

# Colorimetric Determination of Anions on Discrete and Segmented Flow Analyzers

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## Methods

- Chloride
- Sulfate
- Phenol
- Ortho-Phosphate



# Topics

- Method Principle
- Reagents
- Interferences
- Hardware Requirements
- Additional Considerations



#### Method Principle

- Color Reagent
  - Contains Mercuric Thiocyanate and Ferric Nitrate
- Chloride
  - Displaces Thiocyanate in Mercuric Thiocyanate
- Free Thiocyanate Reacts with Ferric Ion Present
  - Forms Colored Ferric Thiocyanate Complex

- Combined Chloride Color Reagent
  - One Reagent Test
  - Stable Prepared Reagent
  - Store in Amber Bottle at Room Temperature
  - Prepare or Purchase



- Prepared Chloride Color Reagent
  - Requires Two Stock Solutions
  - Easy to Prepare
- Purchased Chloride Color Reagent
  - Removes Risk During Preparation
  - Filter Prior to Use



#### Interferences

- Color and Turbidity
  - Filter Prior to Analysis
  - Software Blanking Feature
- Sulfite and Thiosulfate
  - Reducing Agents
- Bromide and Iodide
  - Positive Interference



#### Hardware Requirements

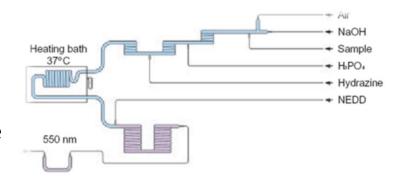
- Discrete or Segmented Flow Analyzer
  - 480 nm Filter Required
- No Heating Bath Required

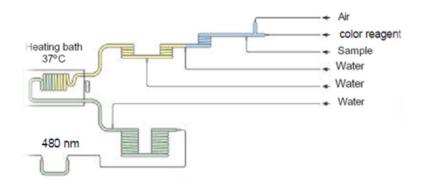


- Color Reagent Stored at Room Temperature
  - Reagent Chiller Off

### Hardware Requirements

- Discrete Analyzer
  - Method Added in Software
- Segmented Flow Analyzer
  - Multi-Test Manifold





#### Additional Considerations

- Waste Disposal
  - Hazardous Waste
    - Isolate Photometer Waste on Flow Analyz
  - Minimal on Discrete Analyzer
    - Isolate Cuvette Waste
  - Waste Treatment
    - Minimize Disposal Cost



### Method Principle

- Color Reagent
  - Barium Chloride + Methylthymol Blue → Blue Complex
- Sulfate Displaces Methylthymol Blue to Form Barium Sulfate
  - Blue Color Diminishes
- Excess Methylthymol Blue Forms Grey Color
  - Equivalent to Sulfate Concentration

- Barium Chloride Solution
  - Easy to Prepare
- Color Reagent
  - Prepare Day Prior to Use
  - Store in Amber Bottle
  - Store in Refrigerator



- Sodium Hydroxide Solution
  - Easy to Prepare
  - Stable as Prepared
- EDTA Buffer
  - Recommended System Wash Solution
  - Stable as Prepared



#### Interferences

- Cations
  - Ion-Exchange Column Included in Method
- Acidic Samples
  - Release Cations in Column
- Color and Turbidity
  - Filter Prior to Analysis



#### Hardware Requirements

- Segmented Flow Analyzer
  - 460 nm Filter
- No Heat Bath Required
- Ion Exchange Column
  - Preparation Considerations
    - Ion Exchange Resin
    - Avoid Air Entering Column

### Method Principle

- Barium Chloride
  - Sulfate + Barium Chloride → Barium Sulfate
- Barium Sulfate Suspension
  - Sodium Chloride + Stabilizer
- Turbidity Measured at 420 nm

- Barium Chloride Crystals
  - Dry Chemical
  - Prepared Reagent
- Conditioning Reagent
  - Hydrochloric Acid
  - Ethanol or Isopropanol
  - Stabilizer



#### Interferences

- Color and Turbidity
  - Filter Prior to Analysis
- Chloride
- Silica and Polyphosphates
- Aluminum



### Hardware Requirements

- Discrete Analyzer
- Hold Reagents at Room Temperature
- Software Capability



### Method Principle

- 4-Aminoantipyrine
- Potassium Ferricyanide
  - Alkaline Conditions

OH 
$$+$$
  $NH_2$   $K_3Fe(CN)_8$   $N=C$ 

- 4-Aminoantipyrine
  - Remake Daily
  - Dry Chemical White in Appearance
  - Prepared Solution is Colorless
  - Store in a Glass Container



- Potassium Ferricyanide
  - Remake Weekly
  - pH Requirements
  - Store in the Refrigerator
  - Store in a Dark Container
  - Store in a Glass Container



#### Interferences

- Sulfur Compounds
  - pH Sample < 4
  - Aerate Sample
- Oxidizing Agents
  - Negative Interference
  - Ferrous Ammonium Sulfate
- Background Contamination
  - Store Reagents and Standards in Glass

#### Hardware Requirements

- Discrete Analyzer
  - Method Added in Software
  - Distillation Required
- Segmented Flow Analyzer
  - Multi-Test Manifold
  - 505 or 520 nm Filter
  - Distillation Required

#### Method Principle

- Ammonium Molybdate
  - Orthophosphate + Molybdate → Phosphomolybdic Acid
- Antimony Potassium Tartrate
  - Catalyst for Phosphomolybdic Acid
- Ascorbic Acid
  - Phosphomolybdic Acid + Ascorbic Acid → Blue Phosphomolybdic Complex

- Ammonium Molybdate
  - Remake Monthly
  - Stored in Plastic Bottle
  - Store in the Refrigerator
  - Prone to Precipitate and Turbidity
  - Dry Chemical White in Appearance



- Sulfuric Acid
  - Stored Indefinitely at Room Temperature
  - Cool Prior to Final Dilution
- Antimony Potassium Tartrate
  - Remake Monthly for Best Results
  - Stored in Glass Container
  - Store in the Refrigerator



- Prepared Color Reagent
  - Add Reagents in Specific Order
    - Sulfuric Acid
    - Antimony Potassium Tartrate
    - Ammonium Molybdate
  - 1 − 3 Week Stability
  - Store in Refrigerator in Plastic Bottle
  - 4 Hour Stability if Ascorbic Acid Added



- Ascorbic Acid Reagent
  - Stable 1 Week
  - Prepare Daily for Best Results
  - Discard if Yellow
  - Expired Reagent Concerns



#### Interferences

- Nitrite and Hexavalent Chromium
  - Low Concentration Readings
- Arsenate
  - Positive Interference
  - Similar Colorimetric Reaction
- Salt Concentrations
  - Less Than 1% Error for Salt Concentrations up to 20%(w/v)

#### Interferences

- Iron, Copper, and Silica
  - High Silica Concentrations May Cause Positive Interference
  - 50 mg Fe<sup>3+</sup>/L, 10 mg Cu/L and 10 mg SiO<sub>2</sub>/L Tolerated
  - High Iron Concentrations May Cause Precipitation of Phosphorous
- Turbidity
  - Filtration if Applicable

#### Hardware Requirements

- Discrete Analyzer
  - Method Added in Software
- Segmented Flow Analyzer
  - Multi-Test Manifold
  - 880 or 660 nm Filter

### Additional Methods

#### **Anions**

- Bromide
- Fluoride
- Nitrite
- Sulfide

- Cyanide
- Iodide
- Nitrate + Nitrite
  - Vanadium Reduction
  - Cadmium Reduction
  - Hydrazine Reduction
  - Enzymatic Reduction

