



United States
Department of
Agriculture

Forest Service

Pacific Northwest
Research Station

Research Note
PNW-449 August
1986



An Improved Method of Chemical Analysis for Low Levels of Nitrogen in Forest Streams or in Rainwater

Elly E. Holcombe, Duane G. Moore, and
Richard L Fredriksen

Abstract

A modification of the macro-Kjeldahl method that provides increased sensitivity was developed for determining very low levels of nitrogen in forest streams and in rainwater. The method is suitable as a routine laboratory procedure. Analytical range of the method is 0.02 to 1.5 mg/L with high recovery and excellent precision and accuracy. The range can be increased to include higher levels of nitrogen by dilution of samples at time of Nesslerization, or by using smaller volumes of samples at the start of the analysis.

Keywords: Chemical analysis, nitrogen, water analysis, laboratory methods.

Introduction

The Kjeldahl method (Kjeldahl 1883) for determination of nitrogen (N) in soil, in plant materials, in biological tissues, and in wastewater is well known and widely used. The standard Kjeldahl method, which uses boric acid in the final step, is satisfactory for levels of N as low as 1.0 mg/L (Crawther and Evans 1980). But the N content of streams and rainwater in uncontaminated forests of the Pacific Northwest is often as low as 0.01 mg/L (Fredriksen 1972). Successful routine analysis of a large number of water samples at this level requires improved sensitivity and elimination or control of contamination.

The method we developed is a combination of techniques from several sources (American Public Health Association 1976, Kjeldahl 1883, Morrison 1971, Rainwater and Thatcher 1960) augmented by careful control of contamination and a high degree of quality control. This paper describes this modified macro-Kjeldahl method and its accuracy, precision, and sensitivity. Procedures used to minimize contamination during the collection, subsampling, and analysis are also presented.

ELLY E. HOLCOMBE (retired) was a chemist, DUANE G. MOORE is research soil scientist, and RICHARD L. FREDRIKSEN (retired) was research soil scientist, Pacific Northwest Research Station, Forestry Sciences Laboratory, 3200 Jefferson Way, Corvallis, Oregon 97331.

The use of trade, firm, or corporation names in this publication is for the information and convenience of the reader. Such use does not constitute an official endorsement or approval by the U.S. Department of Agriculture of any product or service to the exclusion of others that may be suitable.

Our method allows determination of 0.02 to 1.5 mg/L of N in water with a high degree of accuracy and precision. This is achieved by:

1. Carefully preventing contamination of the sample during collection, storage, and analysis.
2. Concentrating the sample fivefold.
3. Using a colorimetric method with Nessler's reagent and a 25-mm light path (Morrison 1971).
4. Using the most stable range on the colorimeter for measurements.

Description of the Method

The description of the method has been divided into four parts: equipment, reagents, working solutions, and analytical procedures.

Equipment

The equipment included:

1. Commercially available macro-Kjeldahl digestion and distillation equipment with hoods.
2. Kjeldahl flasks and racks, 800 mL.
3. Distillation receivers, 32- x 200-mm Pyrex glass test tubes, calibrated to 100 mL.
4. Fisher Electrophotometer II with 25-mm light path.
5. Teflon boiling chips, 1-cm cubes washed with dilute HCl to remove all traces of $\text{NH}_4\text{-N}$.
6. Pipettes and other glassware used only for this analysis to avoid contamination.

Reagents

The reagents required for the analytical procedures include:

1. Double-distilled water (DDW) or de-ionized water (DIW), free of ammonia. Distilled water is passed through a column of acid-washed granular charcoal and a mixed bed (anionic and cationic forms) of ion exchange resins. The water is redistilled in an all-glass still and is stored in a glass carboy, protected from the atmosphere. A commercial de-ionization system (Aqua Media), which incorporates a prefiltration step to remove particulates and organics, also produces water of adequate purity. Water purity is verified daily and must meet the following criteria:
 - a. Conductivity— 6.25 to 12.5×10^{-5} Seimens/m; and
 - b. Nesslerization—100 mL DIW or DDW + 2 mL Nessler's reagent yields an optical density (O.D.) of less than 0.01 at 425 nm, usually 0.003 to 0.005 O.D.
2. NaOH, 10N. American Scientific, or dissolve 40 g Mallinckrodt reagent-grade NaOH in DIW and dilute to 1000 mL.
3. H_2SO_4 , concentrated (36N). NH_3 free, analytical grade (J.T. Baker or Mallinckrodt).

4. CuSO_4 , reagent grade (J.T. Baker or Mallinckrodt).
5. KCl , reagent grade (J.T. Baker).
6. Nessler's reagent. A.P.H.A. reagent for ammonia nitrogen (Hartman-Leddon or Fisher).
7. K_2HPO_4 , reagent grade (J.T. Baker).
8. KH_2PO_4 , reagent grade (J.T. Baker).
9. $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, reagent grade (J.T. Baker or Mallinckrodt).
10. NH_4Cl , reagent grade (J.T. Baker or Mallinckrodt).
11. Na thiosulfate, reagent grade (J.T. Baker).

Working Solutions

Working solutions for the routine analysis of samples include:

- 1.a. 10N NaOH. Either dissolve 40 g NaOH and dilute to 1000 ml_ with DIW or use commercially prepared 10N NaOH.
- b. 0.1N NaOH. Dilute 10 ml_ of 10N NaOH to 1000 ml_ with DIW.
2. Digestion mix. Dissolve 20 g CuSO_4 + 100 g KCl in DDW or DIW and dilute to 1000 ml_ (equals 2 percent CuSO_4 and 10 percent KCl).
3. Borate buffer. Add 88 ml_ of 0.1 N NaOH solution to 500 mL of a 0.025N sodium tetraborate solution (5.0 g anhydrous $\text{Na}_2\text{B}_4\text{O}_7$ or 9.5 g $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ per 1000 mL) and dilute to 1000 mL with DIW or DDW.
4. Phosphate buffer solution, pH 7.4. Add 68.76 g K_2HPO_4 and 14.30 g KH_2PO_4 to a 1000-mL volumetric flask and add 800 mL DIW. Heat to just under boiling for 40 minutes. Cool and bring to volume with DDW or DIW. Adjust to pH 7.4 with appropriate acidic or basic phosphate salt. Store in refrigerator.
5. NH_4Cl . Dissolve 3.819 g oven-dried NH_4Cl in DIW and dilute to 1000 mL (equivalent to 1000 mg NH_4^+ -N/L).
6. NH_4Cl standard, 10 mg/L NH_4^+ -N. Dilute 10 mL of 1000 mg/L NH_4Cl stock solution with DIW to make 1000 mL (10 mg NH_4^+ -N/L).
7. Trap solution. Dilute 1500 mL 10N NaOH and 75 g Na thiosulfate to 15 L with DIW.
8. Comparison digestion mix.^ 133 g K_2SO_4 + 100 g NaCl + 20 g CuSO_4 made to 1000 mL DDW. Use 5 mL for each sample or blank.

¹The comparison digestion mix is used by EPA and can be substituted for our mixture if desired.

Analytical Procedure

The analytical procedure permits analysis of free ammonia by distillation (item I, below) plus Nesslerization (III), by autoanalyzer (IV) (Crawther and Evans 1980), or by I plus titration (V) with 0.02 N H_2SO_4 plus indicator. Analysis of organic nitrogen is by digestion and distillation (II) plus III, or by II and V if concentration is greater than 1.5 mg/L (American Public Health Association 1976, Crawther and Evans 1980).

I. Distillation of free NH_3 .—In duplicate, add 500 mL aliquots of a filtered or unfiltered water sample plus two Teflon boiling chips to 800-mL Kjeldahl flasks. Include duplicate 500 mL DIW reagent blanks in each set of 12 samples. Add 5 mL DIW to distillation receivers and set in place on distillation unit with tips of condenser delivery tubes extending into the water. Add 25 mL of phosphate buffer or borate solution^{2/} to each sample and blank and immediately set each flask on the distillation unit. Stopper tightly and swirl each flask to mix the contents thoroughly. Heat to distill the free NH_3 into the distillation receivers. When distillation is well advanced, lower the receiver tubes to prevent aspirating the distillate into the flasks. Collect about 95 mL of distillate, rinse the tips of the delivery tubes with DIW into the receivers, and bring the volume of distillate to 100 mL with DIW. Cover the tubes with plastic wrap until ready to determine the N content by Nesslerization (III).

II. Digestion and distillation of organic nitrogen.—When the Kjeldahl flasks from the above distillation (I) have cooled sufficiently, add 5 mL of the digestion mixture and 10 mL H_2SO_4 to each flask.

Digestion: Mix the contents of each flask thoroughly by swirling, then heat on the digestion unit to remove water. For filtered samples, reflux for exactly 30 minutes beginning when the acid mixture starts to fume. For unfiltered samples, digest for 45 minutes after acid fumes (longer digestion periods result in loss of nitrogen). Remove the flasks, cool, and add 300 mL DIW. The samples are now ready for distillation.

Distillation: Add 10 mL DIW to Kimax receiver tubes and set in place on the distillation unit; be certain the receiver tips are submerged. Slowly add 50 mL 10N NaOH to each Kjeldahl flask; allow the solution to run down the side of the flask to the bottom. Then place the flask on the distillation unit, stopper tightly, and swirl to mix. Conduct the distillation as in (I) and determine N content by Nesslerization (III), or I and IV, or I and V, depending on concentration.

^{2/}We compared results using both borate and phosphate buffers and found no difference.

III. Determination of N in standards and samples by Nesslerization.—

1. Prepare standard concentrations (100 ml_) in duplicate using DIW. Prepare at least three concentrations that range from 0.2 to 1.50 mg/L $\text{NH}_4^+ \text{-N}$ and include 100 mL of DIW in duplicate as a blank.
2. Add 2 mL of Nessler's reagent to each sample distillate, standard, and blank. Mix well, and allow color to develop in the dark for 30 minutes (cover tubes with foil or place in dark to protect from fluorescent light, which changes the color of Nessler's reagent). The color development process is influenced by N concentration, temperature, time, and pH. Because of these variables, samples, standards, and blanks must all be handled in the same manner. At concentrations of N greater than 0.5 mg/L, it is important that the length of time between addition of the Nessler's reagent and reading the spectrophotometer must be the same ± 1 minute for each sample, standard, or blank in the set.
3. Measure color intensity with a spectrophotometer at 425 nm. Use DIW as a blank to zero the instrument. Record all readings in percentage of transmittance (%T) or O.D., depending on the most sensitive scale on the instrument. If %T is used, convert to O.D. by using conversion tables. Obtain corrected optical density (C.O.D.)^{3/} readings for standards and samples by subtracting the average of the O.D. readings of the appropriate blanks. The mean O.D. of the digest blanks should be subtracted from the measured O.D. of the samples.
4. Include 500 mL DIW fortified with NH_4Cl and 500 mL DIW fortified with cysteine (fortified to various levels with standard materials) each time a set of samples is analyzed to test distillation and digestion procedures for percentage of recovery. Recoveries are commonly 94 to 100 percent for NH_4Cl by distillation and 94 to 98 percent for cysteine by digestion and distillation. If recovery is less than 89 percent, the entire distillation apparatus is checked for proper operation. Once each month a complete standard curve (0 to 2.5 mg/L NH_4Cl , and 0 to 1.0 mg/L cysteine) is run to monitor the accuracy of digestion and distillation steps.

After analysis of a set of samples, the Kjeldahl flasks must be thoroughly cleaned to ensure complete removal of any NaOH residue. Flasks are emptied as soon as possible and rinsed three times with hot tap water. (They may sit overnight filled with DIW at this step.) Next, the flasks are rinsed with dilute (0.5 N) HCl, followed by three rinses with hot tap water, and finally rinsed three times with DIW. The clean flasks are stored upside down on drying racks until used.

We originally used mercuric chloride (HgCl_2) as a preservative but discontinued this because it interfered with the determination of phosphate and reduced the recovery of N. Adding HgCl_2 to samples spiked with $\text{NH}_4^+ \text{-N}$ reduced recovery 12 to 15 percent during the first distillation. Part of the N complexed by the Hg is recovered in the second distillation, but recovery is still incomplete. We also elected not to use HgCl_2 to prevent possible mercury contamination of the laboratory. If HgCl_2 is used as a preservative, it must be added to blanks and standards at the same concentration.

^{3/} C.O.D. is corrected optical density; it equals the optical density of the standard or sample minus the optical density of the blank.

IV. Autoanalyzer.—Analysis of free $\text{NH}_3\text{-N}$ may also be accomplished by an automated method (Crawther and Evans 1980, United States Environmental Protection Agency 1979). Start with II and omit the distillation step in I. Add the digestion reagents, concentrated H_2SO_4 , and boiling chips to each sample and begin the digestion. The analysis is for total dissolved N or, in the case of an unfiltered sample, total N. The concentration of free $\text{NH}_3\text{-N}$ determined on the autoanalyzer must be subtracted from the results for total dissolved N to give the amount of organic N present in the sample. We use 500 mL of an unfiltered sample to analyze for total organic N. In analysis for the total N, the digestion time is increased to 45 minutes.

V. Calculations.—The concentration of N in a sample is calculated as follows:

1. Standard factor: Calculate a mean standard factor over the range of the standard concentrations:

$$\text{mg/L N per optical density unit} = \frac{\text{concentration of standard (mg/L)}}{\text{C.O.D. of standard}}.$$

3. Concentration factor: $\frac{\text{sample volume}}{\text{distillate volume}}.$

3. Calculation factor: $\frac{\text{mean standard factor}}{\text{concentration factor}}.$

4. Milligrams per liter of N sample: C.O.D. of sample multiplied by the calculation factor.

Example: C.O.D. of 0.5 mg/L $\text{NH}_3\text{-N}$ standard = 0.082;

C.O.D. of sample = 0.072;

$$\text{Standard factor} = \frac{0.5}{0.082} = 6.09^{4/}$$

$$\text{Concentration factor} = \frac{500 \text{ mL}}{100 \text{ mL}} = 5;$$

$$\text{Calculation factor} = \frac{6.09}{5} = 1.22; \text{ and}$$

$$\text{mg/L N in sample} = 0.072 \times 1.22 = 0.088 \text{ mg N/L}.$$

^{4/} Normally the average of all the curve factors on the standard curve is used if the values are within the acceptable range of occurrence as defined by the United States Environmental Protection Agency (1979);

Evaluation of the Method

Sensitivity

The quantitative range of detection of this method for nitrogen in streamwater and precipitation is 0.02 to 1.50 mg/L. The sensitivity of this method is increased over the standard Kjeldahl method (1) by including a fivefold concentration step during digestion and distillation, (2) by Nesslerization of the distillate and colorimetric determination of $\text{NH}_3\text{-N}$ (Fredriksen 1972), and (3) by employing a spectrophotometer with a 25-mm light path. The sensitivity can be further increased to approximately 0.002 mg/L N by using a spectrophotometer with a 100-mm light path.

The sensitivity of this method is illustrated by the results of analysis of 12 replicate aliquots of a single, filtered streamwater sample for free $\text{NH}_3\text{-N}$ and soluble organic N (table 1). The sample contained 0.021 ± 0.003 mg/L free $\text{NH}_3\text{-N}$ and 0.116 ± 0.006 mg/L soluble organic N. It is apparent from the optical density values presented that the readings are outside the instrument response range normally considered optimum (O.D. from 0.071 to 0.699, or between 85 and 20 %T). We have found, however, that adequate precision can also be obtained in that portion of the standard curve representing O.D. readings between 0.011 and 0.071 by (1) employing all the precautions noted to avoid contamination and (2) reading all reagent blanks, standards, and samples against a DIW blank. Reading against a DIW blank shifts the working range as far as possible toward the most stable region of instrument response. Similar data have been obtained with samples from other streams. These results demonstrate that our method adequately quantifies levels of N at one-fiftieth the level characteristic of the standard Kjeldahl method.

Table 1—Sensitivity and precision of the modified Kjeldahl method for determination of free $\text{NH}_3\text{-N}$ and soluble organic N in a streamwater sample

Sample	Free $\text{NH}_3\text{-N}$		Soluble organic N	
	O.D. ^{1/}	mg/L	O.D.	mg/L
1	0.014	0.0178	0.088	0.1118
2	.015	.0191	.092	.1168
3	.015	.0191	.089	.1130
4	.018	.0228	.093	.1181
5	.019	.0241	.098	.1245
6	.015	.0191	.095	.1206
7	.016	.0203	.096	.1219
8	.018	.0228	.096	.1219
9	.021	.0267	.087	.1105
10	.014	.0178	.088	.1118
11	.014	.0178	.083	.1054
12	.019	.0241	.089	.1130
Mean	0.021		0.116	
Range	0.0178 to 0.0267		0.1054 to 0.1245	
SD ^{2/}	± 0.003		± 0.006	
CV ^{3/} (%)	14.29		5.17	

^{1/} O.D. = optical density.

^{2/} SD = standard deviation.

^{3/} CV = coefficient of variation.

Precision

Precision can be greatly influenced by the care taken in laboratory operations. This is particularly important in achieving the full sensitivity and precision of this method. We have found the following to be important:

1. Include duplicate reagent blanks with each set of samples to detect contamination (possible sources are the Kjeldahl equipment, reagents, glassware, de-ionized water, and the air).
2. Prepare all reagents with DDW or DIW.
3. Chemically weather all new glassware in 2 N HCl for 3 days and then thoroughly wash and rinse prior to initial use.
4. Check each batch of Nessler's reagent for uniformity of color development by running a complete standard curve. The color should range from a light yellow to a red-brown. If it has a greenish tinge, or if a precipitate forms when the reagent is added to DDW, DIW, or standards, check all equipment and reagents for contaminants. Verify the purity of the water first; then check all possible sources of heavy metal or other contaminants. If all possible sources of interference are ruled out, the shipment of Nessler's reagent should not be used.

The precision we obtained with the modified Kjeldahl procedure had a coefficient of variation of about 5 percent for soluble organic N and 14 percent for $\text{NH}_3\text{-N}$. This was acceptable, especially considering the low levels of nitrogen being determined. Reproducibility of spectrophotometer readings for reagent blanks and standards is illustrated in table 2.

Free $\text{NH}_3\text{-N}$ can be determined accurately at levels below 1.00 mg/L N (± 0.003 mg/L); however, the coefficient of variation increases sharply as the concentration decreases below 0.1 mg/L N (fig. 1). Based on analysis of 11 replicate samples over a range in concentrations from 0.007 to 1.182 mg/L, the coefficient of variation was 16 percent or less for concentrations at 0.02 mg/L or less, 5 percent at 0.10 mg/L, and 0.4 percent at 1.18 mg/L. Because the soluble organic N content of stream samples being analyzed usually ranges from 0.10 to 1.00 mg/L, the coefficient of variation associated with such analyses will seldom exceed 5 percent.

Table 2—Precision of modified Kjeldahl method for 10 replicate reagent blanks and NH_4Cl standards

	Reagent blanks	Standards ^{1/}
	----- O.D. ^{2/} -----	
Mean (n = 10)	0.007	0.1234
Standard deviation	$\pm .0013$	$\pm .0023$
Coefficient of variation (%)	18.57	1.86

^{1/}0.8 mg/L N as NH_4Cl .

^{2/}O.D. = optical density.

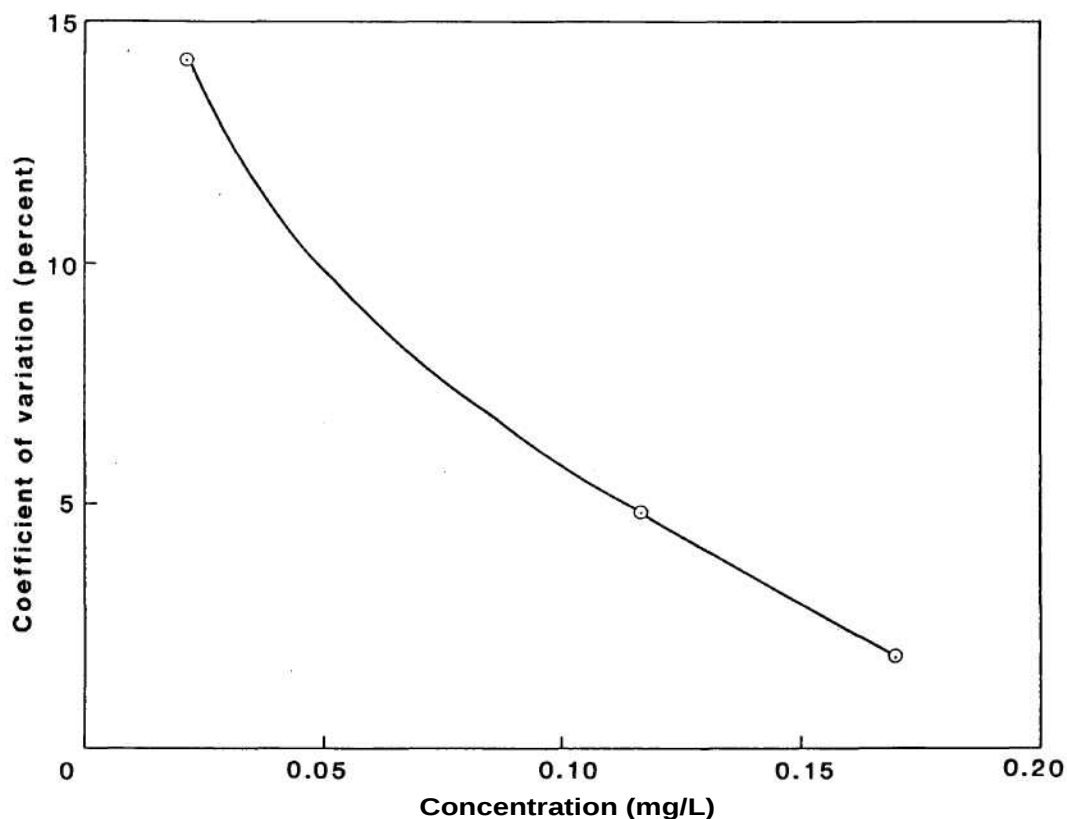


Figure 1.—The coefficient of variation of measuring various concentrations of organic nitrogen compounds in streamwater.

Accuracy

The accuracy of our procedure was evaluated by conducting recovery studies using bulk stream samples fortified with known amounts of inorganic and organic N compounds. NH_4Cl was used to measure the recovery of free $\text{NH}_3\text{-N}$, and L-glutamic acid was used to measure recovery of easily digested organic N compounds. More complex nitrogenous materials were represented by the ring compound, L-histidine. In 1982 we checked recovery with cysteine and NH_4Cl (table 3).

Table 3—Recovery of inorganic and organic forms of nitrogen-added to samples of streamwater

Form of N added	Number of determinations	Range	Mean	SD ^{2/}	CV ^{3/}
- - - Percent recovery - - -					
NH_4Cl	18	97.1 - 105.0	102.09	± 2.14	2.10
L-Glutamic acid-HCl	12	95.6 - 103.4	99.5	± 3.60	3.62
L-Histidine-HCl-H ₂ O	12	92.5 - 100.5	95.5	± 3.50	3.66
Cysteine	14	83.2 - 109.9	97.08	± 6.55	6.7

^{1/}Data include recovery of N added at low (0.02 mg/L), intermediate (0.2 mg/L), and high (1.00 mg/L) concentrations of NH_4Cl , L-glutamic acid, and L-histidine, and 0.233 mg/L cysteine.

^{2/}SD = standard deviation.

^{3/}CV = coefficient of variation.

Recovery of inorganic N appears to have exceeded the amount of $\text{NH}_3\text{-N}$ added to the stream samples. Analysis of the samples for free $\text{NH}_3\text{-N}$ prior to fortification indicated no detectable (less than 0.02 mg/L) inorganic nitrogen present. Adding a known amount of N to the sample to achieve a concentration of more than 0.02 mg/L made it possible to quantify the "total" $\text{NH}_3\text{-N}$ present; that is, the added N plus any background N that was present at levels less than 0.02 mg/L. Our analysis of the fortified sample indicated that it contained approximately 0.003 mg/L $\text{NH}_3\text{-N}$ as background before the NH_4Cl was added. These data show that if it is necessary to quantify background levels of less than 0.02 mg/L N, this can be done by the method of "known additions" using the modified Kjeldahl procedure. Essentially the same results were obtained using the EPA method (United States Environmental Protection Agency 1979).

Absolute accuracy determinations were lower for organic N than for inorganic N (table 3)—probably because of the additional steps in the procedure. Recovery of low levels of N in the straight-chain compound, L-glutamic acid, was slightly lower (97 percent) than recovery of intermediate and high concentrations. Recovery of N from the ring structure of L-histidine was reproducible but consistently lower (95.5 percent) than recovery from L-glutamic acid. Perhaps more complete recovery could be obtained by digesting for more than 30 minutes; however, our studies indicated that losses begin to occur after 30 minutes. This means that net recovery would probably not be increased. Using mercury as a catalyst would increase recovery, but we have avoided using mercury in the laboratory because of problems in the recovery of phosphorus and disposal of wastes.

Quality Control in Field and Laboratory

Quality control in both the field and laboratory is essential for accurate and precise measurements of N at the low levels made possible by our method. Contamination is the primary problem in obtaining consistent and accurate results. Sample collection, processing, subsampling, storage, atmospheric contaminants, and each analytical step are potential contributors to this problem.

We collected streamwater samples with automatic proportional stream samplers (Fredriksen 1969) that pumped the samples into acid-cleaned 20-L Nalgene carboys, which were protected by plastic garbage cans buried in the ground to minimize rapid changes in temperature and resultant loss of free NH_3 . Protection from light limits the growth of bacteria and mold and prevents elevated levels of total nitrogen caused by contaminants. We collected composite samples at 3-week intervals and transported them to the laboratory as quickly as possible where they were filtered and analyzed immediately, or frozen until analyses could be done. During the summer months, we collected grab samples in 8-L, acid-rinsed containers once every 3 weeks and transported them to the laboratory in refrigerated chests. These samples of relatively unaltered streamwater can be frozen for up to 3 months without detectable loss of total nitrogen. There is some loss of $\text{NO}_3\text{-N}$ during storage, however, and an aliquot must be removed for immediate $\text{NO}_3\text{-N}$ analysis before freezing. The presence of heavy metals (including Hg) results in low recovery of N and may interfere with Nesslerization and with the determination of inorganic phosphorus. Calcium (above 250 mg/L) and the amines may also cause some interference.

We isolate the entire analytical process in a separate laboratory to minimize contamination from atmospheric sources (smoke, volatile contaminants, and nitrogenous reagents). All glassware and containers used in collection and analyses are used only for analyses in the Kjeldahl room. Washing of glassware and sample bottles, filtering and subsampling of stream samples, preparation of reagents, and the entire Kjeldahl analysis are conducted exclusively in this one laboratory. Smoking is not permitted, and only reagents used in this analysis should be stored here. Every container that will come in contact with a sample or a reagent used in the procedure has been treated for 1 week in 2 N HCl, thoroughly rinsed with DIW, and then stored covered or upside-down in the Kjeldahl laboratory until needed. Detergents and soaps are not used and are not kept in the laboratory.

Regular maintenance and cleaning of the Kjeldahl system is important. Connect 800-mL Kjeldahl flasks containing 400 mL of the trap solution to the distillation unit when it is not in use to prevent contamination (Crawther and Evans 1980). Keep the tips of the condenser delivery tubes under water. Before each use distill about 200 mL of water to flush the system. Periodically the distillation traps, stoppers, and glass tips of the condenser delivery tubes need to be removed, soaked in acid, thoroughly rinsed with DIW, and reinstalled. Rinse the stoppers daily with dilute HCl when the system is in use. The digestion manifold is also rinsed daily. Change the trap solution monthly, and follow a strict maintenance schedule to check for leaks, broken tubing, stiff corks, and decreased burner efficiency. Document any problems encountered as a means of evaluating past test results and building a solid base of information for future system operators.

Conclusions

The modified Kjeldahl procedure described has been used successfully in our laboratory for over 10 years with only minor modifications—such as switching from DDW to DIW, modifying the digestion mixture, and using some commercially prepared reagents. Large numbers of water samples have been analyzed for inorganic and organic N concentrations in the range of 0.02 to 1.5 mg/L with excellent precision and efficiency. A trained technician can analyze 100 to 150 samples (500 mL) per week for free $\text{NH}_4\text{-N}$ and soluble organic-N. Many more samples can be analyzed if the sample volume is smaller and only total N is determined.

English Equivalents

1 gram (g) = 2.2046×10^{-3} pounds (avdp)
 1 milligram (mg) = 2.2046×10^{-6} pounds (avdp)
 1 liter (L) = 2.1134 pints (U.S., liq.)
 1 milliliter (mL) = 2.1134×10^{-3} pints (U.S., liq.)
 1 meter (m) = 39.37 inches
 1 centimeter (cm) = 39.37×10^{-2} inches
 1 millimeter (mm) = 39.37×10^{-3} inches
 1 nanometer (nm) = 39.37×10^{-9} inches
 1 milligram per liter (mg/L) = 1 part per million parts

Literature Cited

- American Public Health Association, American Water Works Association, and Water Pollution Control Federation.** Standard methods for the examination of water and wastewater. 14th ed. Washington, DC: American Public Health Association; **1976**: 407-415, 437-440.
- Crawther, Joan; Evans, John.** Automated distillation-spectrophotometry procedure for determining ammonia in water. *Analyst*. 105: 841-848; **1980**.
- Fredriksen, R.L.** A battery powered proportional stream water sampler. *Water Resources Research*. 5(6): 1410-1413; **1969**.
- Fredriksen, R.L.** Nutrient budget of a Douglas-fir forest on an experimental watershed in western Oregon. In: Franklin, J.F.; Dempster, L.J.; Waring, R.H., eds. *Research on coniferous forest ecosystems: first year progress in the coniferous biome, US/IBP*. Portland, OR: U.S. Department of Agriculture, Forest Service, Pacific Northwest Forest and Range Experiment Station; **1972**: 115-131.
- Kjeldahl, J.** A new method for the determination of nitrogen in organic matter. *Zeitschrift für Analytische Chemie*. 22: 366; **1883**.
- Morrison, George.** Increased sensitivity for determining ammonia in biological samples with Nessler's reagent. *Analytical Biochemistry*. 43: 527-532; **1971**.
- Rainwater, F.H.; Thatcher, L.L.** Methods for determination of organic substances. In: U.S. Geol. Survey Water-Supply Pap. 1454. Washington, DC: U.S. Geological Survey; **1960**: 211-215.
- United States Environmental Protection Agency.** Methods for chemical analysis of water and wastes. EPA-600/4-79-020. Washington, DC: U.S. Environmental Protection Agency; **1979**. 460 p.

The Forest Service of the U.S. Department of Agriculture is dedicated to the principle of multiple use management of the Nation's forest resources for sustained yields of wood, water, forage, wildlife, and recreation. Through forestry research, cooperation with the States and private forest owners, and management of the National Forests and National Grasslands, it strives — as directed by Congress — to provide increasingly greater service to a growing Nation.

The U.S. Department of Agriculture is an Equal Opportunity Employer. Applicants for all Department programs will be given equal consideration without regard to age, race, color, sex, religion, or national origin.

Pacific Northwest Research Station
319 S.W. Pine St.
PO. Box 3890
Portland, Oregon 97208