Scope

This method is used for the determination of nitrite nitrogen in drinking water, groundwater, surface water, and domestic and industrial wastes according to USEPA Method 353.2 and Standard Methods 4500–NO_3^- I. Additionally, this method enables nitrite analysis according to ISO Method 13395. This method can also be used for the determination of nitrite nitrogen in Potassium chloride (KCl) extracts of soils and plants.

Summary

Diazotize any nitrite originally present in the sample with sulfanilamide and subsequently couple with *N*-(1-naphthyl)ethylenediamine dihydrochloride. Colorimetrically detect the resulting highly colored azo dye at 540 nm. A calibration curve allows accurate quantitation of the detected nitrite.

Interferences

Filter turbid samples prior to analysis. Iron, copper, and other metals may interfere with the analysis by binding with the nitrite in the sample, blocking the color formation reaction; eliminate this interference by using ethylenediaminetetraacetic acid (EDTA) in the buffer solution. Samples that are outside the functional pH range of the ammonium chloride buffer may affect the results obtained from this method; adjust the pH of these samples to within a range of 5–9 using either concentrated hydrochloric acid (HCl) or ammonium hydroxide (NH_4OH). Method interferences may be caused by contaminants in the reagents, reagent water, glassware, or other factors, which may bias the results; take care to keep all such items free of contaminants. See Norwitz and Keliher for a comprehensive study of interferences in the spectrophotometric analysis of nitrite.
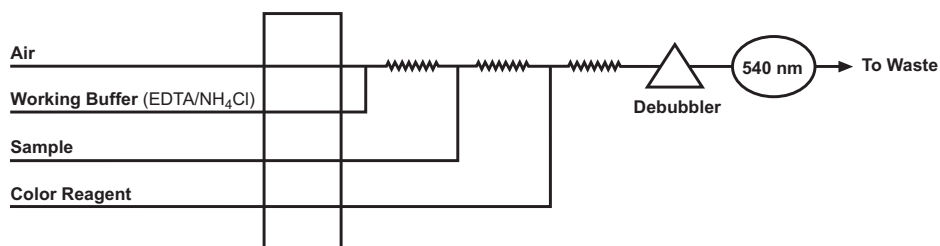
Performance Specifications

Range:	0.005–5.0 mg/L
Throughput:	60 samples/hour
Precision (at 0.01 mg/L):	<2% RSD
Precision (at 0.1 mg/L):	<1% RSD
Precision (at 1.0 mg/L):	<1% RSD
Method Detection Limit (MDL):	0.0004 mg/L
Percent Recovery:	100.99%

Chemicals

Ammonium Chloride, NH_4Cl	Hydrochloric Acid, concentrated, HCl
Ammonium Hydroxide, NH_4OH	*N*-(1-naphthyl)ethylenediamine Dihydrochloride, $C_{12}H_{14}N_2 \cdot 2HCl$
Brij®-35, 21% solution	
Chloroform, $CHCl_3$	Phosphoric Acid, concentrated, H_3PO_4
Deionized (DI) Water (ASTM Type I or II)	Potassium Nitrite, KNO_2
Ethylenediaminetetraacetic Acid, Disodium Salt Dihydrate (EDTA), $C_{10}H_{16}N_2Na_2O_8 \cdot 2H_2O$	Sulfanilamide, $C_6H_8N_2O_2S$

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Basic Flow Diagram

Air

Working Buffer (EDTA/NH$_4$Cl)

Sample

Color Reagent

Debubbler

540 nm → To Waste

Selected References

Methods for 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.

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

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

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); 1sted.: Geneva Switzerland, 1996.
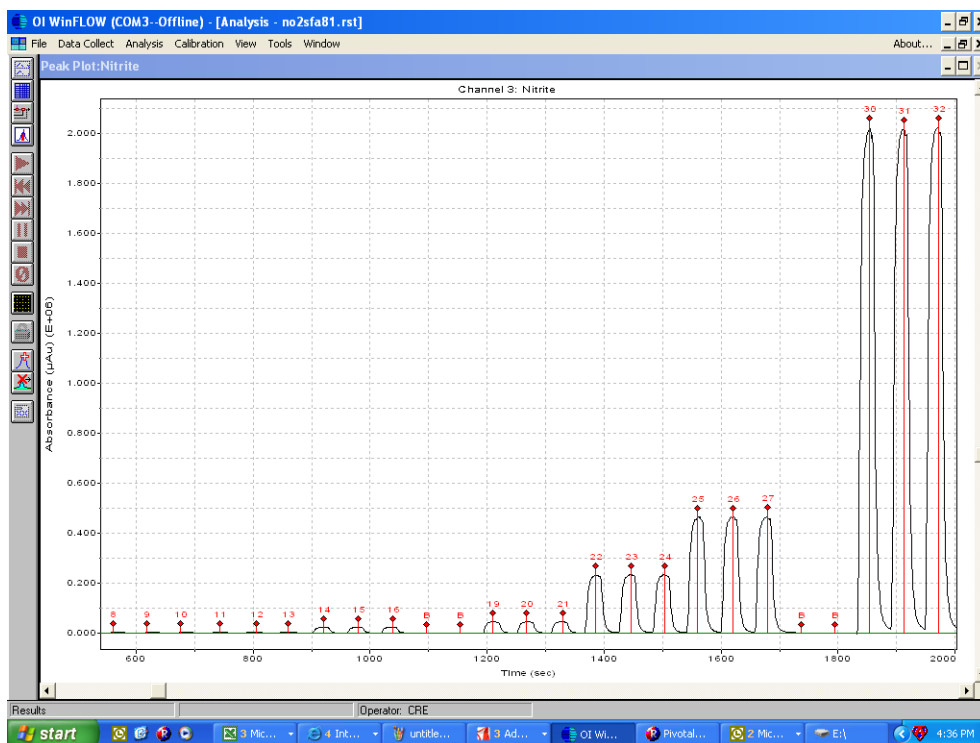
Figures

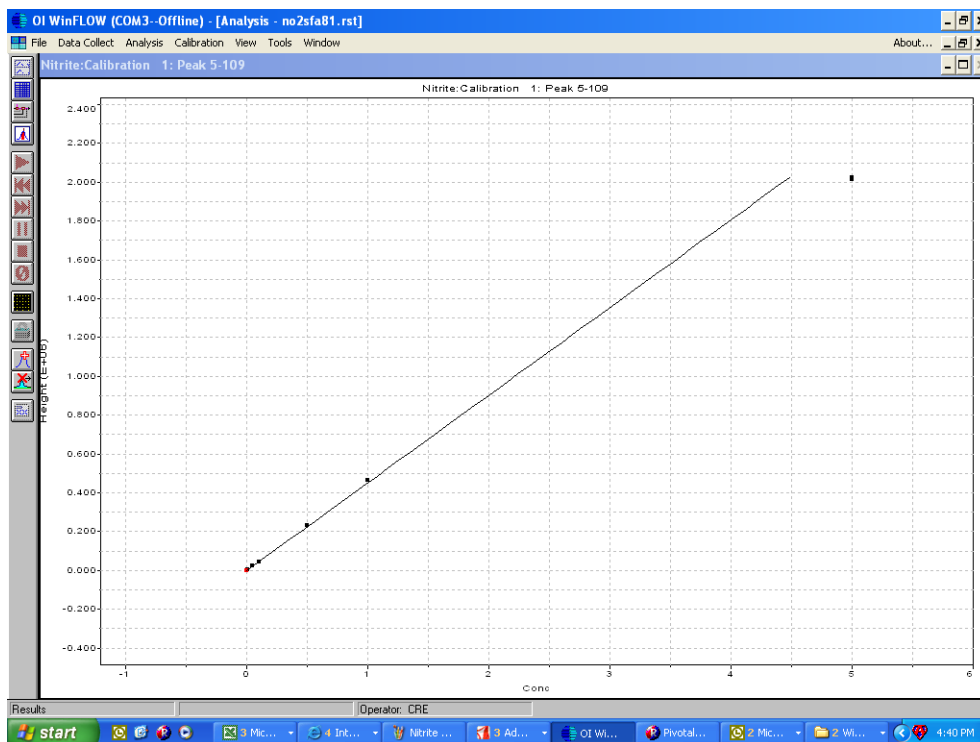Figure 1. Nitrite Nitrogen Calibration (0.005–5.00 ppm)

Cartridge PN 324501
Methodology PN 324502
Publication 27161009

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Figure 2. Nitrite Nitrogen Calibration Curve (0.005–5.00 ppm)
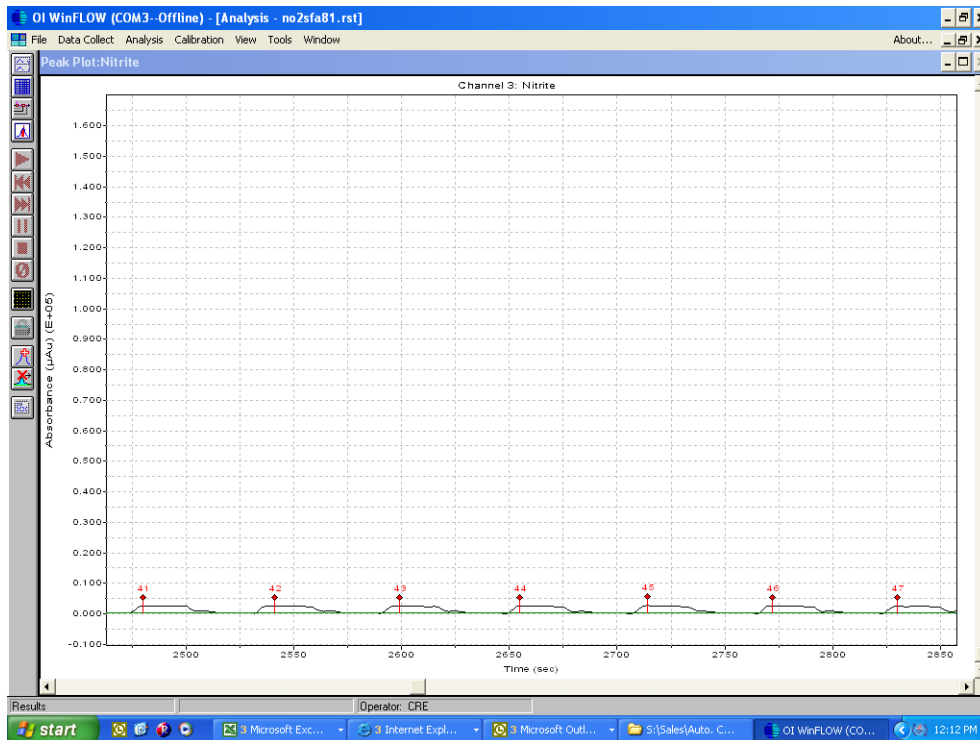
Figure 3. Nitrite Nitrogen Method Detection Limit (at 0.005 ppm)

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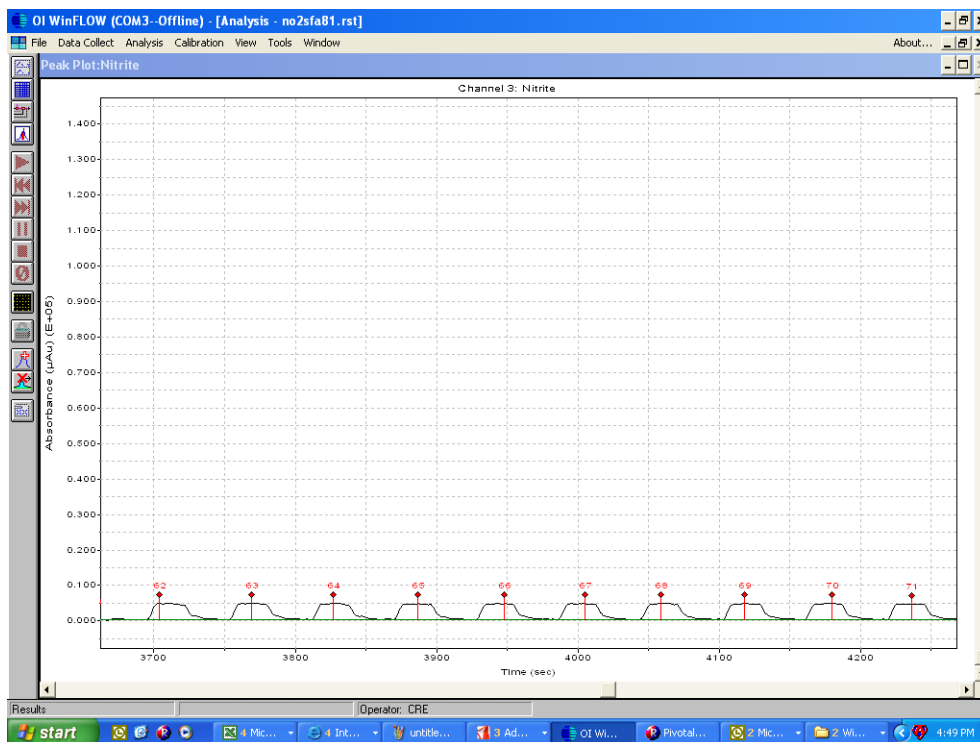**Flow Solution® 3100**

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Figure 4. Nitrite Nitrogen Precision (at 0.01 ppm)
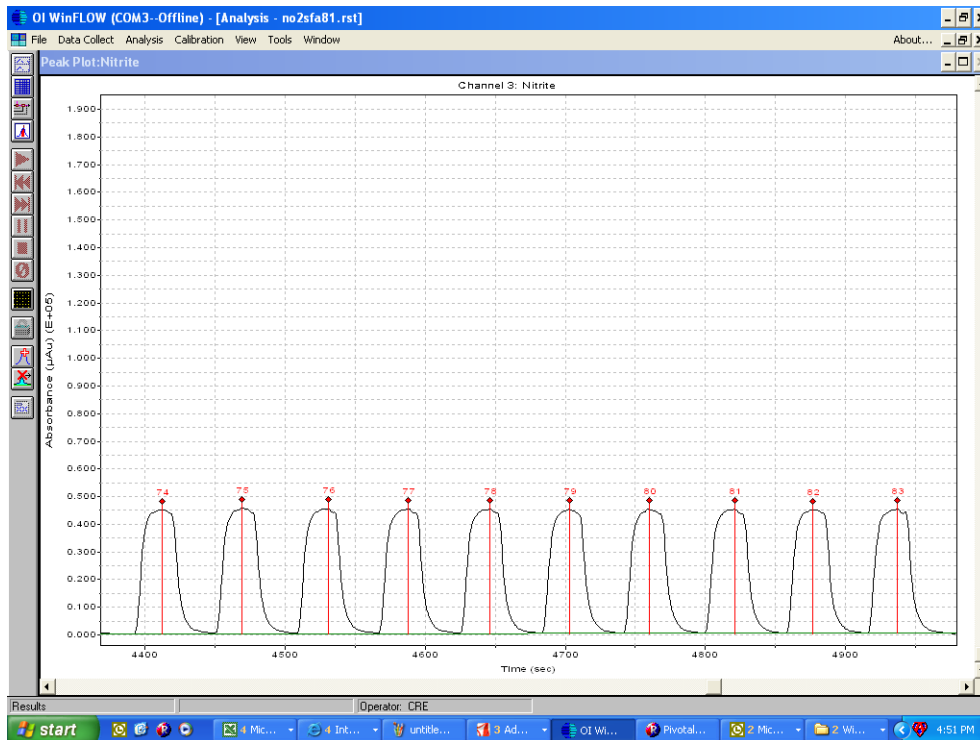
Figure 5. Nitrite Nitrogen Precision (at 0.10 ppm)

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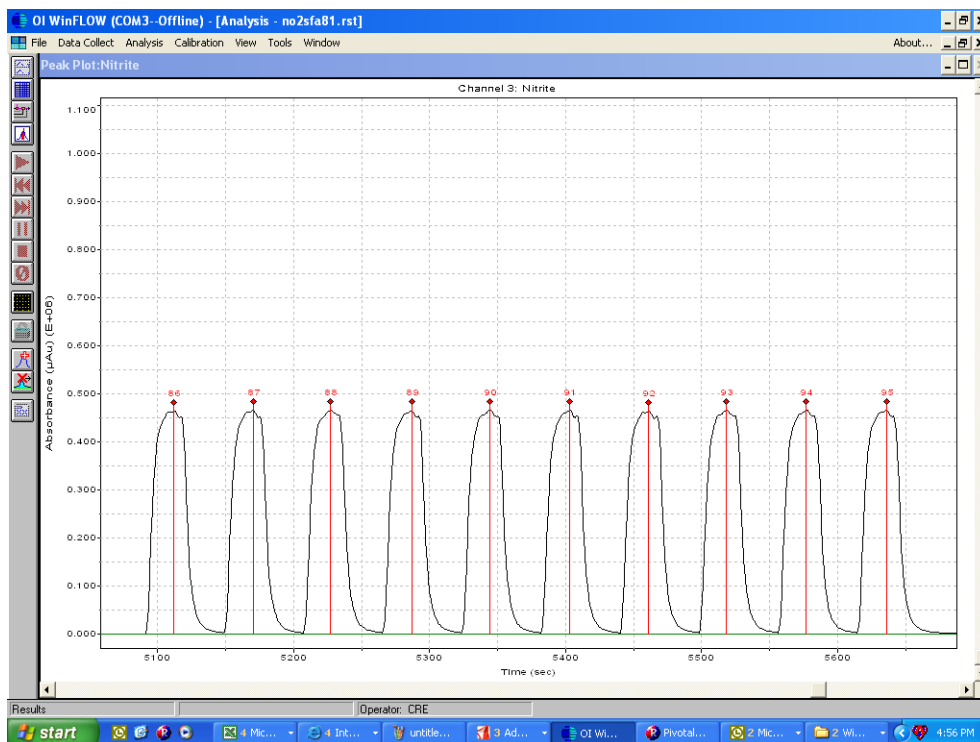*(800) 653-1711*
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Figure 6. Nitrite Nitrogen Precision (at 1.00 ppm)
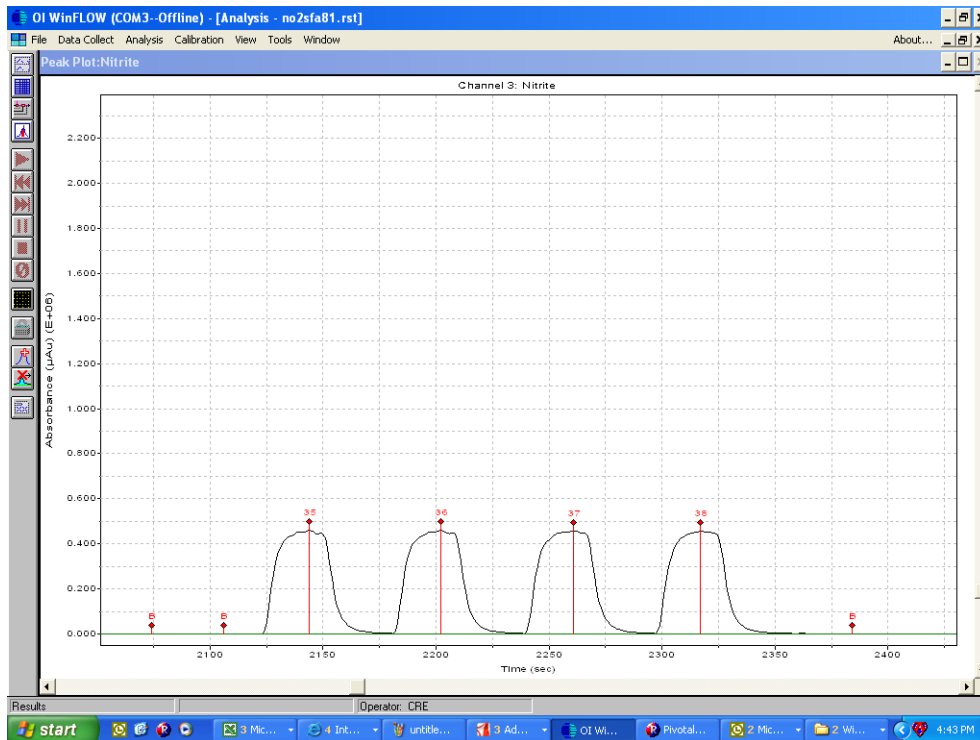
Figure 7. Nitrite Nitrogen QC Sample Precision (Artificial Seawater 1.0 ppm at 101%)

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Figure 8. Nitrite Nitrogen Calibration Results (0.005–5.00 ppm)

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Table 1. Nitrite Nitrogen Method Data

Parameter	Calibrant 0.005 mg/L	Calibrant 0.01 mg/L	Calibrant 0.1 mg/L	Calibrant 1.0 mg/L	Calibrant 5.0 mg/L	Artificial Sea Water Spike 1.0 mg/L
Rep 1	0.0050	0.0102	0.0995	1.026	4.475	1.015
Rep 2	0.0049	0.0103	0.1003	1.028	4.479	1.013
Rep 3	0.0050	0.0100	0.1002	1.028	4.476	1.010
Rep 4	0.0052	0.0099	0.1000	1.029	4.455	1.003
Rep 5	0.0049	0.0100	0.0996	1.028	4.443	—
Rep 6	0.0047	0.0099	0.0995	1.030	4.473	—
Rep 7	0.0049	0.0102	0.0994	1.023	4.475	—
Rep 8	—	0.0099	0.0995	1.031	4.469	—
Rep 9	—	0.0100	0.0992	1.031	4.474	—
Rep 10	—	0.0098	0.0997	1.028	4.475	—
Average	0.0050	0.0100	0.0997	1.028	4.469	1.010
Standard Deviation	0.0001404	0.0001510	0.0003491	0.002321	0.01146	0.005264
% RSD	2.83	1.51	0.35	0.23	0.26	0.52
MDL	0.0004	—	—	—	—	—
% Accuracy	—	—	—	—	—	100.99%

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