Scope

This method is used for determining nitrite nitrogen in drinking water, groundwater, surface water, and domestic and industrial waste 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 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 interfere with the analysis by binding with 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 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). Samples that may contain residual chlorine should be tested for reduction efficiency through the analysis of Matrix Spike/Matrix Spike Duplicate (MS/MSD) samples; when necessary, dechlorinate samples with sodium thiosulfate ($Na_2S_2O_3$). Method interferences caused by contaminants in the reagents, reagent water, glassware, etc., may bias the results; take appropriate precautions are to keep all such items free of contaminants. See Norwitz and Keliher for a comprehensive study of interferences in the spectrophotometric analysis of nitrite.
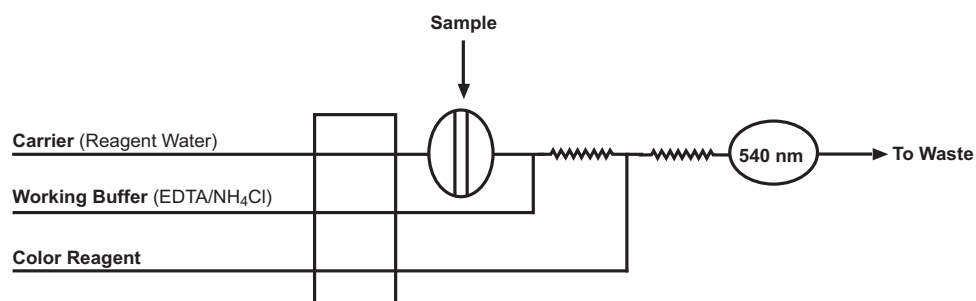
Performance Specifications

Range:	0.01–10 mg/L
Throughput:	60 samples/hour
Precision (at 0.1 mg/L):	<2% RSD
Precision (at 0.5 mg/L):	~1% RSD
Method Detection Limit (MDL):	0.002 mg/L

Chemicals

Ammonium Chloride, NH_4Cl

Ammonium Hydroxide, NH_4OH

Chloroform, $CHCl_3$

Deionized (DI) Water (ASTM Type I or II)

Ethylenediaminetetraacetic Acid, Disodium Salt Dihydrate (EDTA), $C_{10}H_{16}N_2Na_2O_8 \cdot 2H_2O$

Hydrochloric Acid, concentrated, HCl

N-(1-naphthyl)ethylenediamine Dihydrochloride, $C_{12}H_{14}N_2 \cdot 2HCl$

Phosphoric Acid, concentrated, H_3PO_4

Potassium Nitrite, KNO_2

Sulfanilamide, $C_6H_8N_2O_2S$

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Basic Flow Diagram

Sample

Carrier (Reagent Water)

Working Buffer (EDTA/NH$_4$Cl)

Color Reagent

540 nm → To Waste

Selected References

Methods for Chemical Analysis of Water and Wastewater; EPA−600/4−79−020; U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1984; 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.

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

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
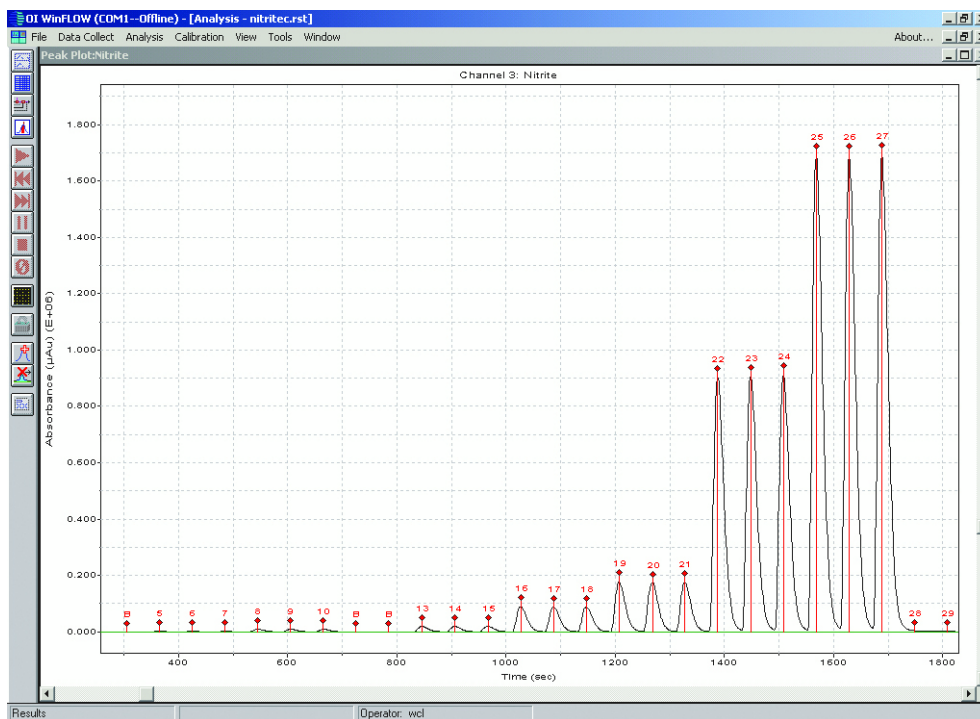
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Figures

Figure 1. Nitrite Calibration (0.01–10.0 ppm at 60 samples/hour)
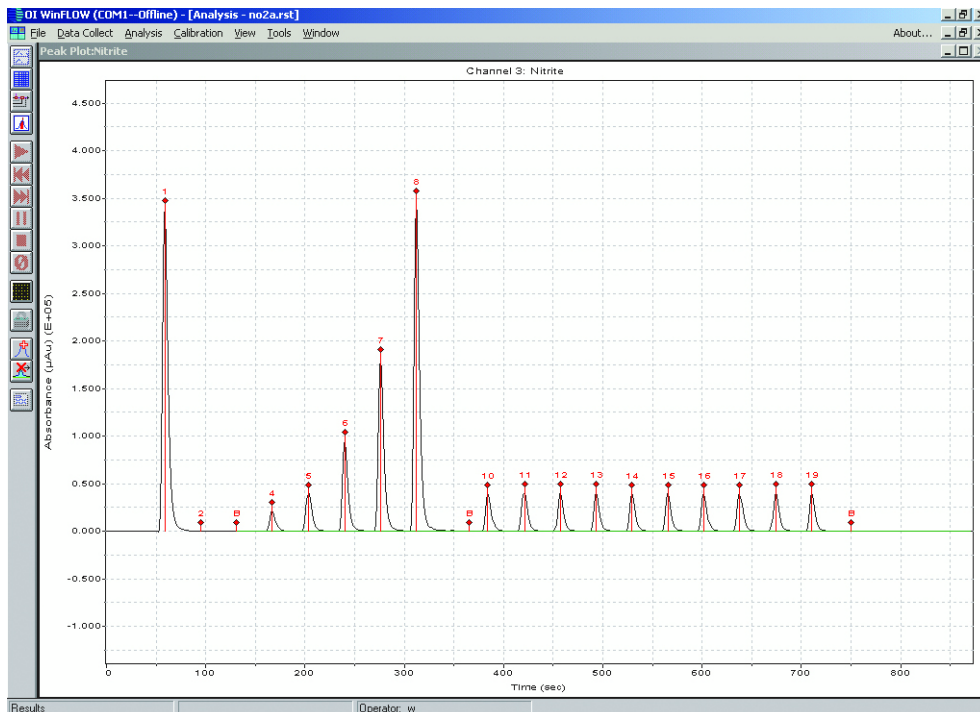
Figure 2. Nitrite Calibration (0.05–1.0 ppm at 120 samples/hour)

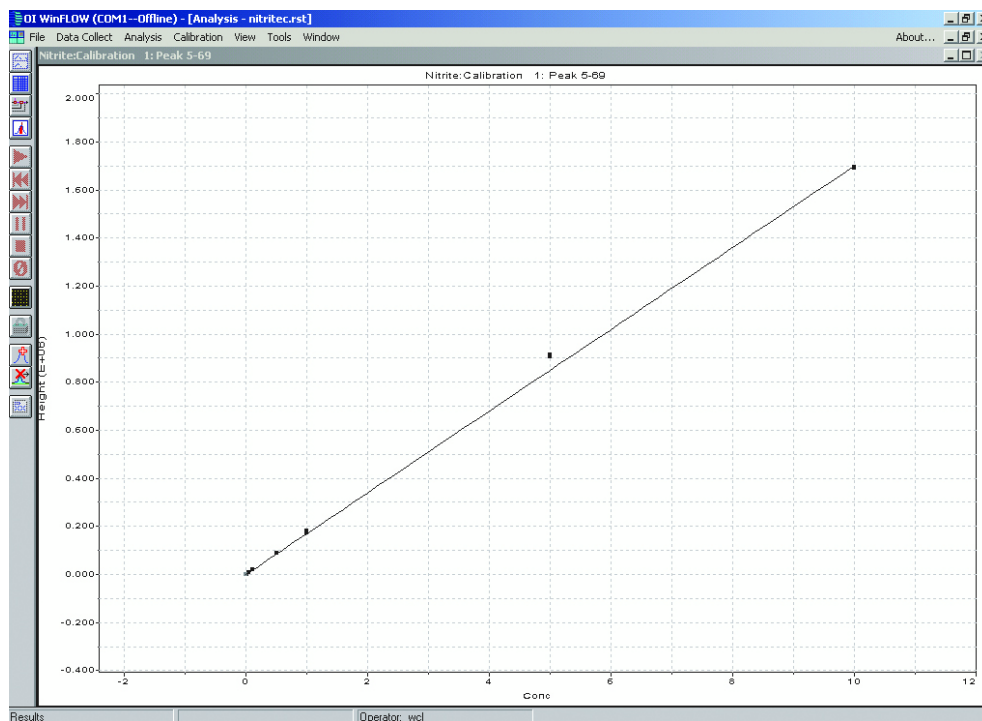Cartridge PN 324255
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Figure 3. Nitrite Calibration Curve (0.01–10.0 ppm)
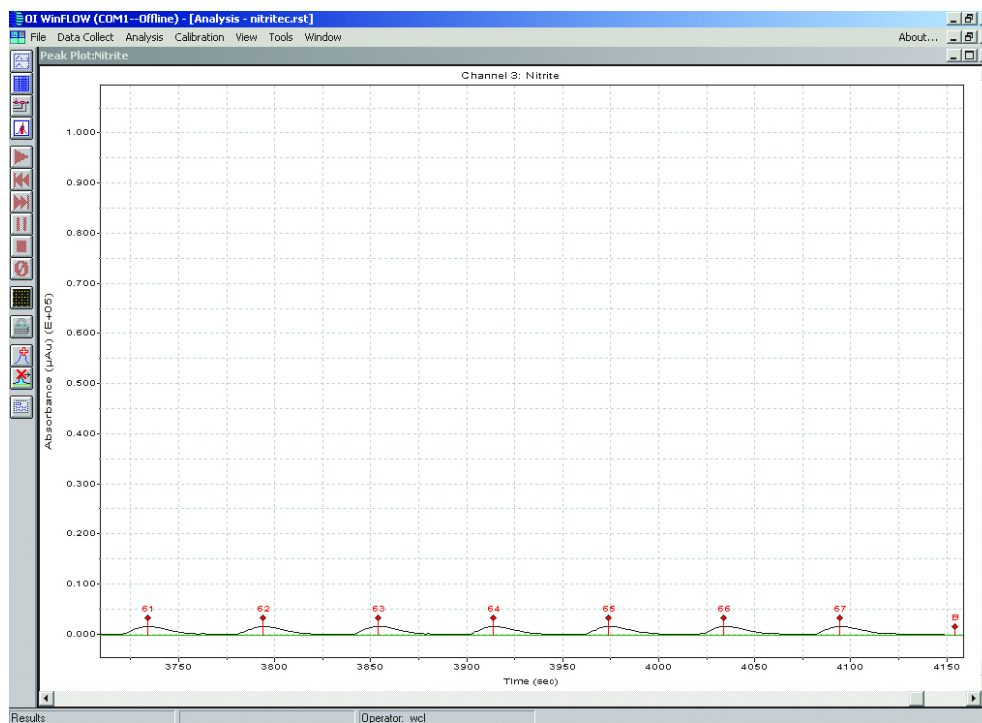
Figure 4. Nitrite Method Detection Limit (at 0.01 ppm)

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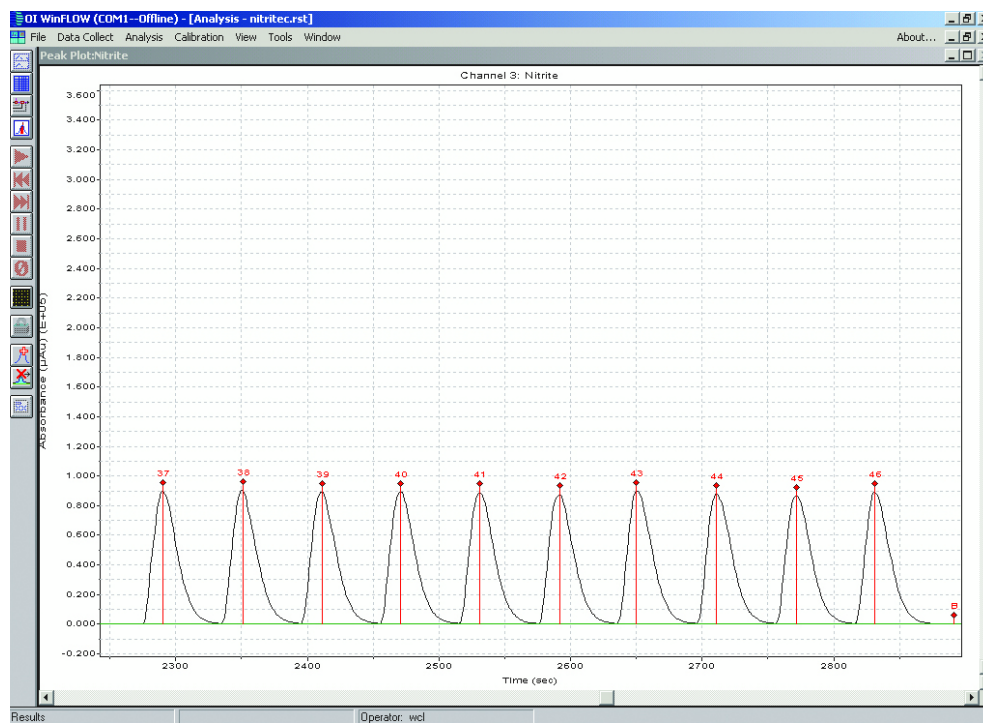Publication 26451009

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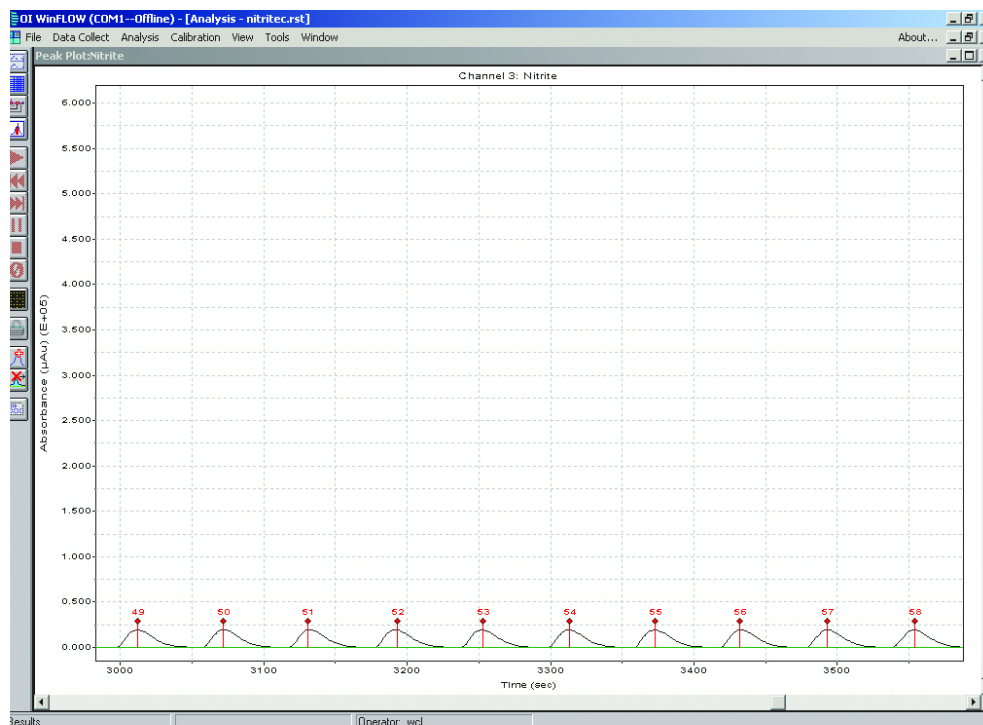
Figure 5. Nitrite Precision (at 0.1 ppm)

Figure 6. Nitrite Precision (at 0.5 ppm)

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Figure 7. Nitrite Calibration Results (0.01–10.0 ppm)

Table 1. Nitrite Nitrogen Precision Calculations

	0.500 mg N/L	0.100 mg N/L	0.010 mg N/L
Replicate 1	0.526	0.112	0.0095
Replicate 2	0.530	0.116	0.0095
Replicate 3	0.524	0.114	0.0089
Replicate 4	0.524	0.114	0.0092
Replicate 5	0.520	0.112	0.0096
Replicate 6	0.515	0.114	0.0093
Replicate 7	0.525	0.112	0.0095
Replicate 8	0.515	0.112	—
Replicate 9	0.507	0.116	—
Replicate 10	0.523	0.113	—
Mean	0.521	0.113	0.0094
Standard Deviation	0.006698	0.001685	0.000239
% RSD	1.28	1.48	2.55
% Recovery	104	113	94
MDL	—	—	**0.008**

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