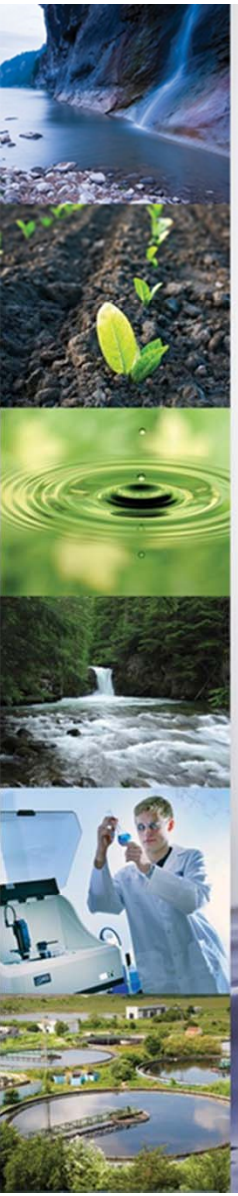

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Ammonia Colorimetric Testing by Gas Diffusion on Segmented Flow Analyzers

Sarah Leibenguth

SEAL Analytical

Topics for Discussion

- Method Approval
- Gas Diffusion
  - Principal
  - Membrane
  - pH
  - Sample Types
  - Benefits
  - Distillation Comparison

Topics for Discussion

- Colorimetric Detection
  - Method Principal
  - Reagents
  - Interferences
  - pH of Color Reaction
  - Matrix Matching

Method Approval

Method Approval

- 40 CFR Part 136.3 Table 1b

4. Ammonia (as N), mg/L	Manual distillation[6] or gas diffusion (pH >11), followed by any of the following:	350.1, Rev. 2.0 (1993)	4500-NH_3 B-1997		973.49[3].
	Nesslerization			D1426-08 (A)	973.49[3], I-3520-85.[2]
	Titration		4500-NH_3 C-1997		
	Electrode		4500-NH_3 D-1997 or E-1997	D1426-08 (B)	
	Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods		4500-NH_3 F-1997		See footnote.[60]
	Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods	350.1[30], Rev. 2.0 (1993)	4500-NH_3 G-1997 4500-NH_3 H-1997.		I-4523-85.[2]
	Automated electrode	Ion Chromatography		D6919-09	See footnote.[7]

Method Approval

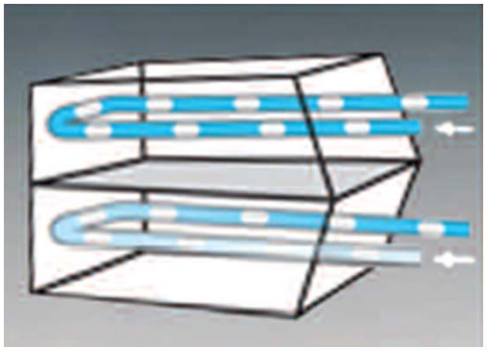
- 40 CFR Part 136.6 (xx)

The use of gas diffusion (using pH change to convert the analyte to gaseous form and/or heat to separate an analyte contained in steam from the sample matrix) across a hydrophobic semi-permeable membrane to separate the analyte of interest from the sample matrix may be used in place of manual or automated distillation in methods for analysis such as ammonia, total cyanide, total Kjeldahl nitrogen, and total phenols.

Gas Diffusion

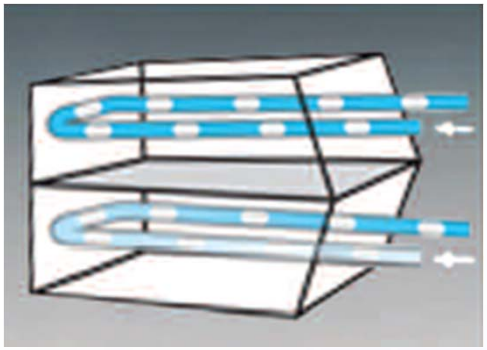
Gas Diffusion

- Principle
  - Sample Enters the System
  - Basic Donor Reagent is Added to Sample
  - Sample + Donor pH > 11
  - Ammonia Now Present as Dissolved Gas

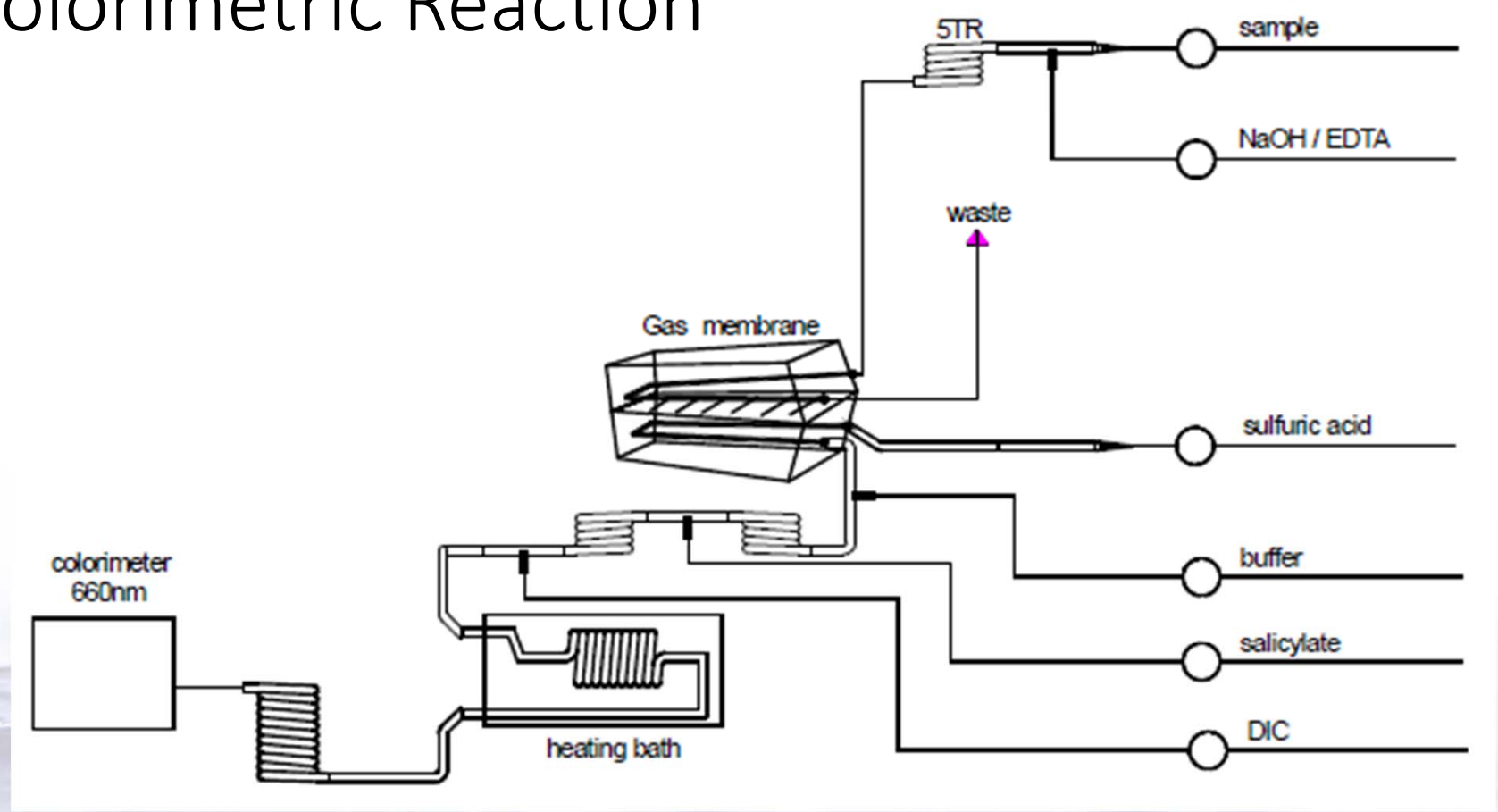


Gas Diffusion

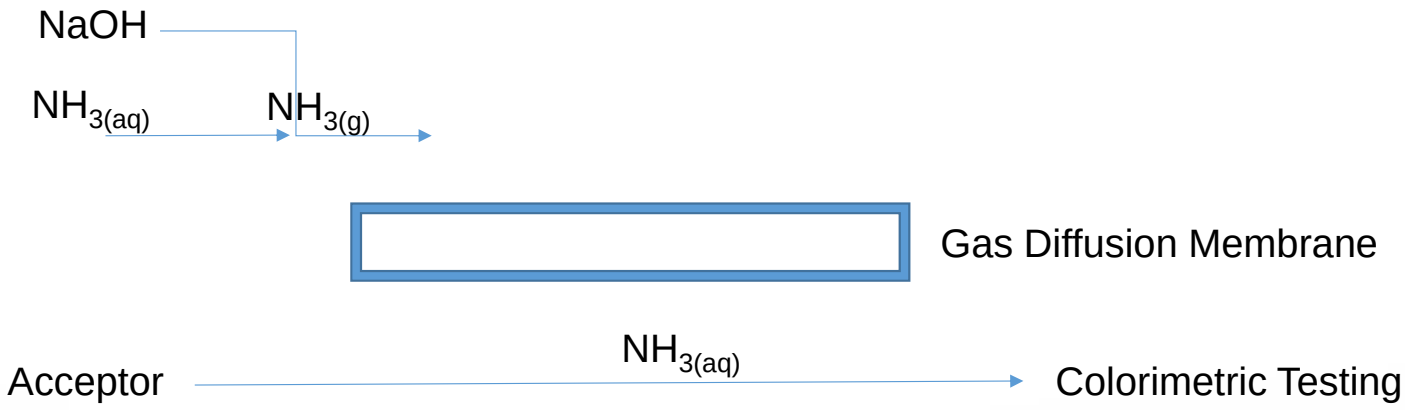
- Principle
  - Alkaline Stream
  - Hydrophobic Diffusion Membrane
  - Ammonia Gas Passes Through Membrane
  - Enters into Acidic Acceptor Stream



Colorimetric Reaction

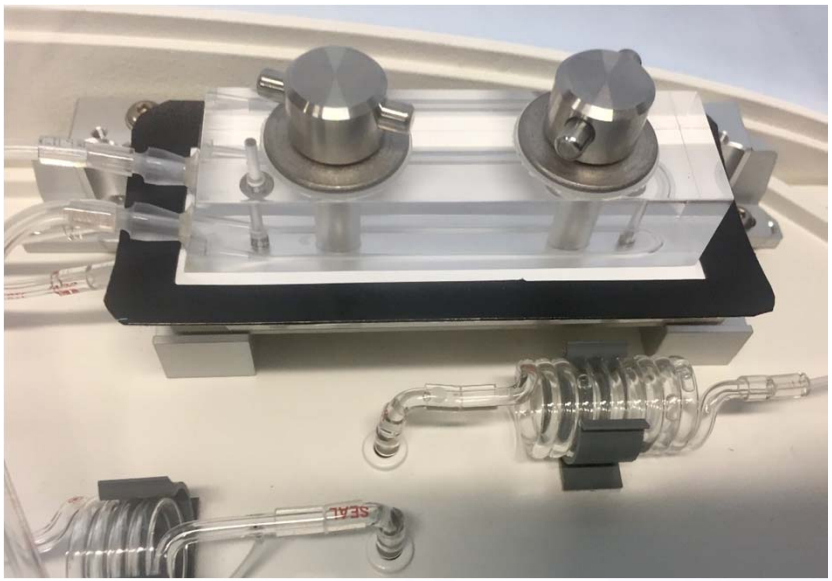


Gas Diffusion



Gas Diffusion

- Gas Diffusion Membranes
  - Membrane Material
    - PTFE
    - Polypropylene
  - Assembly
    - Shape
    - Length
  - Maintenance
    - Use of Surfactant
    - Replacement



Gas Diffusion

- Sample Type
  - Saline
  - Surface
  - Drinking
  - Domestic
  - Industrial
- Sample Pre-Treatment
  - Acidified
  - Digested

Gas Diffusion

- pH Requirements
  - Sample pH
  - Donor Stream pH > 11
  - Acceptor Stream pH < 7

Gas Diffusion

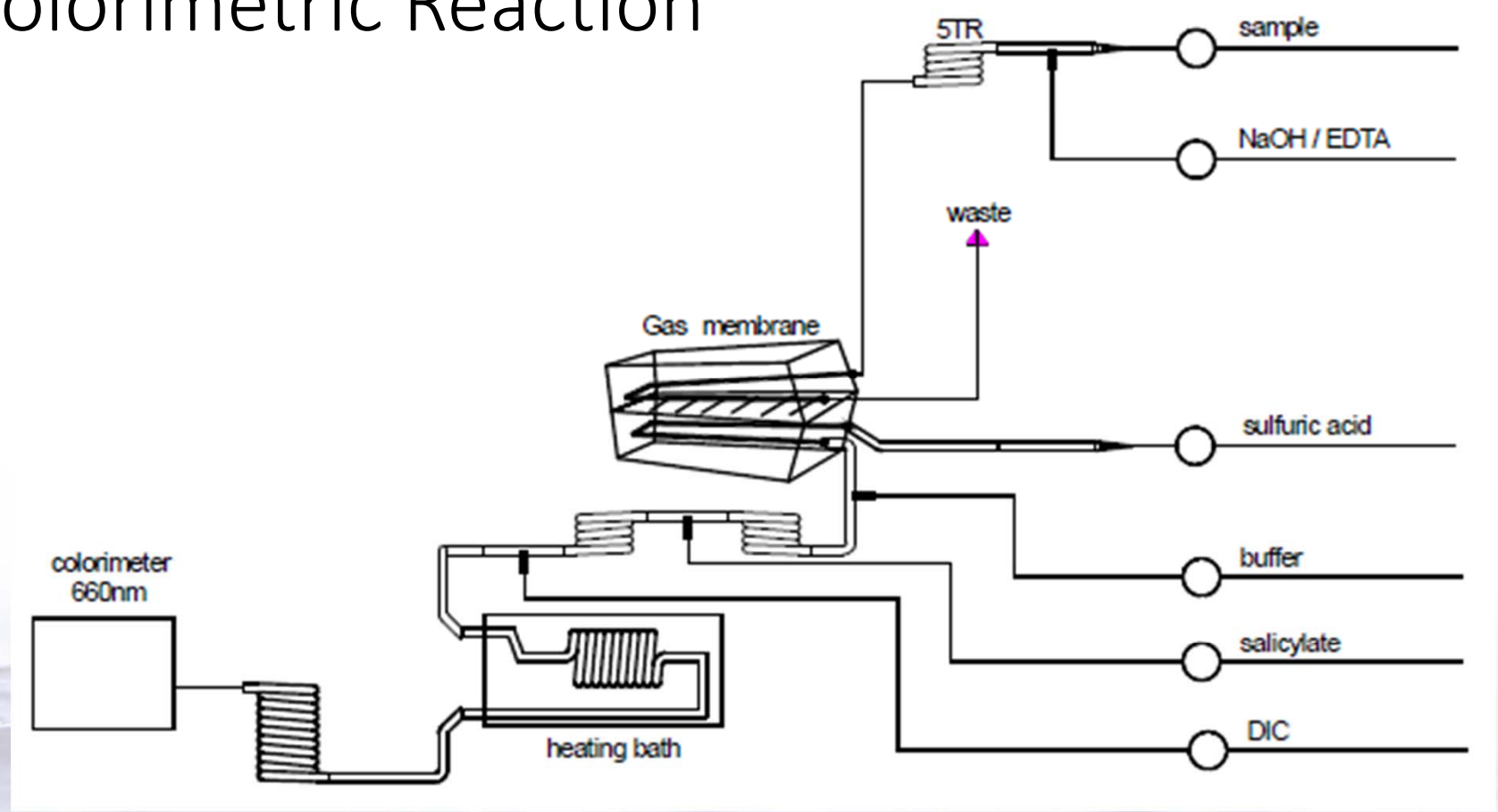
- Distillation vs Diffusion
  - Acceptor Solution
  - Matrix Separation
- Benefits
  - Time
  - Hardware

Ammonia Colorimetric Reaction

Colorimetric Reaction

- Ammonia Colorimetric Reaction
  - Salicylate or Phenate Solution
- Hypochlorite
  - Ammonia + Hypochlorite → Monochloramine
- Salicylate
  - Monochloramine + Salicylate → Indosalicylate
- Phenate
  - Monochloramine + Phenate → Indophenol
- Nitroferricyanide
  - Catalyst

Colorimetric Reaction



Colorimetric Reaction

- Reagents
  - Stability
  - Storage
  - Preparation
  - Additional Considerations

Reagents

- NaOH (Donor) Reagent
  - Store at Room Temperature
  - Remake Weekly if Surfactant Included
  - pH of Donor Reagent + Sample > 11
  - Some Recipes Include EDTA or Sodium Potassium Tartrate

Reagents

- Acidic (Acceptor) Reagent
  - Store at Room Temperature
  - Remake Weekly if Surfactant Included
  - pH of Acceptor Reagent < 7

Reagents

- Sodium Hypochlorite
  - Purchased Reagents
    - Expiration Dates
    - Bleach Stable 1 Month if Opened
  - Store in Refrigerator
    - Sodium Hypochlorite Volatizes in Heat
  - Solution is Unstable
    - Chlorine Evaporates if Exposed to Air
    - Reduced Free Chlorine Concentration
  - DCI Stable 1 Day in Solution

Reagents

- Buffer Solution
  - EDTA or Sodium Potassium Tartrate
  - Store at Room Temperature
  - Extended Shelf Life
  - pH Adjustments

Reagents

- Sodium Potassium Tartrate
  - Ammonia Contamination
  - Alkaline Boil for 1 Hour
  - Adjust pH between 7 and 8
  - Purchase from Vendor
  - Stable 6 Months in Solution

Reagents

- Salicylate
  - Refrigerate and Store in Amber Bottle
  - Stable 1 Month
  - Filtration
  - Precipitates in Acidic Conditions

Reagents

- Phenate
  - Stable 1 Month
  - Discard when Dark Brown
  - Prepare Day Prior to Analysis
- Nitroferricyanide
  - Stable for 1 Month
  - Discard if Blue in Color
  - Included in Phenate or Salicylate Reagent

Interferences

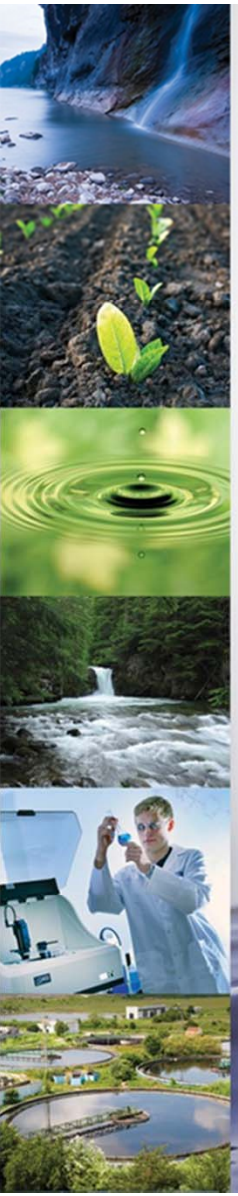
- Calcium and Magnesium
  - Precipitation
  - Addition of EDTA or Sodium Potassium Tartrate
- Turbidity or Color
  - Gas Diffusion Membrane
  - Filtration

pH of Reaction

- Testing Reaction pH
  - Phenate Reagent
    - pH 12 to 12.4
  - Salicylate Reagent
    - pH 12.6 to 13.1
- Adjusting pH
  - Hypochlorite Solution
  - Addition of NaOH

Matrix Matching

- Sample Matrix and Carrier Solution
  - Acidified Samples
  - Saline Samples
  - Digested Samples
- Method Adjustments
  - pH of Donor Reagent and Sample
  - Addition of NaOH



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