

En Chem, Inc.

Quality Assurance Document

SET No: 1

EN CHEM METHOD  
WCM-25  
REVISION NO. 6  
JANUARY 2001  
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ANALYTICAL METHOD

TITLE: Nitrogen, Ammonia, Distillation Procedure  
DEPARTMENT: Inorganic - Wet Chemistry  
APPLICATION: Drinking, surface and saline waters, domestic and industrial wastes  
REFERENCE: EPA 600 4-79-020, Revised March 1983, Method 350.1

PROCEDURE SUMMARY:

The sample is buffered at a pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds. It is then distilled into a solution of boric acid. The ammonia in the distillate can be determined colorimetrically by the LACHAT autoanalyzer or by ion specific electrode. All samples must be distilled prior to analysis.

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### SAMPLE HANDLING AND PRESERVATION:

Preserve samples with concentrated sulfuric acid, 2 mL per liter, and store at 4°C.

### SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

### APPARATUS AND MATERIALS:

Distillation apparatus: All glass, MIDI-Distillation system.

Tygon tubing: 3/8 x 1/16, 26 mm long

Collection Tubes

Graduated cylinders: 50 mL

pH meter

Beakers: 400 mL

Stir plate

Magnetic stir bar

Fixed Volume pipettor

Adjustable Volume pipettor

Boiling chips

### INTERFERENCES:

Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out. Volatile, alkaline compounds, such as certain ketones, aldehydes, and alcohols, may cause an off-color in the distillation method. Some of these, such as formaldehyde, may be eliminated by boiling off at a low pH (approximately 2 to 3) prior to distillation.

### REAGENTS:

Deionized (D.I.) water, ammonia free

Ammonium chloride:  $\text{NH}_4\text{Cl}$

Boric acid:  $\text{H}_3\text{BO}_3$

Borate buffer

Sodium hydroxide: NaOH, 10N

Ammonia Free Water (Mili-Q water)

Sodium hydroxide: NaOH, 0.1N

Sodium Phenolate

Sodium Sulfite

APG reference solution for LCS

Sodium tetraborate,  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$

NOTE: All solutions must be made with ammonia free water.

Prepare Ammonium chloride stock solution,  $\text{NH}_4\text{Cl}$ , 1.0 mL = 1.0 mg  $\text{NH}_3\text{-N}$  (1000 ppm)  
Dissolve 3.819 grams  $\text{NH}_4\text{Cl}$  in D.I. ammonia free water and bring to volume in a 1 liter volumetric flask.

Mix well. Shelf Life = 1 year.

Prepare Ammonium chloride working solution, 1.0 mL = 0.1 mg  $\text{NH}_3\text{-N}$  (100 ppm)  
Dilute 10 mL of stock solution to 100 mL in a volumetric flask using ammonia free D.I. water.

Mix well. Shelf Life = 1 week.

Prepare Boric acid,  $\text{H}_3\text{BO}_3$   
Dissolve 20 grams  $\text{H}_3\text{BO}_3$  in ammonia free D.I. water.

Dilute to 1 liter. Shelf Life = 1 year.

Prepare Borate buffer  
Add 88 mL of 0.1 N NaOH solution to 500 mL of 0.025M sodium tetraborate solution (9.5 grams anhydrous  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$  per liter)

Dilute to 1 liter with D.I. water. Shelf Life = 1 year.

Prepare sodium hydroxide, NaOH, 1N  
Dissolve 40 grams NaOH in ammonia free D.I. water.

Dilute to 1 liter. Shelf Life = 1 year.

Prepare sodium hydroxide, NaOH, 6N  
Dissolve 240 grams NaOH in ammonia free D.I. water.

Dilute to 1 liter. Shelf Life = 1 year.

PROCEDURE:

1. Use a graduated cylinder to measure 50 mL sample, or aliquot of appropriate size, into a 400 mL beaker. Adjust to  $\text{pH} = 9.5 \pm 0.1$  with 10N NaOH, a dilution of 10N NaOH, or diluted  $\text{H}_2\text{SO}_4$ . For soils, weigh out 1.0 gram of the sample into a 400 mL beaker, add 50 mL of ammonia free water and adjust the pH as described above.

NOTE: Do not adjust the pH more than 1 hour before distillation. Ammonia is very volatile.

2. Transfer the sample, pH adjusted, to the distillation tube. Add boiling chips. Add 2.5 mL borate buffer using a pipettor. Distill 40 mL into a 50 mL collection tube containing 6 mL boric acid.

NOTE: The condenser tip or an extension of the condenser tip must extend below the level of the boric acid solution. Pull out the condenser tip before the heat has been turned off.

3. Dilute distillate to 50 mL with ammonia-free water.
4. Distill a Method Blank (MB) using 50 mL of ammonia free water per batch or every 20 samples whichever is more frequent.
5. Distill a Laboratory Control Sample (LCS) using 50 mL of the APG solution per batch or every 20 samples whichever is more frequent.
6. Distill a Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) per batch or every 20 samples of a similar matrix whichever is more frequent. Use 0.1 mL of the ammonium chloride stock solution for the spiking solution. This should yield a final spiking concentration of 2.0 mg/L.

NOTE: If there is insufficient sample volume to perform an MS/MSD; perform an LCS/LCS DUP.

7. Transfer distillates to properly labeled 50 mL centrifuge tubes.
8. Store the distillates in the refrigerator until time of analysis. Analysis should be conducted within 5 days of the distillation. See EN CHEM SOP WCM-58 for the automated analysis procedure or EN CHEM SOP WCM-49 for the ion specific electrode analysis procedure.

#### POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.