

EPA Colorimetric Testing for Nitrate + Nitrite

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Topics for Discussion

- Methods
- Application
- Reagents
- Interferences
- Hardware





Method Principle – Griess Reaction

Sulfanilamide

$$H_2NSO_3$$
 $NH_2 + N_2O_3$
 $NH_2 + N_2O$



- Color Reagent
 - Store in Refrigerator in a Dark Bottle
 - One Month Stability
 - Filter Before Use to Eliminate Color





- Ammonium Chloride Buffer
 - Buffer Specific to Method
 - Suitable for Samples Preserved to 2 mL/L Sulfuric Acid
 - Adjust pH to 8.5
 - Check pH if Low Reduction Efficiency





Cadmium Reduction



Test Procedures

- Methods
 - EPA 353.2 Rev. 2.0 (1993)
 - SM 4500-NO₃⁻ E, F-200
- Applications
 - Saline
 - Surface
 - Drinking
 - Domestic
 - Industrial





- 6 N HCl
- Copper Sulfate
 - Store at Room Temperature
- Cadmium
 - Toxicity
 - Mesh Size of Cadmium Granules
 - Coated Cadmium Coil







Interferences

- Cadmium Reduction
 - Oil and Grease
 - Suspended Matter
 - Iron, Copper, and other Metals
 - Residual Chlorine
 - Color and Turbidity





Hardware

- Cadmium Column
 - Prepare or Purchase
- Cadmium Coil
 - Purchase
 - Regenerate/Condition
- Switching Valve
 - Necessary for Inline Reduction on Discrete Analyzer
 - Additional Protection for Flow Analyzer





Hydrazine Reduction



Test Procedures

- Methods
 - SM 4500-NO₃ H-200
- Applications
 - Surface
 - Domestic
 - Industrial



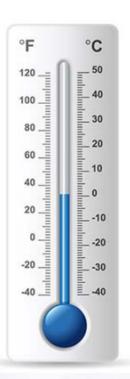


- Hydrazine Sulfate
 - Stable for 6 Months
 - Store in Amber Bottle
 - Store in the Refrigerator
 - Concentration Optimization
- Copper Sulfate
 - Store at Room Temperature



Interferences and Hardware

- Hydrazine Reduction
 - Sulfide
 - Saline Samples
 - Colored Samples
- Heating
 - Consistent Heating During Reduction Period





Vanadium(III) Chloride Reduction



Test Procedures

- Methods
 - Easy (1-Reagent) Nitrate Method, Revision November 12, 2011
- Applications
 - Surface
 - Drinking
 - Domestic
 - Industrial





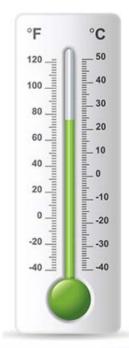
- Vanadium(III) Chloride
 - Easily Oxidized
 - Store at Room Temperature
 - Liquid Waste Product
 - Moisture and Light Sensitive
 - HCl





Interferences and Hardware

- Vanadium (III) Reduction
 - Residual Chlorine
 - Turbidity and Color
 - Sulfate, Phosphate
 - Concentrations Above 100 ppm
- Heating
 - Capable of Heating to 80° C
 - Lower Temperature Heating Lengthens Reduction Period





Enzymatic Reduction



Test Procedures

- Methods
 - USGS Techniques and Methods 5-B8, Chapter 8, Section B, Methods of the National Water Quality Laboratory Book 5, Laboratory Analysis, 2011 USGS.
 - NECi Method N07-0003, "Nitrate Reductase Nitrate-Nitrogen Analysis," Revision 9.0, March 2014, The Nitrate Elimination Co., Inc.
- Applications
 - Saline
 - Surface
 - Drinking
 - Domestic
 - Industrial



- Phosphate Buffer Solution
 - EDTA
 - Potassium Phosphate
 - Potassium Hydroxide
 - Stable 6 Months
- Nitrate Reductase and NADH
 - Non-Hazardous
 - Prepared Stable for 8 Hours
 - Prepared Reagent Volume





Interferences

- Turbidity and Color
- Metal lons
 - EDTA in Approved Method
- Sulfate, Chloride, Bromide
 - High Concentrations May Reduce Nitrate Recovery
- NADH
 - Interference in Griess Reaction





Hardware

- Heating
 - Heating at 37° C Required
- Reagent Cooling
 - Nitrate Reductase and NADH Stable at 2-6° C
- User Requirement
 - Manual Addition of Nitrate Reductase





Summary

	Cadmium	Hydrazine	Vanadium	Enzymatic
Applications	Saline, Surface, Drinking, Waste	Surface, Waste	Surface, Drinking, Waste	Saline, Surface, Drinking, Waste
Hardware	Coil/Column Switching Valves	Heater	High Temperature Heater	Heater
Reagent Price	\$	\$	\$\$	\$\$\$
Reagent Stability	Very Stable	Very Stable	Stable	Least Stable
Safety - Human	Carcinogen Mutagen Acute Toxicity	Carcinogen Irritant Acute Toxicity	Corrosive Eye Damage Acute Toxicity	Corrosive Irritant
Safety - Environmental	Toxic (Handling)	Toxic (Waste)	Non-Toxic	Safe