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Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal-and Coke¹

This standard is issued under the fixed designation D 5373; 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

1.1These test methods cover the instrumental determination of carbon, hydrogen, and nitrogen in laboratory samples of coal and eoke prepared in accordance with Test Methods D2013 and D346.

1.2Within the limitations outlined below, these test methods are applicable to either the air-dry or moisture-free laboratory sample, or both.

1.2.1For instrumental systems in which the moisture and waters of hydration in the sample are liberated with (and only with) the oxidation products upon combustion, the analyses can be performed on a test specimen of the air-dry sample (Note 1). Concentrations determined on this air-dried basis represent the total carbon (including that present as carbonate), total hydrogen (including that present as water), and total nitrogen.

<u>1.1 This test method covers the instrumental the determination of carbon in the range of 54.9 % m/m to 84.7 % m/m, hydrogen in the range of 3.25 % m/m to 5.10 % m/m, and nitrogen in the range of 0.57 % m/m to 1.80 % m/m in the analysis sample (7.1) of coal.</u>

NOTE-1-These systems are also satisfactory for determining the subject materials in the moisture-free sample.

1.2.2For systems in which the moisture and hydrates are otherwise liberated, the analysis shall be performed on the moisture-free sample. Values obtained on this basis represent the total carbon, organic hydrogen, and total nitrogen.

1.3These test methods can be used to provide for the requirements specified in Practice D3176 for the ultimate analysis.

1.4The values stated in SI units shall be regarded as the standard.

1.5This 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. Specific precautionary statements are given in 8.3.1. 1—The coals included in the interlaboratory study employed to derive the precision statement for this standard cover ASTM rank lignite A to low volatile bituminous. Additional information concerning the composition of these coals appears in Annex A5.

1.2 The values stated in SI units shall be regarded as the standard.

<u>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.</u>

2. Referenced Documents

2.1 ASTM Standards: ²

D346Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

Đ 2013 Practice for Preparing Coal Samples for Analysis

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

D3174Test Method for Ash in the Analysis Sample of Coal and Coke from Coal

Đ 3176 Practice for Ultimate Analysis of Coal and Coke

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

D4621Guide for Quality Management in an Organization That Samples or Tests Coal and Coke

D 5142Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures <u>Test</u> Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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D 5865 Test Method for Gross Calorific Value of Coal and Coke

2.2 ISO Standard:

ISO 5725-6 Accuracy (Trueness and Precision) of Measurement Methods and Results Part 6: Use in Practice of Accuracy Values³

3. Summary of Test Methods

3.1Carbon, hydrogen, and nitrogen are determined concurrently in a single instrumental procedure. In some systems, the procedure consists of simply weighing a test specimen, placing the test portion into the instrument, and initiating the (subsequently automatic) analytical process. In other systems, the analytical process may be controlled manually to some degree.

3.2The actual process can vary substantially from instrument to instrument because a variety of means can be used to effect the primary requirements of the test methods. These test methods provide for the following: (1) conversion of the subject materials in an oxygen stream in their entirety to earbon dioxide, water vapor, nitrogen oxides, and ash, respectively; and (2) subsequent, quantitative determination of the gases in an appropriate reference gas stream.

3.2.1The conversion of the subject materials to their corresponding gases occurs largely during combustion of the sample at an elevated temperature in an atmosphere of purified oxygen. The gases that are produced include the following:

3.2.1.1Carbon dioxide from the oxidation of organic and elemental carbon and the decomposition of carbonate minerals; 3.2.1.2Hydrogen halides from organic halides (and organic hydrogen, as required);

3.2.1.3Water vapor from the oxidation of (the remaining) organic hydrogen and the liberation of moisture and waters of hydration;

3.2.1.4Nitrogen and nitrogen oxides from the oxidation of organic nitrogen and the decomposition of nitrates; and

3.2.1.5Sulfur oxides from the oxidation of organic sulfur, and the decomposition of sulfide and sulfate minerals.

(1) In some systems, sulfurous and sulfuric acids can also be obtained from a combination of the sulfur oxides and the water vapor.

3.2.2For hydrogen and nitrogen, the required conversion is completed in a two-step process consisting of the following:

3.2.2.1Removal of the halides and sulfur oxides and liberation of the associated hydrogen (as water), by conducting the combustion gases through a series of absorption traps containing appropriate absorbing materials.

3.2.2.2Reduction of the nitrogen oxides to elemental nitrogen (see Note 2) by passing the resultant gases over copper at an elevated temperature. The carbon dioxide, water vapor, and nitrogen may then be determined via one of several satisfactory detection schemes.

Note2-In this process, residual oxygen is also removed.

3.2.3In one configuration, the gases are conducted through a series of thermal conductivity detectors and gas absorbers aligned so that, at the water vapor detector level, the gases pass through the sample side of the detector, a water vapor absorber, and the reference side of the detector. At the carbon dioxide detector level, the gases are then conducted through the sample side of the detector, a carbon dioxide absorber, and the reference side of the detector. Finally, the resultant gases, which contain only nitrogen and the carrier gas, pass through the sample side of the nitrogen detector and are vented. At this detector level, high-purity carrier gas is used as the reference gas. In these ways, the detectors determine the thermal conductivities solely of the specified components.

3.2.4In a second configuration, the carbon dioxide and water vapor are determined by infrared detection, using an aliquot of the combustion gases from which only the halides and sulfur oxides have been removed. These detectors determine the infrared absorption of the pertinent gases at precise wavelength windows so that the absorbances result from only the specified components. In these systems, nitrogen is determined by thermal conductivity, using a second aliquot of the gases, additionally treated to also reduce the nitrogen oxides to nitrogen and to remove the residual oxygen, carbon dioxide, and water vapor.

3.2.5In a third configuration, which is essentially a modified gas chromatographic system, the nitrogen, carbon dioxide, and water vapor in the treated combustion gases are eluted from a chromatographic column and determined (at appropriate retention times) by thermal conductivity detection.

3.3In all cases, the concentrations of carbon, hydrogen, and nitrogen are calculated as functions of the following:

3.3.1Measured instrumental responses,

3.3.2Values for response per unit mass for the elements (established via instrument calibration), and

3.3.3Mass of the sample.

3.4Or to the following: the instrument response is proportional to the gas density, which has been calibrated against a gas density of known concentration.

3.5A capability for performing these computations automatically can be included in the instrumentation used for these test methods. Summary of Test Methods

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.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

🕼 D 5373 – 08

3.1 Carbon, hydrogen and nitrogen are determined concurrently in a single instrumental procedure. The quantitative conversion of the carbon, hydrogen and nitrogen into their corresponding gases $(CO_2, H_2O, and NO_x)$ occurs during combustion of the sample at an elevated temperature in an atmosphere of oxygen. Combustion products which can interfere with the subsequent gas analysis are removed. Oxides of nitrogen (NO_x) are reduced to N₂ before detection. The carbon dioxide, water vapour and elemental nitrogen in the gas stream are determined by appropriate instrumental detection procedures.

4. Significance and Use

4.1Carbon and hydrogen values are used to determine the amount of oxygen (air) required in combustion processes and for the calculations of efficiency of combustion processes.

4.2Carbon and hydrogen determinations are used in material balance calculations on coal conversion processes; also, one or the other is used frequently in correlations of chemical and physical properties, such as yields of products in liquefaction reactivity in gasification and the density and porosity of coal.

4.3Nitrogen data are required to fulfill the requirements of the ultimate analysis, Practice D3176. Also, the data obtained can be used to evaluate the potential formation of nitrogen oxides as a source of atmospheric pollution.

4.4Nitrogen data are used for comparing coals and in research. If the oxygen content of coal is estimated by difference, it is necessary to make a nitrogen determination.

5.Apparatus

5.1Because a variety of instrumental components and configurations can be used satisfactorily for these test methods, no specifications are presented here with respect to overall system design.

5.2Functionally, however, the following requirements are specified for all approved instruments (Note 3):

4.1 Carbon and hydrogen values can be used to determine the amount of oxygen (air) required in combustion processes and for calculation of the efficiency of combustion processes.

4.2 Carbon and hydrogen determinations can be used in calculations including material balance, reactivity and yields of products relevant to coal conversion processes such as gasification and liquefaction.

4.3 Carbon and nitrogen values can be used in material balance calculations employed for emission accounting purposes.

NOTE3—The approval of an instrument with respect to these functions is paramount to these test methods, since such approval tacitly provides approval of both the materials and the procedures used with the system to provide for these functions.

5.2.1The conditions for combustion of the sample shall be such that (for the full range of applicable samples) the subject components shall be converted completely to carbon dioxide, water vapor (except for hydrogen associated with volatile halides), and nitrogen or nitrogen oxides. Generally, instrumental conditions that effect complete combustion include (1) availability of the oxidant, (2) temperature, and (3) time.

5.2.2Representative aliquots of the combustion gases shall then be treated for the following reasons:

5.2.2.1To liberate (as water vapor) hydrogen present as hydrogen halides and sulfur oxyacids; and

5.2.2.2To reduce (to the element) nitrogen present as nitrogen-oxides. 463e-9ad5-0bc041e326d9/astm-d5373-08

(1)The water vapor and nitrogen so obtained shall be included with the materials originally present in these aliquots.

5.2.3Additional treatment of the test specimens (prior to detection) depends on the detection scheme used for the instrument (Note 4).

Nore4-The additional treatments can be provided by the instrumental components used to satisfy 5.2.2.

5.2.3.1For the configuration described in 3.2.3, the halides proper, sulfur oxides, and residual oxygen shall be removed from the single test specimen in which the water vapor, carbon dioxide, and nitrogen are determined sequentially.

5.2.3.2For the configuration described in 3.2.4, the test specimen in which the water vapor and carbon dioxide are determined, only the halides and sulfur oxides shall be removed from the gas stream in which the water vapor and carbon dioxide are determined. For combusted gases in which the nitrogen is determined, the water, carbon dioxide, and residual oxygen shall also be removed.

5.2.3.3For the configuration described in 3.2.5, the halides and sulfur oxides shall be removed from the combusted gases obtained from the single test specimen.

5.2.4The detection system (in its full scope) shall determine the analytical gases individually and without interference. Additionally, for each analyte, either of the following applies:

5.2.4.1The detectors themselves shall provide linear responses that correlate directly to concentration over the full range of possible concentrations from the applicable samples, or

5.2.4.2The system shall include provisions for evaluating nonlinear responses appropriately so that the nonlinear responses can be correlated accurately with these concentrations.

(1)Such provisions can be integral to the instrumentation, or they can be provided by (auxiliary) computation schemes.

5.2.5Finally, except for those systems in which the concentration data are output directly, the instrument shall include an appropriate readout device for the detector responses. 2—The bulk composition of coal changes at a rate that varies from coal to coal during storage. As a result using coal for calibration can yield incorrect estimates of carbon, and hydrogen content in particular.

🕼 D 5373 – 08

5. Apparatus

5.1 Analytical Instrument—An instrument capable of analyzing a test portion of 6 mg or greater. It includes a furnace capable of maintaining a temperature in the range to ensure quantitative recovery of carbon, hydrogen and nitrogen as their corresponding gases $(CO_2, H_2O, and NO_x)$. The combustion gases or a representative aliquot shall be treated to remove, separate out or convert any components that can interfere with the measurement of carbon dioxide, water vapour or nitrogen. The detection system shall include provisions for evaluating the response in a manner that correlates accurately with the carbon dioxide, water vapour and nitrogen present in the treated combustion gases.

5.2 *Balance*—A stand-alone balance or a balance integrated with the instrument, with a resolution of at least 0.1 % relative of the test portion mass.

6. Reagents

6.1 *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.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

- 6.2 Helium, Carrier Gas, as specified by for the instrument by the manufacturer.
- 6.3 Oxygen, as specified by the instrument manufacturer., as specified for the instrument by the manufacturer.

6.4 Additional Reagents, as specified by the instrument manufacturer. This specification refers to the reagents used to provide for the functional requirements cited in 5.2.2-5.2.3.3. These reagents can vary substantially for different instruments; in all cases, however, for systems that are functionally satisfactory (and therefore approved), the reagents recommended by the manufacturer are also tacitly approved. Consequently, these reagents shall be those recommended by the manufacturer., of types and qualities as specified for the instrument by the manufacturer.

6.5 *Calibration Materials*—Calibration materials shall be accompanied by a certificate of analysis stating the purity of the material. If the certificate of analysis does not provide assigned values for carbon, hydrogen or nitrogen use the purity as stated on the certificate to establish carbon, hydrogen and nitrogen from the theoretical values. Store these pure substances in a desiccator under conditions that maintain the compounds in a dry state.

6.6 *Reference Materials*—Certified reference material (CRM) coal(s) prepared by a recognized authority to monitor changes in instrument response that can be affected by constituents not present in the calibration materials (Note 3) and to verify the acceptability of nitrogen results. Coal(s) traceable to a certified reference material (CRM) coal(s) can also be used. Follow instructions on the reference material certificate with respect to storage of the reference material and use of assigned values. Use only those carbon, hydrogen and nitrogen values that have an assigned uncertainty or are traceable to a value with an assigned uncertainty.

NOTE 3—Coal contains mineral constituents as well as acid gas forming constituents that can affect the performance of chemicals used to ensure the consistent and uniform conversion and recovery of carbon, hydrogen and nitrogen.

NOTE 4—The amount of nitrogen in the majority of coal employed for power production purposes is in the range of 0.5 % m/m to 1.8 % m/m. None of the calibrants yields percent nitrogen values within the ranges expected for coal. For this reason reference material (RM) coal is used to check the acceptability of nitrogen results.

7. Preparation of Analysis Sample

7.1The samples shall initially be prepared in accordance with Test Methods D2013 or D346<u>Preparation of Analysis Sample</u> 7.1 The sample shall be the analysis sample prepared to a top size of less than 250 µm (60 mesh). The samples shall be prepared in accordance with Practice D 2013.

⁴ 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.

TABLE 1	Concen	trations	Range	and Lim	its for	Repeatal	oility	and
Reproc	ducibility	for Car	bon, Hy	/drogen,	and N	litrogen i	n Coa	al

EleNament	G <u>F</u> o ncent r <u>mul</u> at ion Range,	<u>C %</u> % <u>m/m</u>	Repeatability <u>H %</u> Limit, +/m	ReproducibilityN % Limit, R/m
			5.5	- 9.6
EDTA	C ₁₀ H ₁₆ N ₂ O ₈	41.1	5.5	9.6
Garbon-	48.6 to90.6	0.64	2.7	8.5 1
Phenylalanine	C ₉ H ₁₁ NO ₂	65.4	6.7	8.5
Hydrogen	0.14 to 5.16	0.16	0.37	10
Acetanilide	C ₈ H ₉ NO	71.1	6.7	10.4
Nitrogen	0.69 to 1.57	-0.11	0.17	_
BBOT	C ₂₆ H ₂₆ N ₂ O ₂ S	72.5	6.1	6.5

∰ D 5373 – 08

7.2If required by characteristics of the instrumental system, reduce the air-dry samples (7.1) typically to pass 75 µm (No. 200 U.S.A. Standard Sieve Series) to obtain test units of the analysis sample in the size range recommended by the instrument manufacturer. If required by characteristics of the instrumental system, as specified in 1.2.2, treat the test specimens in accordance with Test Method D3173 to provide moisture-free materials solely appropriate for these systems. In this and all subsequent sample handling steps, exercise care to minimize changes in moisture content resulting from exposure to the atmosphere.

8. Instrument Preparation

8.1Assemble the instrumental system in accordance with the manufacturer's instructions.

8.2Adjustment of Response of Measurement System — Weigh an appropriate test portion of standard reference material (SRM), ealibrating agent, or reference coal. Analyze the test portion (see 9.1). Repeat this procedure. Adjust instrument response, as recommended by the manufacturer, until the absence of drift is indicated.

8.3

<u>8.1</u> *Instrument Set-Up*—Verify all instrument operating parameters meet the specifications in the instrument operating manual. Verify the condition and quantities of all chemicals currently in use are satisfactory for the number of samples to be analyzed. Prior to any analysis, check for and, if necessary correct any leaks in the combustion system, and the carrier gas system.

<u>8.2 Blank Analyses</u>—Perform blank analyses to establish carbon, hydrogen and nitrogen levels in the combustion and carrier gases. The level of carbon, hydrogen or nitrogen in these gases shall not exceed 1 % relative of the instrument response for the lowest mass of calibration material (8.4) for each analyte. Re-determine blank analyses whenever the carrier gas or oxygen supplies are changed or any chemicals are renewed.

<u>8.3 Conditioning</u>—Select a coal conditioning sample in the range of 75 % m/m carbon dry basis. The moisture content should be less than 3 % m/m. Carry out four determinations on the conditioning sample. Discard the first determination. If the range of the three retained determinations for either carbon, hydrogen or nitrogen values exceed 1.2 r where r is the repeatability of this standard, instrument stability is suspect. In this case take corrective action before proceeding with calibration.

<u>8.4</u> Calibration—Select coal SRMs or other calibrating agents and materials specified by the manufacturer that have certified carbon, hydrogen, and nitrogen values in the range of samples to be analyzed. At least three such SRMs or calibrating agents are recommended for each range of carbon, hydrogen, and nitrogen values to be tested. When possible, two of the SRMs or calibrating agents shall bracket the range of carbon, hydrogen, and nitrogen to be tested, with the third falling within the range.

8.3.1All coal SRMs should be in accordance with 7.1 and shall be supplied by or have traceability to an internationally recognized certifying organization. (Warning—An indicated problem with linearity of the instrument during calibration can result from contamination of the SRM or calibrating agent as the container becomes depleted. It is therefore recommended that the SRM or calibrating agent be discarded when less than five grams remain in the container.)

8.3.2*Calibration Procedure*—Analyze, as samples, portions of an SRM, reference coal, or calibrating agent chosen to represent the level of carbon, hydrogen, and nitrogen in the samples to be tested. If not required by the characteristics of the instrumental system, use the "as-determined" carbon, hydrogen, and nitrogen values for calibration. These values must have been calculated previously from the certified "dry basis" carbon, hydrogen, and nitrogen values and residual moisture determined using either Test Methods D3174 or D5142. Continue analyzing until the results from five consecutive determinations fall within the repeatability interval (see 12.1.1) of these test methods. Calibrate the instrument according to the manufacturer's instructions using these values. Analyze, as samples, two SRMs reference coals or calibrating agents that bracket the range of values to be tested. The results obtained for these samples must be within the stated precision limits of the SRM, reference coal, or calibrating agent, or the calibration procedure must be repeated. Records for all calibrations must be in accordance with Guide D4621—If the instrument has been previously calibrated in accordance with Annex A1, determinations can proceed provided the calibration is verified in accordance with Annex A1.

8.3.3Periodic Calibration Verification and Recalibration —In accordance with Guide D4621, analyze a control sample on a periodic basis. Results obtained for the control sample must be within established limits, or all results obtained since the last successful control check must be rejected and the calibration procedure repeated.

9. Procedure

9.1 Analyze a test specimen of the analysis sample in accordance with the manufacturer's instructions.

9.1 Determination:

<u>9.1.1 Carry out a determination of the moisture content of each analysis sample or each reference material coal, or both, in accordance with Test Method D 3173 or Test Methods D 5142 within 48 h of the determination of carbon, hydrogen and nitrogen on each analysis sample.</u>

9.1.2 Carry out the determination by weighing out a test portion of each analysis sample within a mass range recommended by the manufacturer. Conduct determinations on consecutive test portions of an analysis sample as needed to meet quality control requirements.

9.1.3 Verify the calibration upon completion of all test determinations and as needed to meet quality control requirements according to Annex A2.

9.1.4 Prior to conducting any test determinations, upon completion of all test determinations and as needed to meet quality control requirements conduct determinations on consecutive test portions of a reference material (RM) coal (6.6). Verify the

🕼 D 5373 – 08

acceptability of results for the RM coal in accordance with Annex A3.

NOTE 5—Appendix X2 provides guidance on the selection of the mass of a test portion that will yield for a mass of carbon similar to the mid range mass of the calibration material.

10. Calculation

10.1Calculate the concentrations of carbon, hydrogen, and nitrogen, on the appropriate sample basis, as follows: <u>Calculation</u> 10.1 Record the total carbon, hydrogen and nitrogen as analyzed as a percentage by mass. Report the results on dry basis to the nearest 0.1 % m/m for carbon, 0.01 % m/m for hydrogen and to the nearest 0.01 % m/m for nitrogen.

10.2 Use the following equations for the calculation to dry basis:

10.2.1 For the carbon content:

$$A = \frac{(B \times C)}{D} \times 100 \tag{1}$$

 $Cd = Cad \times 100100 - Mad$

10.2.2 For the nitrogen content:

$$N_d = N_{ad} \times \frac{100}{100 - M_{ad}}$$
(2)

10.2.3 For the hydrogen content:

$$H_d = (H_{ad} - 0.1119 \times M_{ad}) \times \frac{100}{100 - M_{ad}}$$
(3)

d

where:

 $A \underline{d} = \%$ of the analyte, dry basis,

B = detector response for that analyte, as determined (as analyzed) basis,

 $\frac{ad}{C}$ = unit mass per detector response established for the analyte during calibration, and content of carbon, % m/m,

 $D-\underline{N} = \text{mass of test specimen, g. content of nitrogen, <math>\% \text{ m/m}$, 12000 specimen

<u>H</u> = content of hydrogen, % m/m, and

 $\underline{M}_{ad} \equiv \text{moisture content of the sample as analyzed, } \% \text{ m/m.}$

The calculations can be provided automatically by the instrumental system used for these test methods.

11. Report

11.1Report results from the carbon, hydrogen, and nitrogen determinations on any of the several common bases that differ solely with respect to moisture. Procedures for converting the as-determined concentrations to the other bases are specified in Practices $\frac{11.1 \text{ Report}}{13176}$ and $\frac{13180 \text{ Precision}}{13180}$ and $\frac{5}{2}$

11.1 The precision of this test method for the determination of carbon, hydrogen, and nitrogen in coal are shown in Table 2.

12.Precision and Bias

12.1The precision of this test method for the determination of Carbon, Hydrogen, and Nitrogen in coal are shown in Table 1. 12.1.1

<u>11.1.1 Repeatability Limit (r)</u>—The value below which the absolute difference between two test results calculated to a dry basis (Practices D 3180 and D 3176) of separate and consecutive test determinations, carried out on the same sample, in the same laboratory, by the same operator, using the same apparatus on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 %.

12.1.2

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1034.

TABLE 2 Concentrations Range and Limits for Repeatability and Reproducibility for Carbon, Hydrogen, and Nitrogen in Coal							
Element	Concentration Range, <u>% m/m</u>	Repeatability Limit, <u>r % m/m</u>	Reproducibility Limit, <u>R % m/m</u>				
Carbon	54.9 to 84.7	0.45	1.00				
Hydrogen	3.25 to 5.10	0.10	0.25				
Nitrogen	0.57 to 1.80	0.05	0.15				