Method Abstract

Summary:

Cadmium metal reduces nitrate quantitatively to nitrite. The nitrite formed, in addition to any nitrite originally present in the sample, becomes diazotized with sulfanilamide and subsequently coupled with *N*-1-naphthylethylene-diamine dihydrochloride. The resulting highly-colored azo dye is colorimetrically detected at 540 nm.

Measure nitrite singly by performing the same analysis but without the cadmium reduction. Without the cadmium, nitrate does not reduce to nitrite and is not detected since only nitrite forms the azo dye.

Interferences:

Filter turbid samples prior to analysis.

Iron, copper, and other metals may interfere with the analysis by binding with the nitrate or nitrite in the sample, thus blocking the color formation reaction. Eliminate this interference by using ethylenediaminetetraacetic acid (EDTA) in the buffer solution.

Samples outside of 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).

Oil and grease 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 forms cadmium sulfide (CdS), which precipitates from solution. Samples containing sulfide cannot be determined by this method without first removing the sulfide by precipitation with cadmium salts.

Chlorine may reduce the reduction efficiency of the cadmium reactor. When necessary, dechlorinate samples with sodium thiosulfate ($Na_2S_2O_3$).

Performance Specifications:

Range	1–100 mg/L nitrate plus nitrite nitrogen
Throughput	30 samples/hour
Precision at 1 mg/L	<1% RSD
100 mg/L	<1% RSD
Method Detection Limit (MDL)	0.05 mg/L

Chemicals:

Ammonium chloride, NH_4Cl

Ammonium hydroxide, NH_4OH

Brij®-35, 30% w/v (OI Analytical PN A21-0110-33)

Cadmium, Cd

Chloroform, $CHCl_3$

Cupric sulfate pentahydrate, $CuSO_4 \cdot 5H_2O$

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

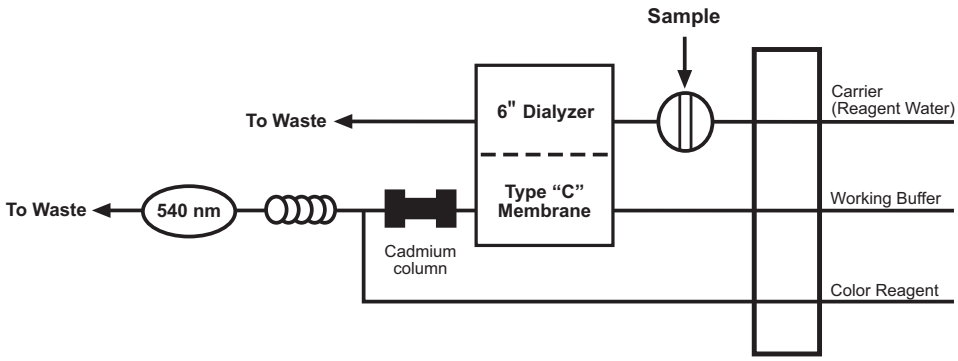
Potassium nitrate, KNO_3

Potassium nitrite, KNO_2

Sulfanilamide, $C_6H_8N_2O_2S$

Method Abstract

Basic Flow Diagram:



Selected References:

Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Cadmium Reduction). *Methods for the Chemical Analysis of Water and Wastes*; EPA/600/R-79-020; U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1984; Method 353.2.

Standard Methods for the Examination of Water and Wastewater, 20th ed.; American Public Health Association: Washington, D.C., 1998.

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