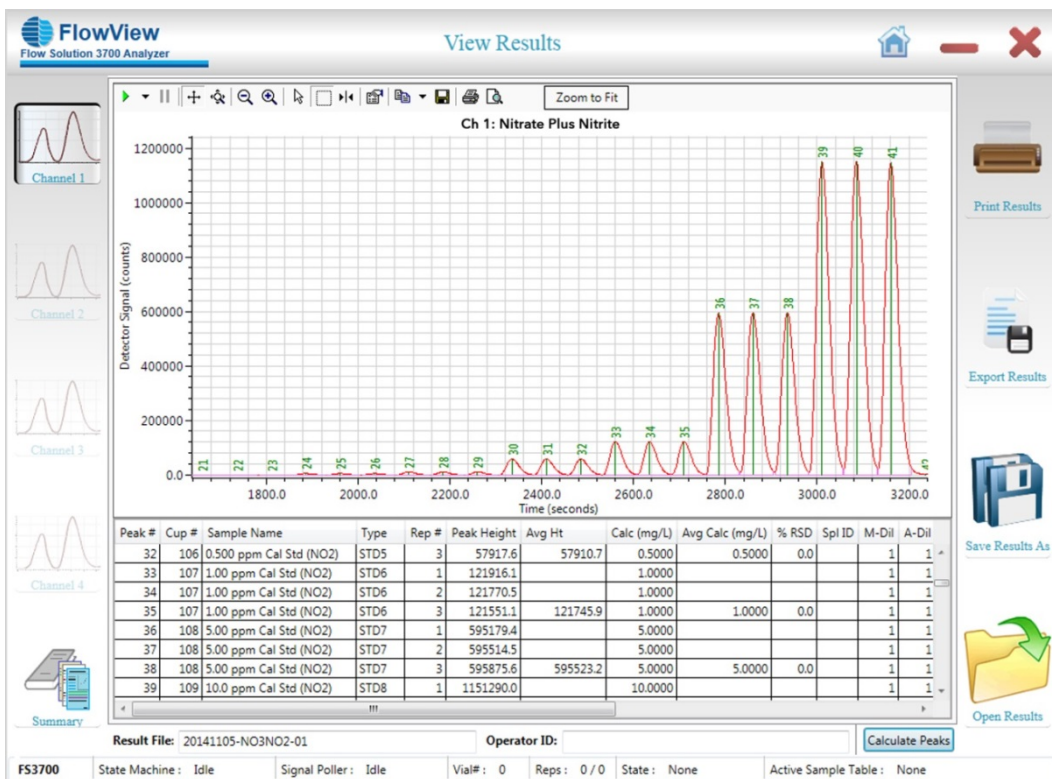


Figure 7. Nitrate Plus Nitrite Calibration Series (FIA)

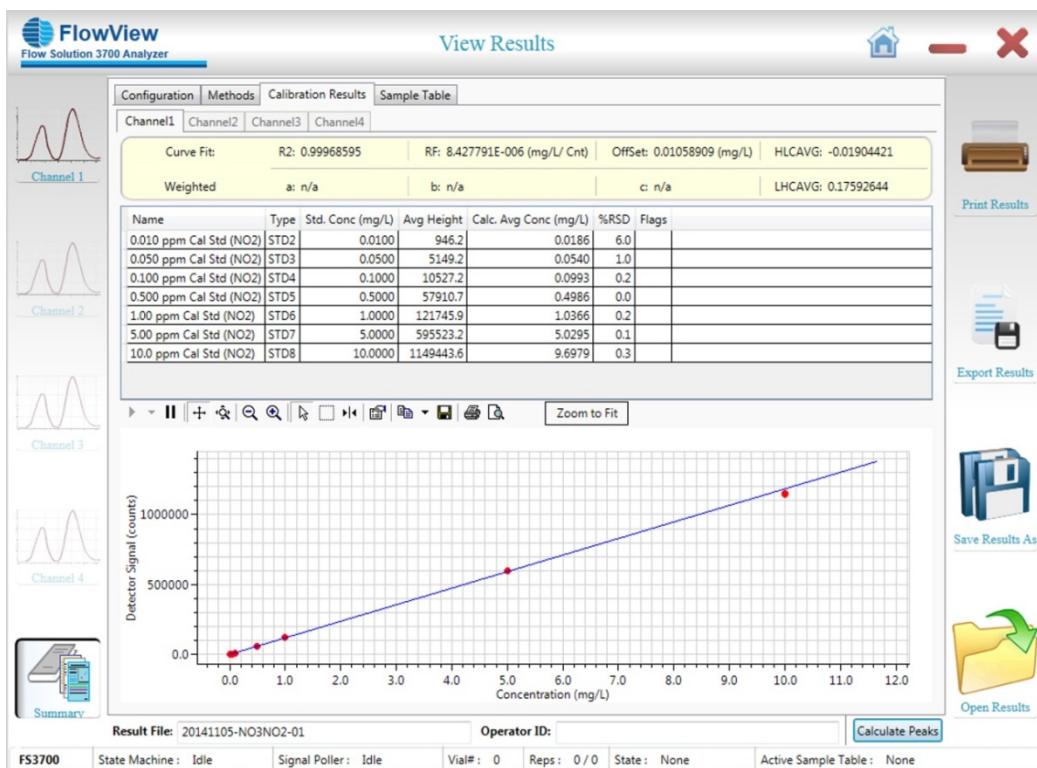


Figure 8. Calibration Curve and Statistics (FIA)

Table 1. Consumables and Spare parts for Nitrate/Nitrite by USEPA 353.2

Consumable	Part Number
FIA pump tubes kit - Nitrate/Nitrite by USEPA 353.2	330093TK
SFA pump tubes kit - Nitrate/Nitrite by USEPA 353.2	331376TK
Brij®-35 (21% solution)	326126
24" OTCR (Cadmium coil assembly) – with nut and ferrule	A000897
Nitrogen Pillow Assembly	A000811
100 µL Injection/Bypass Loop	285676
PEEK Autosampler Probe for RA/3090/3360 Sampler	325331

Pump tubes should be replaced monthly or as-needed to maintain system performance. Maximum life expectancy for pump tubes is approximately 800 hours.



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