Designation: D 3179 – 89 (Reapproved 1997)

Standard Test Methods for Nitrogen in the Analysis Sample of Coal and Coke[1]

This standard is issued under the fixed designation D 3179; 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.

1. Scope

1.1 These test methods cover the determination of total nitrogen in samples of coal and coke. The analytical data from these test methods shall be reported as part of ultimate analysis where ultimate analysis is requested. If ultimate analysis is not requested, the value shall be reported according to the request. Two methods are included as follows:

	Sections
Test Method A—Kjeldahl-Gunning Macro Analysis, with an alternative technique included	9 to 16
Test Method B—Kjeldahl-Gunning Semi-Micro Determination	17 to 23

1.2 *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.*

1.3 The values stated in SI units are to be regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 121 Terminology of Coal and Coke[2]

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis[2]

D 1193 Specification for Reagent Water[3]

D 2013 Method of Preparing Coal Samples for Analysis[2]

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke[2]

D 3176 Practice for Ultimate Analysis of Coal and Coke[2]

D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases[2]

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)[4]

3. Terminology

3.1 For definitions of terms used in these test methods, refer to Definitions D 121. For an explanation of the metric system including units, symbols, and conversion factors, see Practice E 380.

4. Summary of Test Methods

4.1 In these procedures, nitrogen is converted into ammonium salts by destructive digestion of the sample with a hot, catalyzed mixture of concentrated sulfuric acid and potassium sulfate. These salts are subsequently decomposed in a hot alkaline solution from which the ammonia is recovered by distillation and finally determined by alkalimetric or acidimetric titration.

5. Significance and Use

5.1 Nitrogen results obtained by these test methods are required to fulfill the requirements of the ultimate analysis, Test Method D 3173. Also, results obtained may be used to evaluate the potential formation of nitrogen oxides as a source of atmospheric pollution.

5.2 Nitrogen data are used in comparing coals and in research. When the oxygen content of coal is estimated by difference, it is necessary to make a nitrogen determination.

6. Interferences

6.1 No significant interferences have been determined using these procedures. However, strict adherence is necessary when using these nitrogen procedures to obtain good reproducible results.

7. Reagents

7.1 *Purity of Reagents*—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.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to meet its use without lessening the accuracy of the determination.

7.2 *Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent water, conforming to Specification D 1193.

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[1] These test methods are under the jurisdiction of ASTM Committee D-5 on Coal and Coke and are the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

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[2] *Annual Book of ASTM Standards,* Vol 05.05.

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

[4] *Annual Book of ASTM Standards,* Vol 14.02. Excerpts appear in the gray pages of all the volumes.

[5] *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.

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8. Sampling and Preparation

8.1 The sample shall be the material pulverized to pass No. 60 (250-µm) sieve and well mixed in accordance with Method D 346 or Method D 2013. In the case of coke and anthracite, grinding the sample to pass a No. 200 (75-µm) or finer sieve is recommended.

8.2 A separate portion of the analysis sample should be analyzed for moisture content in accordance with Test Method D 3173, in order to allow calculation of the as-analyzed data to other bases.

TEST METHOD A—MACRO-NITROGEN DETERMINATION WITH ALTERNATIVE METHOD INCLUDED

9. Scope and Application

9.1 This test method describes a macro procedure for the determination of nitrogen in both coal and coke, by two alternative procedures. In both procedures, a 1 g sample is digested with a hot catalyzed mixture of concentrated sulfuric acid and potassium sulfate, which converts the nitrogenous compounds to ammonium salts. The salts are then decomposed in a hot alkaline solution, releasing the ammonia, which is then distilled into either standard sulfuric-acid or boric-acid solution and finally determined by alkalimetric or acidimetric titration.

10. Apparatus

10.1 *Digestion Unit*—An electrically heated digestion rack or a gas burner; either type of heater shall be provided with adequate means of control to maintain digestion rates as described in 12.1. It is essential that an electric digestion rack provides adjustable controls to regulate desirable digestion temperatures. To eliminate emission of sulfur-acid fumes, the digestion process must be carried out under a well-ventilated fume hood. Commercially made multiple-unit digestion racks provided with fume exhaust ducts may be used.

10.2 *Digestion Flasks*—Made of heat-resistant glass,[6] having a capacity of 500 or 800 mL.

10.3 *Distillation Unit*—A suitable glass steam distillation unit with a splash head to trap any entrained caustic soda and also provided with adequate means of control to maintain distillation rates as described in 12.1. Commercially made multiple unit distillation racks provided with water-cooled glass or block-tin condensers may be used.

10.4 *Buret*—Microburet graduated in 0.01 mL. A50 mL microburet is needed for Method A.

10.5 *Erlenmeyer Flask*—Having a capacity of 250 to 300 mL.

10.6 *Rubber Tubing*—Sufficient for attaching condenser to cooling water supply and drain.

10.7 *Pipets*—As required.

11. Reagents

11.1 *Alkali Solution*—Cautiously dissolve 8.0 g of potassium sulfide (K_2S) and 500 g of sodium hydroxide (NaOH) (**Warning**—This solution becomes very hot.) Cool the solution and dilute to 1 L. The use of appropriate amounts of sodium sulfide (Na_2S) or potassium hydroxide (KOH) may be substituted (Note 1(*3*)).

11.2 *Ethyl Alcohol* (95 %)—Ethyl alcohol conforming to Formula No. 30 or 2A of the U.S. Bureau of Internal Revenue. Methyl alcohol may be used.

11.3 *Mercury*.

NOTE 1—Other satisfactory and permissible catalysts for the digestion, together with the quantities of K_2SO_4 required in their use are as follows:

(1) Five grams of a mixture containing 32 parts by weight of K_2SO_4, 5 parts by weight of mercuric sulfate ($HgSO_4$), and one part by weight of selenium.

(2) Three-tenths gram of mercuric selenite ($HgSeO_3$) with 7 to 10 g of K_2SO_4.

(3) Three-tenths gram of cupric selenite dihydrate ($CuSeO_3 \cdot 2H_2O$) with 7 to 10 g of K_2SO_4. When this mixture is used, the addition of a sulfide to the alkali solution is not necessary.

11.4 *Potassium Permanganate* ($KMnO_4$), crystals.

11.5 *Potassium Sulfate* (K_2SO_4), crystals.

11.6 *Sucrose*, National Institute of Standards and Technology primary-standard grade.

11.7 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

11.8 *Zinc*, mossy or granular.

REAGENTS REQUIRED ONLY FOR KJELDAHL-GUNNING METHOD

11.9 *Methyl Red Indicator Solution* (0.4 to 1 g/L)—Dissolve 0.04 to 0.1 g of methyl red in 50 mL of 95 % ethyl alcohol or methyl alcohol and add 50 mL of water. Bromcresol green solutions to equal concentrations may be used.

11.10 *Sodium Hydroxide, Standard Solution* (0.1 to 0.2 *N*)—Prepare and accurately standardize a 0.1 to 0.2 *N* sodium hydroxide (NaOH) solution against a primary standard.

11.11 *Sulfuric Acid* (0.2 *N*)—Prepare and standardize a 0.2 *N* sulfuric acid (H_2SO_4) solution. The solution need not be standardized against a primary standard.

REAGENTS REQUIRED ONLY FOR ALTERNATIVE METHOD

11.12 *Boric Acid Solution* (50 g/L)—Dissolve 5 g of boric acid (H_3BO_3) in 100 mL of boiling water. Allow to cool before use.

11.13 *Mixed Indicator Solution*—Prepare a solution containing 0.125 % methyl red and 0.083 % methylene blue in 95 % ethyl alcohol or in methyl alcohol. Prepare a fresh solution at bimonthly intervals.

11.14 *Sulfuric Acid* (0.1 to 0.2 *N*)—Prepare and accurately standardize a 0.1 to 0.2 *N* sulfuric acid (H_2SO_4) solution against a primary standard; hydrochloric acid (HCl) of similar concentration may be substituted.

12. Procedure

12.1 Weigh approximately 1 g (weighed to nearest 1 mg) of the analysis sample and carefully transfer into a 500 or 800-mL Kjeldahl flask containing 7 to 10 g of K_2SO_4 and 0.6 to 0.8 of mercury (Note 1). Add 30 mL of H_2SO_4(sp gr 1.84) to the mixture by pouring down the neck of the flask with rotation, in order to wash any adherent sample material into the mixture.

[6] Borosilicate glass has been found satisfactory for this purpose.

Swirl the contents of the flask several times to ensure thorough mixing and wetting of the sample. Incline the flask at an angle of 45 to 60° on the digestion heater in a fume hood (Note 2), and heat the contents to boiling; controlling the heat so the H_2SO_4 vapors condense no more than halfway up the neck of the flask. Continue the boiling until all sample particles are oxidized, as evidenced by a nearly colorless solution, or for at least 2 h after the solution has reached a straw-colored stage. The total time of digestion will require 3 to 6 h, except in the case of coke and anthracite, which may require 12 to 16 h (Note 3). When the digestion is completed and the solution has cooled, a few crystals of $KMnO_4$ may be added to ensure complete oxidation; further heating may be necessary to destroy the excess permanganate and decolorize the solution.

NOTE 2—When fume exhaust ducts or hoods are not available, a Hengar tube may be inserted in the neck of the flask.

NOTE 3—Addition of 0.1 g of chromic trioxide (CrO_3) to the digestion mixture has been found very helpful in reducing the time of digestion for coke.

12.2 Dilute the cooled digestion mixture to about 300 mL with cold water, and remove any heat of dilution by cooling with water. Meanwhile, pipet into the 250 or 300-mL Erlenmeyer flask, 20.0 mL of 0.2 *N* H_2SO_4 and add 6 drops of methyl red or bromcresol green indicator solution. Attach the glass connecting tube to the discharge end of the condenser, using the short piece of rubber tubing as a seal. Incline the Erlenmeyer flask at a suitable angle, and insert this tube so that the end is immersed to the maximum depth in the acid. Add 1 to 2 g of granular zinc to the mixture in the Kjeldahl flask (two or three small pieces of mossy zinc is used), and slowly add 100 mL of the alkali solution so that it forms a distinct layer under the acid solution. (**Caution**—If the layers are mixed, considerable heat may be generated with subsequent spray of the flask contents. The flask opening should be directed away from the operator and others during this step.) This may be accomplished by inclining the flask at an angle of 45 to 60° and pouring the alkali solution down the neck. Failure to maintain discrete layers during this operation may lead to loss of ammonia. Quickly connect the flask to the distilling condenser through the Kjeldahl connecting bulb, and then swirl the contents to promote thorough mixing.

12.3 Bring the contents of the Kjeldahl flask to a boil carefully, in order to avoid violent bumping, and then distill the ammonia over into the acid solution in the Erlenmeyer flask. Continue the distillation at a maximum rate of approximately 350 mL/h until 150 to 175 mL of distillate have been collected. Discontinue the boiling, and remove the glass connecting tube from the condenser and Erlenmeyer flask. Rinse the tube with distilled water, collecting the washings in the Erlenmeyer flask, and then back-titrate the excess acid with 0.1 to 0.2 *N* NaOH solution.

12.4 Run a blank determination in the same manner as described in 12.1-12.3 using approximately 1 g of sucrose (weighed to the nearest 1 mg) as the sample material.

13. Calculation and Report

13.1 Calculate the percentage of nitrogen in the analysis sample as follows:

$$\text{Nitrogen, \%} = [(B - A)N \times 0.014/C] \times 100 \quad (1)$$

where:

A = millilitres of NaOH solution required for titration of the sample,
B = millilitres of NaOH solution required for titration of the blank,
N = normality of the NaOH solution, and
C = grams of sample used.

14. Procedure for Alternative Technique

14.1 Digest the sample as described in 12.1.

14.2 Dilute and cool the digestion mixture as described in 12.2. Add to the 250 or 300-mL Erlenmeyer flask approximately 20 mL of H_3BO_3 solution and 5 drops of mixed indicator solution. Then proceed as described in the remainder of 12.2.

14.3 Distill the ammonia into the H_3BO_3 solution exactly as described in 12.3 and finally titrate the ammonia with 0.2 *N* H_2SO_4.

14.4 Run a blank determination in the same manner as described in 14.1-14.3, using approximately 1 g (weighed to the nearest 1 mg) of sucrose as the sample material. Blank determinations must be made to correct for nitrogen from sources other than the sample. A blank determination shall be made whenever a new batch of any one reagent is used in the analysis.

14.5 *Calculation*—Calculate the percentage of nitrogen in the sample as follows:

$$\text{Nitrogen, \%, in the analysis sample} = [(A - B)N \times 0.014/C] \times 100 \quad (2)$$

where:

A = millilitres of H_2SO_4 required for titration of the sample,
B = millilitres of H_2SO_4 required for titration of the blank,
N = normality of the H_2SO_4, and
C = grams of the sample used.

15. Report

15.1 The results of the nitrogen analysis may be reported on any of a number of bases, differing from each other in the manner by which moisture is treated.

15.2 Use the percentage of moisture in the sample passing a No. 60 (250-µm) sieve to calculate the results of the analysis sample to a dry basis.

15.3 Procedures for converting the value obtained on the analysis sample to other bases are described in Methods D 3176 and D 3180.

16. Precision and Bias

16.1 The permissible differences between duplicate determinations shall not exceed the following values in more than 5 of 100 instances.

	Repeatability, %	Reproducibility, %
Nitrogen	To be determined	

16.2 *Bias*—Bias of this stoichiometrically based test method has not been determined at this time. Standard samples

of known certified nitrogen content such as NIST SRM coal samples are to be analyzed at the same time as samples.

TEST METHOD B—KJELDAHL-GUNNING SEMI-MICRO NITROGEN DETERMINATION

17. Scope and Application

17.1 The semi-micro test method differs primarily from the macro-methods in that smaller sized equipment is used, smaller samples are analyzed (0.1 g compared with a 1.0-g sample for the macro-methods), and ammonia is separated from the alkalinized digestion mixture by steam distillation. The same catalysts may be used, although it is more common to use a mixed catalyst in this test method. The acid-base finish may be used, but the boric acid finish is more common.

18. Apparatus

18.1 *Digestion Unit*—An electrically heated digestion rack with adequate means of control to maintain digestion rates as described in 20.3 on which the digestion flasks may be supported at about a 35° angle.

18.2 *Digestion Flasks*, 150 mL, 198-mm length with 29/42 joint.

18.3 *Distillation Unit*—A suitable glass steam distillation unit with a splash head to trap any entrained caustic soda with adequate means of control to maintain proper distillation rate.

18.4 *Buret*—Microburet of minimum 5-mL capacity, graduated in 0.01 mL.

18.5 *Rubber Tubing*—Sufficient for attaching condenser to cooling water supply and drain.

18.6 *Erlenmeyer Flask*, 50 mL, to catch distillate.

18.7 *Pipets*—As required.

19. Reagents

19.1 *Boric Acid Solution*—See 11.12.

19.2 *Chromic Trioxide*, CrO_3.

19.3 *Cupric Selenite Dihydrate*, $CuSeO_3 \cdot 2H_2O$.

19.4 *Indicator Solution*—Dissolve 0.25 g of sodium salt of bromocresol green in 250 mL water.

19.5 *Nessler's Reagent*—Dissolve 50 g of KI in the smallest possible quantity of cold water (50 mL). Add a saturated solution of mercuric chloride (about 22 g in 350 mL of water will be needed) until an excess is indicated by the formation of a precipitate. Then add 200 mL of 5 *N* NaOH and dilute to 1 L. Let settle and draw off the clear liquid.

19.6 *Potassium Sulfate* (K_2SO_4), crystals.

19.7 *Nitrogen Standard*—Dissolve 1.000 g of cystine ($C_6H_{12}N_2O_4S_2$, pre-dried at 110°C for 2 h) in 250 mL of 10 % NaOH and make up to 500 mL with water. This solution contains 0.233 mg N/mL.

19.8 *Sodium Hydroxide Solution* (500 g/L)—Dissolve 500 g of solid sodium hydroxide pellets in 500 mL of water. (*Caution*—This solution becomes very hot.) Cool the solution and dilute to 1 L.

19.9 *Sucrose*—See 11.6.

19.10 *Sulfuric Acid*—See 11.7.

19.11 *Sulfuric Acid* (0.5 *N*)—Prepare and accurately standardize a 0.05 *N* sulfuric acid (H_2SO_4) solution against a primary standard; hydrochloric acid (HCl) of similar concentration may be substituted.

20. Procedure

20.1 Weigh accurately 0.100 g of the analysis sample ground to pass a No. 60 (250-μm) or finer sieve and 0.1 g of the cupric selenite catalyst into a weighing scoop. Carefully transfer to the digestion flask. Care must be taken not to leave sample powder on the neck of the flask.

20.2 Add 1 g of potassium sulfate to the digestion flask. Allow the potassium sulfate to slide down the neck of the flask so that it carries any adhering particles into the bottom of the flask. Gently shake the flask to mix the contents, then add 5.0 mL of concentrated sulfuric acid.

20.3 Place the flask on the digestion stand and heat the contents at a low temperature (about 150°C) to boil off the water. Gradually increase the temperature to about 200°C. If bumping does not occur within 30 min, proceed with the slow-temperature increase to 360°C. Digest the sample at this temperature until the sample is clear and there is a complete absence of grey-black color. Caution must be taken that the digestion temperature does not exceed 400°C because serious loss of nitrogen may occur above this temperature. Minimize the heated area about the flask. The addition of chromic trioxide (0.1 g) to coke samples may shorten their initial digestion time (that is, before the clearing point is reached).

NOTE 4—Incomplete conversion of the nitrogen to ammonium sulfate will lead to low results. Thus, in the digestion step of this procedure, the mixture may become clear but have a light straw color. If digestion is stopped at this point, not all of the nitrogen will be recovered. To secure complete conversion, digestion of the sample must be continued well beyond the straw-colored stage. Anthracite and coke may need a longer digestion time than lower rank coals.

20.4 *Kjeldahl-N Standard and Blank*—Pipet 10 mL of the cystine solution into a Kjeldahl flask, followed by the addition of the same amounts of K_2SO_4 catalyst and concentrated H_2SO_4 described for the sample digestion (20.3). The Kjeldahl blank shall contain 200 mg of sucrose and the same amounts of catalyst, water, and concentrated sulfuric acid as that added to the samples. Proceed with the digestion of the cystine standard and blank in the same manner, and at the same time as the samples.

20.5 *Steam Distillation of the Blank, Standard and Samples*—Remove the Kjeldahl flasks from the digestion stand, allow the digest to cool, and add 10 mL of water to the flask. Mix the contents on a Vortex mixer to thoroughly disintegrate the solidified digest. Without a Vortex mixer, gentle heating and hand mixing of the contents should accomplish the disintegration of the digest solid. Cool the digest.

20.6 *Cleaning the Distillation Apparatus*—A contaminated distillation apparatus shall be thoroughly flushed with dilute HCl (0.1 *N*) for 5 min. This is accomplished by passing steam through the HCl-filled Kjeldahl flask attached to the apparatus. Then, remove the HCl and wash the apparatus with water. Attach a flask filled with water to the apparatus, continue passing steam through the apparatus for an additional 15 min until the distillate is free from NH_4^+ as indicated by Nessler's reagent.

20.7 *Distillation of Blank and Standard Followed by Samples*—Care must be taken not to contaminate the blank and

standard. Therefore, the sequence of successive steam distillation shall be (*1*) the blank, (*2*) the cystine standard, and (*3*) the samples. Place a 50-mL Erlenmeyer flask containing 5 mL of boric acid and 0.1 mL of indicator underneath the condenser tip. Incline the Erlenmeyer flask at a suitable angle and insert tube so that the end is immersed to the maximum depth in the acid. Securely attach the Kjeldahl flask to the distillation apparatus. Pour 20 mL of the 50 % NaOH solution into the funnel while the flask is immersed in ice water to absorb the heat of neutralization. A dark-brown colored precipitate will form. If the solution remains clear, a few millilitres of NaOH must be added until the dark precipitate forms. Close the stopcock and open the steam vent; proceed with the distillation for 4 min (begin the time at the appearance of the first drop of distillate at the tip of the condenser).

NOTE 5—In the distillation step, a too-rapid distillation rate may result in carryover of the alkaline digestion mixture, which will ruin the determination. The addition of a few boiling stones, such as mossy zinc, will help to prevent carryover.

20.8 Rinse the condenser tip into the Erlenmeyer flask with a fine stream of water. Stopper the flask with a rubber stopper. Titrate the distillate with 0.05 N H_2SO_4 until the blue color has a definite tinge of green.

21. Calculation

21.1 The percentage of nitrogen in the analysis sample is calculated as follows:

$$\text{Nitrogen, \%} = \left[\frac{(A - B) \times N \times 14}{W}\right] \times 100 \quad (3)$$

where:

A = millilitres of H_2SO_4 used for titrating the sample,
B = millilitres of H_2SO_4 used for titrating the blank,
N = normality of H_2SO_4, and
W = weight of sample used in milligram.

22. Report

22.1 The results of the nitrogen analysis may be reported on any of a number of bases, differing from each other in the manner by which moisture is treated.

22.2 Use the percentage of moisture in the sample passing a No. 60 (250-μm) sieve to calculate the results of the analysis sample to a dry basis.

22.3 Procedures for converting the valve obtained on the analysis sample to other bases are described in Practices D 3176 and D 3180.

23. Precision and Bias [7]

23.1 The relative precision of this test method, for the determination of nitrogen, covers the concentration range from 0.85 to 1.68 % nitrogen.

23.2 *Repeatability*—The difference in absolute value between two consecutive tests results carried out on the same sample, in the same laboratory, by the same operator, using the same apparatus should not exceed the repeatability interval I(r) more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the repeatability interval, there is reason to question one or both of the test results. The repeatability interval may be determined by use of the following equation:

$$I(r) = 0.31\bar{x} - 0.24 \quad (4)$$

where $\bar{x}$ is the average of the two test results.

NOTE 6—This equation applies to the relative spread of a measurement that is expressed as a percentage and is derived from the statistical evaluation of the round robin results.[7]

23.2.1 *Example*—Duplicate analysis for nitrogen gave values of 1.67 and 1.53 %. The average nitrogen of the duplicate analysis value is 1.60 % and the calculated repeatability interval I(r) is 0.26. The difference between the two values is 0.14 and does not exceed the I(r) of 0.26; therefore, these two values are acceptable at the 95 % confidence level.

23.3 *Reproducibility*—The difference in absolute value of replicate determinations, carried out in different laboratories on representative samples prepared from the same bulk sample after the last stage of reduction, should not exceed the reproducibility interval I(R) more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the reproducibility interval, there is reason to question one or both of the test results. The reproducibility interval may be determined by use of the following equation:

$$I(R) = 0.41\bar{x} - 0.26 \quad (5)$$

where: $\bar{x}$ is the average of the two results (see Note 6).

23.3.1 *Example*—Duplicate analysis for nitrogen in one laboratory gave an average value of 1.60 % and a value of 1.95 % was obtained in a different laboratory. The between laboratory average nitrogen value is 1.78 %, the calculated I(R) interval is 0.47, and the difference between the different laboratory value is 0.35. Since this is less than the I(R), these two values are acceptable at the 95 % confidence level.

23.4 *Bias*—Bias of this stoichiometrically based test method has not been determined at this time. Standard samples of known certified nitrogen content such as NIST SRM coal samples are to be analyzed at the same time as samples.

[7] Supporting data are available from ASTM Headquarters. Request RR:D05-1007.

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