



# Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal<sup>1</sup>

This standard is issued under the fixed designation D5373; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 1. Scope

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.

NOTE 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.

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:<sup>2</sup>

- D2013 Practice for Preparing Coal Samples for Analysis
- D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke
- D3176 Practice for Ultimate Analysis of Coal and Coke
- D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases
- D5142 Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures
- D5865 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 Values<sup>3</sup>

## 3. Summary of Test Methods

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 ( $\text{CO}_2$ ,  $\text{H}_2\text{O}$ , and  $\text{NO}_x$ ) 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 ( $\text{NO}_x$ ) are reduced to  $\text{N}_2$  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

4.1 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.

NOTE 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.

## 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 ( $\text{CO}_2$ ,  $\text{H}_2\text{O}$ , and  $\text{NO}_x$ ). The combustion

<sup>1</sup> 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.

Current edition approved Feb. 1, 2008. Published February 2008. Originally approved in 1993. Last previous edition approved in 2007 as D5373 – 02 (2007). DOI: 10.1520/D5373-08.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

**TABLE 1 Calibration Materials and Their Theoretical Contents of C, H and N**

Name	Formula	C % m/m	H % m/m	N % m/m
EDTA	C <sub>10</sub> H <sub>16</sub> N <sub>2</sub> O <sub>8</sub>	41.1	5.5	9.6
Phenylalanine	C <sub>9</sub> H <sub>11</sub> NO <sub>2</sub>	65.4	6.7	8.5
Acetanilide	C <sub>8</sub> H <sub>9</sub> NO	71.1	6.7	10.4
BBOT	C <sub>26</sub> H <sub>26</sub> N <sub>2</sub> O <sub>2</sub> S	72.5	6.1	6.5

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.<sup>4</sup> 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 *Carrier Gas*, as specified for the instrument by the manufacturer.

6.3 *Oxygen*, as specified for the instrument by the manufacturer.

6.4 *Additional Reagents*, 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

<sup>4</sup> *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.

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

7.1 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 **D2013**.

## 8. Instrument Preparation

8.1 *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.

8.2 *Blank Analyses*—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.

8.3 *Conditioning*—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.

8.4 *Calibration*—If the instrument has been previously calibrated in accordance with **Annex A1**, determinations can proceed provided the calibration is verified in accordance with **Annex A2**. Otherwise calibrate the instrument as described in **Annex A1**.

## 9. Procedure

### 9.1 Determination:

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 **D3173** or Test Methods **D5142** within 48 h of the determination of carbon, hydrogen and nitrogen on each analysis sample.

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 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

10.1 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:

$$C_d = C_{ad} \times \frac{100}{100 - M_{ad}} \quad (1)$$

10.2.2 For the nitrogen content:

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

10.2.3 For the hydrogen content:

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

where:

$d$  = dry basis,

$ad$  = as determined (as analyzed) basis,

$C$  = content of carbon, % m/m,

$N$  = content of nitrogen, % m/m,

$H$  = content of hydrogen, % m/m, and

$M_{ad}$  = moisture content of the sample as analyzed, % m/m.

**TABLE 2 Concentrations Range and Limits for Repeatability and Reproducibility for Carbon, Hydrogen, and Nitrogen in Coal**

Element	Concentration Range, % m/m	Repeatability Limit, $r$ % m/m	Reproducibility Limit, $R$ % m/m
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

## 11. Precision and Bias<sup>5</sup>

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

11.1.1 *Repeatability Limit (r)*—The value below which the absolute difference between two test results calculated to a dry basis (Practices [D3180](#) and [D3176](#)) 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 %.

11.1.2 *Reproducibility Limit (R)*—The value below which the absolute difference between two test results calculated to a dry basis (Practices [D3180](#) and [D3176](#)), carried out in different laboratories, using samples taken at random from a single quantity of material that is as homogeneous as possible, may be expected to occur with a probability of approximately 95 %.

NOTE 6—Additional information concerning precision statistics appears in [Annex A4](#) as well as the composition of the coals employed in the interlaboratory study (RR:D05-1034) appears in [Annex A5](#).

11.2 *Bias*—Two materials EDTA and phenylalanine distributed from a common source were included in the interlaboratory study as reference pure substances. Each participating laboratory calibrated their instrument using one or more of the pure substances (6.5) obtained from a different source. The reference pure substances were analyzed in two phases of the study conducted one year apart. A comparison of the certificate of analysis values and those obtained in the interlaboratory study for the reference pure substances are shown in [Table 3](#).

NOTE 7—Guidelines for reporting test sample results that do not meet the repeatability requirements of this standard when all calibration verification and reference material check sample acceptance requirements are met are provided in [Appendix X3](#).

## 12. Calculation to Other Bases

12.1 Report 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 [D3176](#) and [D3180](#).

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

**TABLE 3 Comparison of Reference EDTA and Reference Phenylalanine Certificate of Analysis Values with Interlaboratory Values for Carbon, Hydrogen, and Nitrogen**

Reference Pure Substance	Phase	Parameter	Number of Results	Certificate of Analysis Value	Interlaboratory Study Value	Bias	Significant (95 % Confidence)
EDTA	1B	Carbon	34	41.09	41.10	0.01	no
EDTA	1C	Carbon	24	41.09	40.99	-0.10	no
Phenylalanine	1B	Carbon	32	65.43	65.49	0.06	no
Phenylalanine	1C	Carbon	22	65.43	65.37	-0.06	no
EDTA	1B	Hydrogen	33	5.52	5.52	0.00	no
EDTA	1C	Hydrogen	24	5.52	5.54	0.02	no
Phenylalanine	1B	Hydrogen	32	6.71	6.72	0.01	no
Phenylalanine	1C	Hydrogen	24	6.71	6.71	0.00	no
EDTA	1B	Nitrogen	32	9.59	9.55	-0.04	yes
EDTA	1C	Nitrogen	21	9.59	9.54	-0.05	no
Phenylalanine	1B	Nitrogen	32	8.48	8.46	-0.02	no
Phenylalanine	1C	Nitrogen	21	8.48	8.45	-0.03	no

## ANNEXES

### (Mandatory Information)

#### A1. NUMBER OF CALIBRATION POINTS

A1.1 To meet the precision requirements of this method 6 calibration points are required for a linear fit and 8 calibration points are required for a non linear fit. These requirements are summarized for various fits in [Table A1.1](#). A calibration point consists of a determination on a single test portion of calibration material.

NOTE A1.1—The interlaboratory study employed to validate the precision of this test method required laboratories to employ 6 calibration points for a linear fit and 8 for a non linear fit. Additional calibration points can be used.

A1.2 Guidance on the selection of masses of pure substance for calibration is given in [Appendix X1](#).

A1.3 Verify the calibration according to [Annex A2](#).

**TABLE A1.1 Minimum Number of Calibration Points for Common Regressions**

Regression	Number of Calibration Points Required
Linear	6
Quadratic	8
Power	8

#### A2. CALIBRATION VERIFICATION

A2.1 Some instruments allow for calibration drift correction. Verify the acceptability of a drift correction immediately after conducting the drift correction.

A2.2 Verify the acceptability of the calibration by determining the carbon, hydrogen and nitrogen in % m/m of a calibration material (6.5) not used for calibration. [Appendix X1](#) provides guidance on selection of the mass of verification material.

A2.3 [Table A2.1](#) lists the relative percentages the verification determinations shall agree with the values of the carbon, hydrogen and nitrogen from the certificate of analysis.

NOTE A2.1—These limits are from the calibration data supplied by the 14 laboratories that took part in the interlaboratory study (ILS) employed to validate this standard. The calibration data includes results from the pure substances listed in 6.5 and five instrument configurations covering two phases of the ILS conducted 1 year apart. These limits can serve as reasonable performance criteria for calibrations conducted in the same or different laboratories over an extended period of time.

A2.4 If any verification determination does not agree within the limits specified in [Table A2.2](#), conduct three additional verification determinations. Use masses of verification calibrant that yield a mass of carbon within 5 mg of the calibration masses at the lower extreme, mid-point and upper extreme of the calibration (see [Appendix X1](#)). If the additional verification determinations agree with the values from the certificate of analysis within the limits specified in [Table A2.2](#) then it is acceptable to continue with determinations on the analysis samples. Otherwise reject the calibration as well as all determinations back to the last acceptable verification. Check instrument set-up (8.1), conduct blank analyses (8.2) and condition the instrument (8.3). Calibrate the instrument according to [Annex A1](#) before proceeding with analysis.

A2.5 Since the absolute difference between the verification determination and the certificate of analysis value are within the absolute limit for carbon, hydrogen and nitrogen, the laboratory can continue with analysis of test samples without recalibrating the instrument.

**TABLE A2.1 Percent (%) Relative Calibration Acceptance Limits**

Carbon	1.20 %
Hydrogen	2.10 %
Nitrogen	1.80 %

**TABLE A2.2 Example of Calibration Verification with EDTA**

Parameter	Certificate of Analysis % m/m	Relative Limit	Absolute Limit % m/m	Verification Result % m/m	Absolute Difference % m/m
Carbon	41.08	1.20 %	0.49	41.27	0.19
Hydrogen	5.51	2.10 %	0.12	5.59	0.08
Nitrogen	9.60	1.80 %	0.17	9.50	0.11

### A3. ACCEPTANCE OF RESULTS FOR REFERENCE MATERIAL COAL(S)

A3.1 Calculate the as determined carbon, hydrogen and nitrogen results to a dry basis (Section 10). Verify the dry basis results for consecutive carbon, hydrogen and nitrogen determinations agree within the repeatability limit (Section 11).

A3.2 For nitrogen verify each dry basis nitrogen result agrees with the assigned value within 0.11 % m/m.

NOTE A3.1—The 0.11 % m/m acceptance limit is derived from the reproducibility limit for nitrogen (Section 11) in accordance with the section on “Comparison with a reference value for one laboratory” of [ISO 5725-6](#).

A3.3 If any of these acceptance criteria fails, reject all determinations back to the last acceptable reference material determinations. Check instrument set-up (8.1), conduct blank

analyses (8.2) and condition the instrument (8.3). Calibrate the instrument according to [Annex A1](#) before proceeding with determinations.

NOTE A3.2—For carbon and hydrogen it is recommended a record is kept of the dry basis results. Each dry basis carbon result should agree with the dry basis certificate value within 0.7 % m/m. Each dry basis hydrogen result should agree with the dry basis certificate value within 0.2 % m/m. The certifying agency should be notified when more than 7 consecutive dry basis results for carbon and/or hydrogen fall outside these limits. The certifying agency should be provided with the purchase date, in service date and expiry date of the reference material coal. The comparison limits for carbon and hydrogen are derived from the reproducibility limit (Section 11) in accordance with the section on “Comparison with a reference value for one laboratory” of [ISO 5725-6](#).

### A4. PRECISION STATISTICS

A4.1 The precision of this standard characterized by repeatability ( $s_r$ ,  $r$ ) and reproducibility ( $s_R$ ,  $R$ ) has been determined for the materials listed in [Tables A4.1-A4.3](#).

**TABLE A4.1 Repeatability ( $s_r$ ,  $r$ ) and Reproducibility ( $s_R$ ,  $R$ ) for Carbon % m/m**

Coal Source	Rank	Average Carbon	$s_r$	$r$	$s_R$	$R$
Australia	hvAb	72.84	0.17	0.48	0.32	0.90
New Zealand	hvAb	84.68	0.20	0.56	0.34	0.96
Canada	Sub	54.86	0.14	0.39	0.40	1.13
United Kingdom	hvAb	65.55	0.14	0.39	0.36	1.02
Australia	lvb	79.88	0.19	0.54	0.28	0.79
Australia	hvAb	74.31	0.13	0.37	0.30	0.85
Australia	hvAb	77.58	0.16	0.45	0.32	0.90
Australia	Brown Coal	67.53	0.13	0.37	0.43	1.21
United States	hvAb	77.22	0.15	0.42	0.27	0.76

**TABLE A4.2 Repeatability ( $s_r$ ,  $r$ ) and Reproducibility ( $S_R$ ,  $R$ ) for Hydrogen % m/m**

Coal Source	Rank	Average Hydrogen	$s_r$	$r$	$S_R$	$R$
Australia	hvAb	4.59	0.022	0.06	0.114	0.32
New Zealand	hvAb	5.08	0.048	0.14	0.096	0.27
Canada	Sub	3.26	0.032	0.09	0.107	0.30
United Kingdom	hvAb	4.19	0.022	0.06	0.06	0.17
Australia	lvb	4.30	0.023	0.06	0.083	0.23
Australia	hvAb	4.47	0.022	0.06	0.07	0.20
Australia	hvAb	4.98	0.017	0.05	0.068	0.19
Australia	Brown Coal	4.53	0.026	0.07	0.08	0.23
United States	hvAb	4.94	0.017	0.05	0.067	0.19

**TABLE A4.3 Repeatability ( $s_r$ ,  $r$ ) and Reproducibility ( $S_R$ ,  $R$ ) for Nitrogen % m/m**

Coal Source	Rank	Average Nitrogen	$s_r$	$r$	$S_R$	$R$
Australia	hvAb	1.58	0.014	0.04	0.036	0.10
New Zealand	hvAb	1.16	0.018	0.05	0.05	0.14
Canada	Sub	0.70	0.011	0.03	0.045	0.13
United Kingdom	hvAb	1.40	0.01	0.03	0.041	0.12
Australia	lvb	1.76	0.021	0.06	0.05	0.14
Australia	hvAb	1.67	0.013	0.04	0.047	0.13
Australia	hvAb	1.75	0.015	0.04	0.058	0.16
Australia	Brown Coal	0.57	0.017	0.05	0.051	0.14
United States	hvAb	1.46	0.012	0.03	0.052	0.15

## A5. COMPOSITION OF COALS

A5.1 The composition of the coals employed to establish the precision of this standard are listed in [Table A5.1](#). This table lists those parameters known to affect the determination of carbon, hydrogen and nitrogen in coal.

**TABLE A5.1 Composition of Interlaboratory Study Coals**

Country	Rank	Moisture % mm	Ash % m/m Dry Basis	Sulfur % m/m Dry Basis	Chlorine $\mu\text{g/g}$ Dry Basis
Australia	hvAb	3.27	10.71	0.52	225
New Zealand	hvAb	0.96	1.76	1.50	830
Canada	Sub	10.24	24.23	0.25	37
United Kingdom	hvAb	2.97	19.18	1.90	4877
Australia	lvb	1.40	9.81	0.59	530
Australia	hvAb	3.20	11.05	0.56	410
Australia	hvAb	3.10	6.50	0.45	210
Australia	Brown Coal	10.20	1.22	0.31	1220
United States	hvAb	1.61	8.51	0.92	1479

**APPENDIXES**

**(Nonmandatory Information)**

**X1. PURE SUBSTANCE MASSES FOR CALIBRATION AND CALIBRATION VERIFICATION**

X1.1 This appendix describes a procedure that can be used to calculate the range of calibrant masses from the expected range of carbon, % m/m and hydrogen, % m/m in the analysis sample (7.1).

NOTE X1.1—The hydrogen, % m/m determined on a test portion of the analysis sample includes the hydrogen in the residual moisture of the test portion.

NOTE X1.2—The amount of hydrogen in majority of coal employed for power production purposes is in the range of 4.5 % m/m to 6.1 % m/m when the hydrogen in the coal moisture is taken into account. This information is provided to assist in the selection of calibration and calibration verification materials.

NOTE X1.3—The amount of nitrogen in 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 calibration materials (6.5) yield percent nitrogen values within the ranges expected for coal.

X1.2 A laboratory intends to calibrate an instrument to cover carbon in the range of 55 % m/m to 85 % m/m and hydrogen in the range 4.5 % m/m to 5.3 % m/m. The laboratory uses a test portion mass of  $75 \pm 5$  mg.

X1.3 Calculate to the nearest milligram the mass of calibrant required for the lower and upper limits of carbon % m/m as well as the lower and upper limits of hydrogen % m/m using the following equation.

$$Mc = (Mt \times Av) / C_{AC} \quad (X1.1)$$

where:

- $Mc$  = mass of test portion in milligrams,
- $C_{AC}$  = mid range mass of the calibration material,
- $Mt$  = as determined heating value of the analysis sample, and
- $Av$  = heating value of the calibration material.

X1.4 The mass of EDTA in milligrams with a certificate of analysis value of 41.02 % m/m carbon required to produce the same mass of carbon as a 75 mg test portion containing 55 % m/m carbon is given by:

$$Mc = (75 \times 55) / 41.02 = 101 \text{ mg to the nearest mg}$$

X1.5 The mass of EDTA with a certificate of analysis value of 41.02 % m/m carbon required to produce the same mass of carbon as a 75 mg test portion containing 85 % m/m carbon is given by:

$$Mc = (75 \times 85) / 41.02 = 155 \text{ mg to the nearest mg}$$

X1.6 The mass of EDTA with a certificate of analysis value of 5.56 % m/m hydrogen required to produce the same mass of hydrogen as a 75 mg test portion containing 4.5 % m/m hydrogen is given by:

$$Mc = (75 \times 4.5) / 5.56 = 60 \text{ mg to the nearest mg}$$

X1.7 The mass of EDTA with a certificate of analysis value of 5.56 % m/m hydrogen required to produce the same mass of hydrogen as a 75 mg test portion containing 5.3 % m/m hydrogen is given by:

$$Mc = (75 \times 5.3) / 5.56 = 71 \text{ mg to the nearest mg}$$

X1.8 Pick the smallest calculated calibration mass. In this case it is 60 mg.

X1.9 Pick the largest calculated calibration mass. In this case it is 155 mg.

X1.10 Calculate the difference. In this case it is 95 mg.

X1.11 The mid range calibration mass of EDTA is  $(60+155)/2 = 108$  mg to the nearest mg.

X1.12 The calibrant mass interval for a linear fit using 6 points is given by  $95/5$  or 19 mg the nearest mg.

X1.13 The calibrant mass interval for a non linear fit using 8 points is given by  $95/7$  mg or 14 mg to the nearest mg.

X1.14 **Table X1.1** summarizes the masses EDTA for the range of 55 % m/m to 85 % m/m carbon and 4.5 % m/m to 5.3 % m/m hydrogen for a test portion of  $75 \pm 5$  mg.

X1.15 Calculate the mass of the material used for calibration verification that will yield the same mass of carbon as a mass of the material within the range of the calibration using the following equation.

$$Mv = (Mc \times C_{AC}) / C_{AV} \quad (X1.2)$$

where:

- $Mv$  = mass of verification material in milligrams,
- $Mc$  = mass of calibrant in milligrams,
- $C_{AC}$  = value for carbon or hydrogen from the certificate of analysis of the calibrant, % m/m, and
- $C_{AV}$  = value for carbon or hydrogen from the certificate of analysis of the verification material, % m/m.

X1.16 The laboratory uses phenylalanine as the calibration verification material.

X1.17 The mass of phenylalanine with a certificate of analysis of 65.38 % m/m carbon that will yield the same mass of carbon as the mid range mass of EDTA is given by:

$$(108 \times 41.02) / 65.38 = 68 \text{ mg to the nearest mg}$$

**TABLE X1.1 Masses of EDTA in mg for Calibration**

Linear	60	79	98	117	136	155		
Non Linear	60	74	88	102	116	130	144	158

X1.18 The mass of phenylalanine with a certificate of analysis of 65.38 % m/m carbon that will yield the same mass of carbon as the smallest calculated calibration mass of EDTA is given by:

$$(60 \times 41.02) / 65.38 = 38 \text{ mg to the nearest mg}$$

X1.19 The mass of phenylalanine with a certificate of

analysis of 65.38 % m/m carbon that will yield the same mass of carbon as the largest calculated calibration mass of EDTA is given by:

$$(155 \times 41.02) / 65.38 = 98 \text{ mg to the nearest mg}$$

## X2. OPTIMUM TEST SAMPLE MASS FOR THE DETERMINATION OF TOTAL CARBON

X2.1 This appendix describes a procedure that can improve the reliability of carbon results. This can be important in cases where carbon results are employed for carbon accounting purposes.

X2.2 The minimum error of a result derived from a regression occurs near the mid range of the calibration. The mass of the test portion that produces a mass of carbon similar to the mid range mass of the calibration material can be estimated from the as determined heating value (HV) of the analysis sample, determined according to Test Method **D5865** and the heating value of the calibration material.

X2.3 The test portion mass equivalent to the mid range mass of calibration material is given by:

$$M_t = M_{CM} \times (H_{VC} / H_{VS}) \quad (X2.1)$$

where:

- $M_t$  = mass of test portion in milligrams,
- $M_{CM}$  = mid range mass of the calibration material,
- $H_{VS}$  = as determined heating value of the analysis sample, and
- $H_{VC}$  = heating value of the calibration material.

**TABLE X2.1 Heating Value (HV) of Calibrants**

Calibrant	MJ/kg	Cal/gm	Btu/lb
EDTA	16.91	4038	7268
Phenylalanine	26.96	6439	11590
Acetanilide	31.34	7485	13473
BBOT	29.84	7127	12828

X2.4 A laboratory employs EDTA to calibrate an instrument.

X2.5 The test portion mass is  $75 \pm 5$  mg.

X2.6 The mid range calibration mass is 108 mg.

X2.7 Two analysis samples are to be tested for carbon, hydrogen and nitrogen.

X2.8 The as determined heating value of analysis sample A is 23.88 MJ/kg.

X2.9 The test portion mass for analysis sample A that would yield a mass of carbon similar to the mid range mass of calibration material is given by:

$$M_t = 108 \times (16.91/23.88) = 76 \text{ mg to the nearest mg}$$

X2.10 The as determined heating value of analysis sample B is 34.03 MJ/kg.

X2.11 The test portion mass for analysis sample B that would yield a mass of carbon similar to the mid range mass of calibration material is given by:

$$M_t = 108 \times (16.91/34.03) = 54 \text{ mg to the nearest mg}$$

## X3. CHECKING TEST RESULTS AND DETERMINING THE FINAL QUOTED RESULT

X3.1 The procedures described in this appendix are taken from the section on “Methods for checking the acceptability of test results and determining the final quoted result,” of **ISO 5725-6**.

X3.2 *Checking the Acceptability of Results Obtained Under Repeatability Conditions:*

X3.2.1 For analysis samples that do not meet the repeatability limit (Section 11) of this standard conduct analysis of two additional consecutive test portions on the same day as the initial runs. Calculate the as determined carbon, hydrogen and nitrogen results to a dry basis (Section 10). Calculate the range of all four carbon, hydrogen and nitrogen values.

X3.2.2 If the range is less than or equal to 1.3 times the repeatability limit report the mean of the four determinations. Indicate the value reported is a mean value. If the range is greater than  $1.3r$ , where  $r$  is the repeatability limit (Section 11) of this standard, report the median of the four determinations. Indicate the value reported is a median value.

X3.3 *Checking the Acceptability of Results Obtained Under Reproducibility Conditions:*

X3.3.1 The cause for discrepancy between the final quoted results from two different laboratories that exceeds the reproducibility limit (Section 11) of this standard can be resolved by exchanging analysis samples and/or reference materials (6.6) as well as calibrant(s) (6.5). Where such an exchange is not



practical each laboratory should obtain test results on a common sample preferably of known composition. Where use of a common material of known composition is not possible agreement should be reached between the two laboratories to refer to a third laboratory.

*ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.*

*This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or [service@astm.org](mailto:service@astm.org) (e-mail); or through the ASTM website ([www.astm.org](http://www.astm.org)). Permission rights to photocopy the standard may also be secured from the ASTM website ([www.astm.org/COPYRIGHT/](http://www.astm.org/COPYRIGHT/)).*