Designation: D 3590 – 89 (Reapproved 1994)ε1

Standard Test Methods for Total Kjeldahl Nitrogen in Water [1]

This standard is issued under the fixed designation D 3590; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Section 24, Keywords, was added in April 1994.

1. Scope *

1.1 These test methods cover the determination of total Kjeldahl nitrogen. The following test methods are included:

	Sections
Test Method A—Manual Digestion/Distillation	8 to 14
Test Method B—Semiautomated Colorimetric Bertholt	15 to 23

1.2 The analyst should be aware that precision and bias statements included may not necessarily apply to the water being tested.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water [2]

D 1193 Specification for Reagent Water [2]

D 1426 Test Methods for Ammonia Nitrogen in Water [2]

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water [2]

D 3370 Practices for Sampling Water from Closed Conduits [2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *total Kjeldahl nitrogen*—the sum of the nitrogen contained in the free ammonia and other nitrogen compounds which are converted to ammonium sulfate [$(NH_4)_2SO_4$] under the specified digestion conditions.

4. Significance and Use

4.1 These test methods are useful for measuring organic nitrogen and ammoniacal nitrogen, which are essential growth nutrients.

4.2 Nitrogen compounds are widely distributed in the environment. Sources of nitrogen include surface-applied fertilizers, cleaning products, and drinking water treatment aids. Because nitrogen is a nutrient for photosynthetic organisms, it may be important to monitor and control discharge into the environment.

5. Interferences

5.1 Nitrate is known to cause a serious negative interference in the test. Reportedly, a concentration of 250 mg/L NO_3 results in zero recovery of mg/L N.

5.2 The analyst is cautioned that ammonia in the laboratory may easily become an interference in these test methods from contamination of reagents, caps, or from the laboratory atmosphere. Care should be taken that ammonium hydroxide, either as a reagent or as a cleaning substance, is not used in the same room.

6. Purity of Reagents

6.1 Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. [3] Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean Type III reagent water conforming to Specification D 1193 for reagent water prepared by the passage through a strong, acid-cation exchange resin in the hydrogen form.

7. Sampling and Preservation

7.1 Collect the sample in accordance with applicable Practices D 3370.

7.2 Samples may be preserved up to 28 days by adding concentrated sulfuric acid to adjust to pH 2 or less and storing

[1] These test methods are under the jurisdiction of ASTM Committee D-19 on Water and are the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

***A Summary of Changes section appears at the end of this standard.**

at 4°C. The preserved sample should be analyzed as soon as possible; data on decomposition are not available.

TEST METHOD A—MANUAL DIGESTION/ DISTILLATION

8. Scope

8.1 This test method covers the determination of total Kjeldahl nitrogen in water. It measures free ammonia or ammonia formed from the conversion of nitrogen components of biological origin such as amino acids and proteins. However, the procedure may not convert the nitrogenous compounds of some wastes to ammonia. Examples of such compounds that may not be measured are nitro compounds, hydrozones, oximes, nitrates, semicarbazones, pyridines, and some refractory tertiary amines.

8.2 Three alternatives are described for the final determination of the ammonia: a titrimetric method, which is applicable to concentrations above 1 mg N/L; a Nesslerization method, which is applicable to concentrations below 1 mg N/L; and a potentiometric method which is applicable to the range from 0.04 to 1000 mg N/L.

8.3 This test method is described for micro and macro systems. Micro determination can be made on sample aliquots containing up to 10 mg of nitrogen.

9. Summary of Test Method

9.1 The sample is heated in the presence of concentrated H_2SO_4, K_2SO_4, and $HgSO_4$, and is digested until SO_3 fumes are obtained and the solution becomes colorless or pale yellow. The residue is cooled, diluted, and is treated and alkalized with a hydroxide-thiosulfate solution. The ammonia is distilled into a boric acid solution and total Kjeldahl nitrogen is determined by colorimetry, titrimetry, or potentiometry.

10. Apparatus

10.1 *Digestion Apparatus*—A Kjeldahl digestion apparatus with 800 to 100-mL flasks and suction takeoff to remove SO_3 fumes and water.

10.2 *Distillation Apparatus* [4]—A macro Kjeldahl flask connected to a condenser and an adaptor so that the distillate can be collected.

10.3 *Spectrophotometer or Colorimeter*, for use at 425 nm with a spectral band path of not more than ± 20 nm and a light path of 1 cm or longer.

10.4 *Electrometer* (*pH Meter*), with expanded millivolt scale, or a specific ion meter.

10.5 *Ammonia Selective Electrode*. [5]

10.6 *Magnet Stirrer*, thermally insulated.

11. Reagents and Materials

11.1 *Ammonia Solution Stock*, (1.0 mL = 1.0 mg ammonia nitrogen)—Dissolve 3.819 g of ammonium chloride (NH_4Cl) in water and dilute to 1 L in a volumetric flask with water.

11.2 *Ammonia Solution, Standard* (1.0 mL = 0.01 mg ammonia nitrogen)—Dilute 10.0 mL of the stock solution (see 11.1) with water to 1 L in a volumetric flask.

11.3 *Boric Acid Solution* (2 %)—Dissolve 20 g of boric acid (H_3BO_3) in water and dilute to 1 L with water in a volumetric flask.

11.4 *Mercuric Sulfate Solution*—Dissolve 8 g of red mercuric oxide (HgO) in a mixture of 10 mL of sulfuric acid (H_2SO_4, sp gr 1.84) and 40 mL of water, and dilute solution to 100 mL.

11.5 *Mixed Indicator Solution*—Mix 2 volumes of 0.2 % methyl red in 95 % ethanol with 1 volume of 0.2 % methylene blue in ethanol. Prepare fresh every 30 days.

11.6 *Methyl Purple Indicator Solution* (1 g/L)—Dissolve 0.4 g of dimethyl-aminoazobenzene-*o*-carboxylic acid, sodium salt, in approximately 300 mL of water. To this solution add 0.55 g of a water-soluble blue dyestuff, Color Index No. 714,[6] dissolve, and dilute to 1 L with water. This indicator is available commercially in a prepared form.[7]

11.7 *Nessler Reagent*—Dissolve 100 g of mercuric iodide (HgI_2) and 70 g of potassium iodide (KI) in a small volume of water. Add this mixture slowly, with stirring, to a cooled solution of 160 g of sodium hydroxide (NaOH) in 500 mL of water. Dilute the mixture to 1 L. This solution is stable for at least one year if stored in a thick amber polyethylene bottle out of direct sunlight.

11.8 *Phenolphthalein Indicator Solution*—Dissolve 5 g of phenolphthalein in 500 mL of 95 % ethyl alcohol or isopropanol and add 500 mL of water. Add NaOH (0.8 g/L) solution dropwise until a faint pink color appears.

11.9 *Sodium Hydroxide Solution* (400 g/L)—Dissolve 400 g of NaOH in 800 mL of water, cool, and dilute to 1 L with water.

11.10 *Sodium Hydroxide Solution* (0.8 g/L)—Dilute 2 mL of NaOH solution (400 g/L) (see 11.9) with water to 1 L.

11.11 *Sodium Hydroxide-Sodium Thiosulfate Solution*—Dissolve 500 g of NaOH and 25 g of $Na_2S_2O_3 \cdot 5H_2O$ in water and dilute to 1 L.

11.12 *Sulfuric Acid Solution, Standard* (0.02 N, 1 mL = 0.28 mg ammonia nitrogen)—Prepare a stock solution of approximately 0.1 *N* acid by diluting 3 mL of concentrated H_2SO_4(sp gr 1.84) to 1 L with water. Dilute 200 mL of this solution to 1 L with water. Standardize the approximately 0.02 *N* H_2SO_4 solution against 0.0200 *N* Na_2CO_3 solution. This last solution is prepared by dissolving 1.060 g of anhydrous Na_2CO_3, oven dried at 140°C, and diluting to 1 L with water.

11.13 *Digestion Solution*—Dissolve 267 g of K_2SO_4 in 1300 mL water and 400 mL of concentrated H_2SO_4. Add 50 mL of mercuric sulfate solution (see 11.4) and dilute to 2 L with water. A digestion packet [8] may be used in place of the digestion solution in the macro Kjeldahl system.

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[4] Micro Kjeldahl steam distillation apparatus is commercially available.

[5] EIL Model 8002-2 of Electronics Instruments Ltd. (U. S. Representative: Cambridge Instrument Co., 73 Spring St., Ossining, NY 10562) has been found satisfactory for this purpose. Also, Orion Model 95-12 has been found satisfactory for this purpose.

[6] Refers to compounds, bearing such number, as described in "Color Index," Society of Dyers and Colourists, Yorkshire, England (1924). American Cyanamid Company's "Calcocid Blux AX Double" has been found satisfactory for this purpose.

[7] TM Fleisher Methyl Purple indicator, U. S. Patent No. 241699, is available from Fleisher Chemical Co., P. O. Box 616, Ben Franklin Station, Washington, DC 20004, or from any chemical supply company handling Fleisher Methyl Purple.

[8] Digestion packet, Kel Pak No. 5, available from the Curtin-Matheson Scientific Co., has been found satisfactory for this purpose.

12. Procedure

12.1 Clean the distillation apparatus with steam before use by distilling a 1 + 1 mixture of water and sodium hydroxide-thiosulfate solution (see 11.11) until the distillate is ammonia-free. Repeat this operation each time the apparatus is out of service long enough to accumulate ammonia (usually 4 h or more).

12.2 *Macro Kjeldahl System*:

12.2.1 Place a measured sample into an 800-mL Kjeldahl flask and dilute to 500 mL. The sample size can be determined using the following table:

Kjeldahl Nitrogen in Sample, mg/L	Sample Size, mL
0 to 5	500
5 to 10	250
10 to 20	100
20 to 50	50.0
50 to 500	25.0

Prepare a 500-mL reagent water blank.

12.2.2 Add 100 mL of digestion solution (see 11.13) (see Note 1) and digest the mixture in the Kjeldahl apparatus until SO_3 fumes are given off and the solution turns colorless or pale yellow. Continue heating for an additional 30 min. Cool the residue and add 300 mL of water. Mix well.

NOTE 1—Digesting the sample with a packet [8] and 20 mL of concentrated H_2SO_4 is acceptable. Cut the end of the package and empty the contents into the digestion flask.

12.2.3 Alkalize the digestate by careful addition of 100 mL of sodium hydroxide-thiosulfate solution (see 11.11). Do not mix until the digestion flask has been connected to the distillation apparatus (see 12.2.4).

NOTE 2—Slow addition of the heavy caustic solution down the tilted neck of the digestion flask will cause the heavier solution to underlay the aqueous H_2SO_4 without loss of free ammonia.

12.2.4 Connect the Kjeldahl flask to the condenser with the tip of the condenser (or an extension of the condenser tip) below the level of 50 mL of 2 % boric acid solution (see 11.3) contained in a 500-mL Erlenmeyer flask. Distill 300 mL at the rate of 6 to 10 mL/min.

12.2.5 Transfer the distillate to a 500-mL volumetric flask, dilute to volume with water, and mix. Transfer 250 mL to an Erlenmeyer flask and titrate with H_2SO_4 (see 12.4.1). If the concentration is found to be below 1 mg/L, determine the value colorimetrically. Use the remaining 250 mL for this determination.

12.3 *Micro Kjeldahl System*:

12.3.1 Place 50.0 mL of sample or an aliquot in a 100-mL Kjeldahl flask and add 10 mL of digestion solution (see 11.13). At the same time start a reagent blank. Evaporate the mixture in the Kjeldahl apparatus until SO_3 fumes are given off and the solution turns colorless or pale yellow. Digest for an additional 30 min. Cool the residue and add 30 mL of water.

12.3.2 Alkalize the digestate by careful addition of 10 mL of sodium hydroxide-thiosulfate solution (see 11.11). Do not mix until the digestion flask has been connected to the distillation apparatus (see Note 2).

12.3.3 Connect the Kjeldahl flask to the condenser with the tip of the condenser (or an extension of the condenser tip) below the level of 5 mL of 2 % H_3BO_3 solution (see 11.3) contained in a small Erlenmeyer flask. Distill 30 mL at the rate of 6 to 10 mL/min.

12.3.4 Transfer to a 50-mL volumetric flask, dilute to volume with water, and mix. Pipet 25 mL to an Erlenmeyer flask and titrate with H_2SO_4 (see 12.4.1). If the concentration is found to be below 1 mg/L determine the value colorimetrically. Use 20 mL of the remaining solution for this determination.

12.4 *Determination of Ammonia Distillate*—Determine the ammonia content of the distillate titrimetrically, colorimetrically, or potentiometrically.

12.4.1 *Titrimetric Determination*—Add 3 drops of the mixed indicator (see 11.5) to the distillate and titrate the ammonia with 0.02 *N* H_2SO_4 (see 11.12), matching the end point against a blank containing the same volume of water and H_3BO_3 solution (see 11.3). If a pH meter is preferred, titrate to pH 6.2.

NOTE 3—As an alternative, 2 drops of methyl purple indicator solution (see 11.6) may be used and the titration carried out to the intermediate gray end point.

12.4.1.1 *Calibration Curve*—Prepare a series of standards on a daily basis in 50-mL volumetric flasks and dilute as follows:

Millilitres of Standard (see 11.2) 1.0 mL = 0.01 mg NH_3-N	Milligrams of NH_3-N/50.0 mL
0.0	0.0
0.5	0.005
1.0	0.010
2.0	0.020
4.0	0.040
5.0	0.050
8.0	0.080
10.0	0.10

To the standards diluted to 50 mL add 1 mL of Nessler reagent (see 11.7) and mix. After 20 min read the absorbance at 425 nm against the blank using 1-cm cells. From the values obtained for the standards plot a standard curve of absorbance versus milligrams of NH_3-N.

12.4.2 *Colorimetric Determination (Samples)*—To a 20-mL aliquot from the macro procedure (see 12.2.5) or micro procedure (see 12.3.4) diluted to 50 mL, add 1 mL of Nessler reagent (see 11.7), and mix. After 20 min, read the absorbance at 425 nm against the blank using 1-cm cells. Read the ammonia nitrogen in milligrams for the samples from the standard curve.

12.4.3 *Potentiometric Determination*—Test Method B of Test Methods D 1426 should be used for this determination.

12.4.3.1 It is recommended that at least two standards (a high and a low) be digested, distilled, and compared to similar values on the calibration curve to ensure that the digestion-distillation technique is reliable. If treated standards do not agree with untreated standards, the operator should find the cause of the apparent error before proceeding.

13. Calculation

13.1 If the titrimetric procedure is used, calculate the total Kjeldahl nitrogen in the original sample using Eq 1:

total Kjeldahl nitrogen, mg/L

$$= (A - B)N \times F \times 1000/S \times D/C \quad (1)$$

where:
- A = standard 0.02 N H_2SO_4 solution used in titrating sample, mL,
- B = standard 0.02 N H_2SO_4 solution used in titrating blank, mL,
- N = normality of H_2SO_4 solution,
- F = milliequivalent weight of nitrogen (14 mg),
- S = sample digested, mL,
- C = distillate taken for titration, mL, and
- D = final adjusted distillate volume, mL.

If the H_2SO_4 is exactly 0.0200 N and exactly one half of the distillate is taken for measurement, the equation is shortened, as shown in Eq 2:

$$\text{total Kjeldahl nitrogen, mg/L} = (A - B) \times 560/S \quad (2)$$

13.2 If the Nessler procedure is used, calculate the total Kjeldahl nitrogen in the original sample using Eq 3:

$$\text{total Kjeldahl nitrogen, mg/L} = E \times 1000\ /S \times D/C \quad (3)$$

where:
- E = NH_3-H read from curve, corrected for blank, mg,
- D = final adjusted distillate volume, mL,
- C = distillate taken for Nesslerization, mL, and
- S = sample digested, mL.

13.3 If the potentiometric determination is used, calculate the total Kjeldahl nitrogen in the original sample using Eq 4:

$$\text{total Kjeldahl nitrogen, mg/L} = E \times 1000/S \times D/C \quad (4)$$

where:
- E = NH_3-N/L as determined using Test Method B of Test Methods D 1426,
- S = sample digested, mL,
- D = final adjusted volume, mL, and
- C = distillate taken for measurement, mL.

14. Precision and Bias [9]

14.1 Thirty-one analysts in 20 laboratories used titration and Nesslerization to analyze natural water samples containing exact increments of organic nitrogen and obtained the following results:

Amount Added as Nitrogen, Kjeldahl, mg N/L	Amount Found as Nitrogen, Kjeldahl, mg N/L	Precision as Standard Deviation, mg N/L	Bias, %
0.20	0.23	0.197	+ 15.54
0.31	0.33	0.247	+ 5.45
4.10	4.14	1.056	+ 1.03
4.61	4.53	1.191	− 1.67

14.2 The potentiometric test method has not been validated in conjunction with the digestion-distillation procedure described in this standard. However, since the procedure provides a relatively clean sample, it is thought that the user may be guided by the precision and bias information presented in Test Method B of Test Methods D 1426 and by 12.4.3 of this test method. [10]

14.3 The data in Section 14 may not apply to types of water other than those tested. It is the responsibility of the analyst to ensure the validity of this test method for untested matrices.

TEST METHOD B—SEMIAUTOMATED COLORIMETRIC BERTHOLT

15. Scope

15.1 This test method covers the automated determination of total Kjeldahl nitrogen in water and wastewater and is based on the same principle and subject to the same limitations as the manual method (see 8.1).

15.2 This test method is a semiautomated procedure applicable to drinking water, surface water, and domestic and industrial wastes containing from 0.3 to 5 mg/L of nitrogen.

16. Summary of Test Method

16.1 This test method consists of digesting the sample in a block digestor in the presence of H_2SO_4, K_2SO_4, and $HgSO_4$ at approximately 200°C for 1 h and then at 380°C for 1½h. (The block digestor is an electrically-heated metal block designed to hold block-digestor reaction tubes). [11] During the digestion, organic nitrogen is converted to ammonium ion. After digestion, the residue is cooled, dissolved in water, and an aliquot is transferred to a segmented-flow automated analysis system. The ammonia nitrogen content is determined by means of the automated analysis system using a modification of the Bertholt reaction.

16.2 Ammoniacal nitrogen is determined by means of the salicylate/nitroprusside Bertholt reaction method.

17. Interferences

17.1 Within the analytical range of this test method, there are no significant interferences, with the exception of nitrate at the 200 mg/L level. [12]

17.2 Contamination from airborne nitrogen compounds will cause high results. Washing floors with ammonia solutions in the Kjeldahl nitrogen-analysis laboratory should be avoided.

18. Apparatus

18.1 *Continuous Segmented-Flow Automated Analysis System*, consisting of the following:

18.1.1 *Sampler*—40 samples per hour, 6 + 1, sample time 77 s, wash time 13 s.

18.1.2 *Proportioning Pump*.

18.1.3 *Analytical Cartridge*, with glassware and tubing per Fig. 1.

[9] Supporting data are available from ASTM Headquarters. Request RR D19-1041.

[10] The Results Advisor of Committee D-19 on Water has reviewed and approved this statement for conformity with the requirements of Practice D 2777 – 85; the Technical Operations Section of Executive Subcommittee D19.90 has supported this approval.

[11] The block digestor and continuous flow instruments capable of performing the digestion procedure are available from Scientific Instrument Corp., Hawthorne, NY or The Technicon Instrument Corp, Tarrytown, NY.

[12] EPA-600/7-77-017 report available from Environmental Protection Agency, 401 "M" St., S.W., Washington, DC 20406.

18.1.4 *Colorimeter*, equipped with a 50-mm tubular flow cell and 660 ± 10-nm filters.

18.1.5 *Recorder*.

18.2 *Block Digestor*, capable of operating at 200 and 380°C.[10]

18.3 *Mixer*, vortex.

NOTE 4—All connection nipples and the sample probe used within the automated system should be inert to acid solutions.

19. Reagents and Materials

19.1 *Ammonia Solution, Stock* (1.0 mL = 1.0 mg ammonium nitrogen)—See 11.1.

19.2 *Ammonia Solution, Standard* (1.0 mL = 0.01 mg ammonium nitrogen)—See 11.2.

19.3 *Buffer Solution, Stock*—Dissolve 134 g of sodium phosphate, dibasic (Na_2HPO_4)·H_2O, or 71.0 g sodium phosphate anhydrous Na_2HPO_4, in 800 mL of water. Add 20 g of sodium hydroxide and dilute to 1 L.

19.4 *Buffer Solution, Working*—Combine the reagents in the following order: add 250 mL of stock sodium potassium tartrate solution (see 19.10) to 200 mL of stock buffer solution (see 19.3) and mix. Add 100 mL of sodium hydroxide (200 g/L solution) (see 19.7) and dilute to 1 L with water. Add 1 mL of Brij-35 [13], 30 % solution.

19.5 *Digestion Solution*—See 11.13.

19.6 *Mercuric Sulfate Solution*—See 11.4.

19.7 *Sodium Hydroxide Solution* (200 g/L)—Dissolve 200 g of NaOH in 800 mL of water, cool, and dilute to 1 L with water.

19.8 *Sodium Hypochlorite Solution*—Dilute 6 mL of sodium hypochlorite (household bleach 5.25 %) solution to 100 mL with water. Add 0.2 mL (5 drops) of Brij-35 (30 % solution). Alternatively, 1 g of dichloroisocyanuric acid sodium salt dissolved in 1 L can be used as the chlorinating agent.

19.9 *Sodium Chloride Saline Diluent* (20 g/L)—Dissolve 20 g NaCl in 600 mL of water. Add 1 mL Brij-35, mix, and dilute to 1 L with water and mix.

19.10 *Sodium Potassium Tartrate Solution, Stock* (200 g/L)—Dissolve 200 g of sodium potassium tartrate in 800 mL of water and dilute to 1 L.

19.11 *Sodium Salicylate/Sodium Nitroprusside Solution*—Dissolve 75 g of sodium salicylate and 0.3 g of sodium nitroprusside in about 600 mL of water and dilute to 1 L. Add 1 mL of Brij-35, 30 % solution.

19.12 *Sulfuric Acid Wash Solution*—Dissolve 31.7 g of potassium sulfate in 800 mL of water. Add slowly 48 mL of concentrated sulfuric acid (sp gr 1.84) and dilute to 1 L with water.

20. Calibration

20.1 Prepare standard solutions containing 0, 2.5, and 5 mg/L N by diluting appropriate aliquots of the ammonia standard solution to 100 mL with water.

NOTE 5—The analytical curve obtained by this procedure is linear to 5.0 mg/L.

20.2 Transfer by pipet 20 mL of the blank and each of the prepared standard solutions to separate block-digestor tubes. Digest solutions as described in Section 21.

20.3 Prepare an analytical curve by plotting peak heights of blank and standards versus concentration (mg/L TKN) on linear graph paper. Alternatively, electronic peak reading devices may be used to calibrate the instrument in concentration terms.

21. Procedure

21.1 *Digestion*:

21.1.1 Add 5 mL of digestion solution and 20 or 25 mL of a well-mixed sample into a digestion tube and mix, using a vortex mixer. Add 4 or 5 acid-washed TFE-fluorocarbon boiling stones.

NOTE 6—The same acid concentration is used in the sampler wash receptacle as in the digestion tube. See Fig. 1.

21.1.2 Place tube in digestor that has been preheated to 200°C. Heat at 200°C for 1 h, raise the temperature to 380°C, and heat for an additional 1½h (1½ h digestion at 380°C).

21.1.3 After 21.1.2 has been completed, cool sample (25 ± 5°C), add 20 mL of water, and mix thoroughly. Place an aliquot of this digested sample in a sample cup.

NOTE 7—High-temperature digestion of aqueous sample in sulfuric acid requires the use of an efficient fume hood with wash-down facilities.

21.2 *Colorimetric Determination*:

21.2.1 Check the level of all reagent containers to ensure an adequate supply of reagent to the automated system.

21.2.2 Except for the sodium salicylate/sodium nitroprusside solution line, place all reagent lines in their respective containers, connect the sample probe to the sampler, and start the proportioning pump.

21.2.3 Flush the sampler wash receptacle with sulfuric acid wash solution (see 19.12).

21.2.4 When reagents have been pumping for at least 5 min, place the sodium salicylate/sodium nitroprusside solution line in its respective container and allow the system to equilibrate.

21.2.5 After a stable baseline has been obtained, initiate sampler and analyze blank, standards, and samples.

22. Calculation

22.1 Determine the concentration of TKN (mg/L N) by referring the peak heights obtained for each sample to the prepared analytical curve (see 20.3).

22.2 Calculate the sample concentration using Eq 5:

$$\text{total Kjeldahl nitrogen, mg/L} = (A \times F) - B \quad (5)$$

where:

A = concentration mg/L, found in sample aliquot, mg/L,
B = concentration mg/L, found in blank, mg/L, and
F = dilution factor, if any.

23. Precision and Bias [14]

23.1 The collaborative test of the semiautomated colorimetric Bertholt test method was performed at three levels in a

[13] Brij-35, a registered trademark product of Atlas Chemical Co., is available from Fisher Scientific, Pittsburgh, PA 15238.

[14] Supporting data are available from ASTM Headquarters. Request RR:D19-1101.

water of choice and at four levels in reagent water (Type II). The analyses were conducted by six laboratories (six operators), each performing replicates for each level and water type of three separate days.

23.2 Recoveries of known amounts of total Kjeldahl nitrogen from reagent water Type II and selected water matrices for the same laboratories are described in Table 1.

23.3 These collaborative test data were obtained on reagent-grade water and various other selected water types (that is, sewage wastewater (municipal and industrial), estuary waters, and well water). For other matrices, these data may not apply.

24. Keywords

24.1 analysis; colorimetric; distilation; TKN; water

SUMMARY OF CHANGES

This section identifies the locations of selected changes to these test methods that have been incorporated since the last issue. For the convenience of the user, Committee D-19 has highlighted those changes that may impact the use of these test methods. This section may also include descriptions of the changes or reasons for the changes, or both.

(1) The potentiometric determination portion of Test Method A (Manual Digestion/Distillation) was reinstated.

TABLE 1 Recoveries of Known Amounts of Total Kjeldahl Nitrogen

	Amount Added, mg/L	Amount Found, mg/L	Precision, mg/L		% Bias	Statistically Significant (95 % Confidence Level)
			S_T	S_O		
Reagent water, Type II	0.10	0.17	0.13	0.07	+ 70	yes
	1.0	1.18	0.28	0.10	+ 18	yes
	2.0	2.05	0.13	0.11	+ 2.5	no
	4.0	3.94	0.13	0.17	−1.5	no
Water of choice	1.0	1.07	0.14	0.08	+ 7	no
	2.0	2.03	0.12	0.14	+ 1.5	no
	2.5	2.44	0.18	0.15	−2.4	no

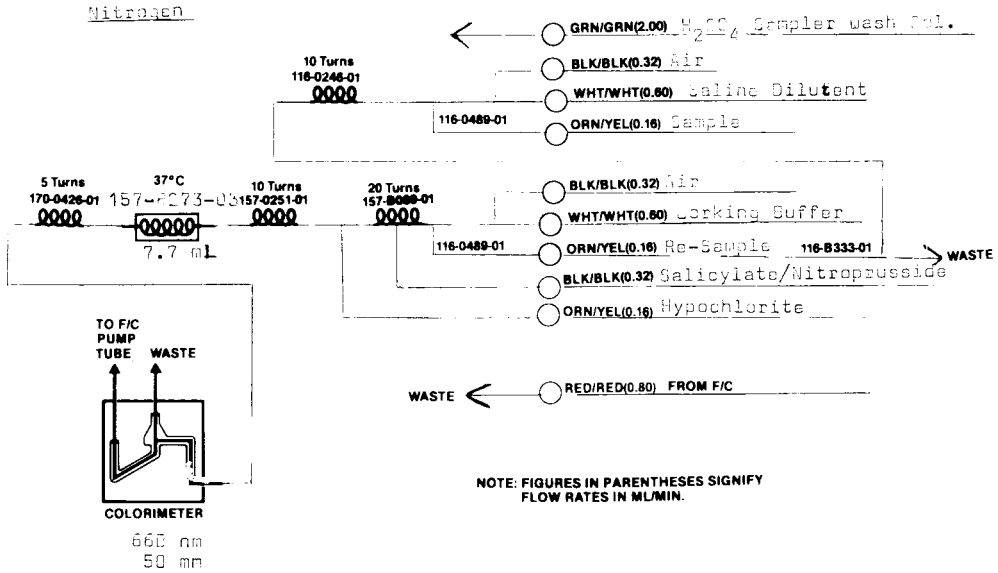

FIG. 1 Digestion Tube