

Nitrogen, ammonia, colorimetric, distillation-nesslerization

Parameters and Codes:

Nitrogen, ammonia, dissolved, I-1520-85 (mg/L as N): 00608

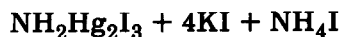
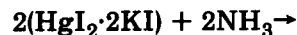
Nitrogen, ammonia, total, I-3520-85 (mg/L as N): 00610

1. Application

This method may be used to analyze water and water-suspended sediment containing from 0.01 to 2 mg/L of ammonia-nitrogen. Samples containing more than 2 mg/L need either to be diluted or to be analyzed by an alternative titration procedure.

2. Summary of method

2.1 The sample is buffered to a pH of 9.5 to minimize hydrolysis of organic nitrogen compounds. Ammonia is distilled from the buffered solution, and an aliquot of the distillate then is nesslerized. Essentially, nesslerization is the reaction between potassium mercuric iodide and ammonia to form a red-brown colloidal complex of mercuric ammonio-basic iodide:



Concentrations of ammonia are then determined by standard spectrometric measurements. Alternatively, the distillate may be titrated with standard sulfuric acid solution.

2.2 Additional information on the principle of the determination was given by Blaedel and Meloche (1963).

3. Interferences

3.1 Calcium, magnesium, iron, and sulfide interfere with the nesslerization, but the interference of the metals is eliminated by the distillation, and sulfide can be precipitated in the distillation flask by lead carbonate.

3.2 Some organic compounds may distill with the ammonia and form colors with nessler reagent, which cannot satisfactorily be read

with the spectrophotometer. Under such conditions, the sample should be titrated with standard sulfuric acid solution.

4. Apparatus

4.1 *Cylinder, graduated, with ground-glass stopper, 50-mL capacity* (Corning No. 3002 or equivalent).

4.2 *Kjeldahl distillation apparatus, 500-mL flasks.*

4.3 *Spectrophotometer, for use at 425 nm.*

4.4 Refer to the manufacturer's manual to optimize instrument.

5. Reagents

5.1 *Ammonia standard solution I, 1.00 mL = 1.00 mg NH₃-N:* Dissolve 3.819 g NH₄Cl, dried overnight over sulfuric acid, in ammonia-free water and dilute to 1,000 mL.

5.2 *Ammonia standard solution II, 1.00 mL = 0.010 mg NH₃-N:* Dilute 10.0 mL ammonia standard solution I to 1,000 mL with ammonia-free water. Prepare fresh daily.

5.3 *Borate buffer solution:* Dissolve 9.54 g Na₂B₄O₇·10H₂O in ammonia-free water. Adjust the pH to 9.5 with 1M NaOH (approx 15 mL) and dilute to 1 L with ammonia-free water.

5.4 *Boric acid solution, 20 g/L:* Dissolve 20 g H₃BO₃ in 800 mL ammonia-free water and dilute to 1 L.

5.5 *Nessler reagent—CAUTION: HgI₂ is a deadly poison, and the reagent must be so marked:* Dissolve 100 g HgI₂ and 70 g KI in a small volume of ammonia-free water. Add this mixture slowly, with stirring, to a cooled solution of 160 g NaOH in 500 mL ammonia-free water and dilute to 1 L. Allow the reagent to stand at least overnight and filter through a fritted-glass crucible.

5.6 *Sodium hydroxide solution, 1M*: Dissolve 40 g NaOH in ammonia-free water and dilute to 1 L.

6. Procedure

6.1 Rinse all glassware with ammonia-free water before beginning this determination.

6.2 Free the distillation apparatus of ammonia by boiling ammonia-free water until the distillate shows no trace using nessler reagent—**CAUTION: deadly poison.**

6.3 Pipet a volume of well-mixed sample containing less than 1.0 mg ammonia-nitrogen (250 mL max) into a 500-mL distillation flask, and adjust the volume to approx 250 mL with ammonia-free water (NOTE 1).

NOTE 1. For water-suspended sediment mixtures, rinse the pipet with ammonia-free water to remove adhering particles and combine with sample.

6.4 Add 12.5 mL borate buffer solution, and adjust the pH to 9.5 with 1M NaOH, if necessary.

6.5 Immediately distill at a rate of not more than 10 mL or less than 6 mL per min; collect the distillate in a 250-mL volumetric flask containing 25 mL boric acid solution. The tip of the delivery tube must be below the surface of the boric acid solution in the receiving flask.

6.6 Collect approx 200 mL of distillate, dilute to 250 mL with ammonia-free water, and mix.

6.7 Pipet an aliquot of distillate containing less than 0.1 mg ammonia-nitrogen (50.0 mL maximum) into a glass-stoppered, graduated mixing cylinder, and adjust the volume to 50.0 mL with ammonia-free water.

6.8 Prepare a blank of ammonia-free water and a series of standards in glass-stoppered, graduated mixing cylinders. Add 5 mL boric acid solution to each, and adjust the volume of each to 50.0 mL.

6.9 Add 1.0 mL nessler reagent—**CAUTION: deadly poison**—to each blank, standard, and sample. Stopper and invert several times to mix thoroughly.

6.10 Allow the solutions to stand at least 10 min, but not more than 30 min.

6.11 Determine the absorbance of each test sample and standard against the blank.

7. Calculations

7.1 Determine milligrams of ammonia-nitrogen in each sample from a plot of absorbances of standards.

7.2 Determine the ammonia-nitrogen concentration in milligrams per liter as follows:

Ammonia-nitrogen as N, (mg/L) =

$$\frac{1,000}{\text{mL sample}} \times \frac{250}{\text{mL aliquot}} \times \text{mg N in aliquot}$$

8. Report

Report nitrogen, ammonia, dissolved (00608), and total (00610), concentrations as follows: less than 1.0 mg/L, two decimals; 1.0 mg/L and above, two significant figures.

9. Precision

9.1 Precision for dissolved ammonia-nitrogen for nine samples within the range of 0.10 to 2.0 mg/L may be expressed as follows:

$$S_T = 0.465X + 0.0001$$

where

S_T = overall precision, milligrams per liter, and

X = concentration of ammonia-nitrogen, milligrams per liter.

The correlation coefficient is 0.8140.

9.2 Precision for dissolved ammonia-nitrogen for four of the nine samples expressed in terms of percent relative standard deviation is as follows:

Number of laboratories	Mean (mg/L)	Relative standard deviation (percent)
11	0.104	73
4	.600	33
8	1.51	44
7	2.04	63

9.3 It is estimated that the percent relative standard deviation for total ammonia-nitrogen will be greater than that reported for dissolved ammonia-nitrogen.

Reference

Blaedel, W. J., and Meloche, V. W., 1963, *Elementary quantitative analysis: theory and practice* (2d ed.): New York, Harper and Row, 826 p.