Designation: D 5291 – 02 (Reapproved 2007)

An American National Standard

Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants[1]

This standard is issued under the fixed designation D 5291; 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 instrumental determination of carbon, hydrogen, and nitrogen in laboratory samples of petroleum products and lubricants. Values obtained represent the total carbon, the total hydrogen, and the total nitrogen.

1.2 These test methods are applicable to samples such as crude oils, fuel oils, additives, and residues for carbon and hydrogen and nitrogen analysis. These test methods were tested in the concentration range of at least 75 to 87 mass % for carbon, at least 9 to 16 mass % for hydrogen, and <0.1 to 2 mass % for nitrogen.

1.3 The nitrogen test method is not applicable to light materials or those containing <0.75 mass % nitrogen, or both, such as gasoline, jet fuel, naphtha, diesel fuel, or chemical solvents.

1.4 These test methods are not recommended for the analysis of volatile materials such as gasoline, gasoline-oxygenate blends, or gasoline type aviation turbine fuels.

1.5 The results of these tests can be expressed as mass % carbon, hydrogen or nitrogen.

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

1.7 *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:* [2]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance

3. Summary of Test Methods

3.1 In these test methods, carbon, hydrogen, and nitrogen are determined concurrently in a single instrumental procedure. With some systems, the procedure consists of simply weighing a portion of the sample, placing the portion in the instrument, and initiating the (subsequently automatic) analytical process. In other systems, the analytical process, to some degree, is manually controlled.

3.2 The actual process can vary substantially from instrument to instrument, since a variety of means can be utilized to effect the primary requirements of the test methods. All satisfactory processes provide for the following:

3.2.1 The conversion of the subject materials (in their entirety) to carbon dioxide, water vapor, and elemental nitrogen, respectively, and

3.2.2 The subsequent, quantitative determination of these gases in an appropriate gas stream.

3.3 The conversion of the subject materials to their corresponding gases takes place largely during combustion of the sample at an elevated temperature in an atmosphere of purified oxygen. Here, a variety of gaseous materials are produced, including the following:

3.3.1 Carbon dioxide from the oxidation of organic and elemental carbon,

3.3.2 Hydrogen halides from organic halides (and organic hydrogen, as required),

3.3.3 Water vapor from the oxidation of (the remaining) organic hydrogen and the liberation of moisture,

3.3.4 Nitrogen and nitrogen oxides from the oxidation of organic nitrogen, and

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[1] These test methods are under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and are the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

Current edition approved May 1, 2007. Published July 2007. Originally approved in 1992. Last previous edition approved in 2002 as D 5291–02.

[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.

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

3.3.5 Sulfur oxides from the oxidation of organic sulfur. In some systems, sulfurous and sulfuric acids can also be obtained from a combination of the sulfur oxides and the water vapor.

3.4 There are several accepted ways of isolating the desired gaseous products and quantitatively determining them. These are as follows:

3.4.1 *Test Method A*[3,4]—From the combustion product gas stream, oxides of sulfur are removed with calcium oxide in the secondary combustion zone. A portion of the remaining mixed gases is carried by helium gas over a hot copper train to remove oxygen, and reduce NO_x to N_2, over NaOH to remove CO_2, and over magnesium perchlorate to remove H_2O. The remaining elemental nitrogen is measured by the thermal conductivity cell. Simultaneously, but separately from the nitrogen measurement, the carbon and hydrogen selective infrared cells measure the CO_2 and H_2O levels.

3.4.2 *Test Method B*[4,5]— From the combustion product gas stream (which is cleaned from sulfur oxides, excess oxygen, etc. as in 3.4.1), the remaining CO_2, water vapor, and N_2 are flushed into a mixing chamber and are thoroughly homogenized at a precise volume, temperature, and pressure. After homogenization, the chamber is depressurized to allow the gases to pass through a heated column, where the gases separate as a function of selective retention times. The separation occurs in a stepwise steady-state manner for nitrogen, carbon dioxide, and water.

3.4.3 *Test Method C*[4,6]—The combustion product gas stream, after full oxidation of component gases, is passed over heated copper to remove excess oxygen and reduce NO_x to N_2 gas. The gases are then passed through a heated chromatographic column to separate and elute N_2, CO_2, and H_2O in that order. The individual eluted gases are measured by a thermal conductivity detector.

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

3.5.1 The measured instrumental responses,

3.5.2 The values for response per unit mass for the elements (established via instrument calibration), and

3.5.3 The mass of the sample.

3.6 A capability for performing these computations automatically can be included in the instrumentation utilized for these test methods.

4. Significance and Use

4.1 This is the first ASTM standard covering the simultaneous determination of carbon, hydrogen, and nitrogen in petroleum products and lubricants.

4.2 Carbon, hydrogen, and particularly nitrogen analyses are useful in determining the complex nature of sample types covered by this test method. The CHN results can be used to estimate the processing and refining potentials and yields in the petrochemical industry.

4.3 The concentration of nitrogen is a measure of the presence of nitrogen containing additives. Knowledge of its concentration can be used to predict performance. Some petroleum products also contain naturally occurring nitrogen. Knowledge of hydrogen content in samples is helpful in addressing their performance characteristics. Hydrogen to carbon ratio is useful to assess the performance of upgrading processes.

5. Apparatus

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

5.2 Functionally, however, the following are specified for all instruments:

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

5.2.2 Representative aliquots of the combustion gases must then be treated:

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

5.2.2.2 To reduce (to the element) nitrogen present as nitrogen oxides.

5.2.3 The water vapor and nitrogen so obtained must be included with the materials originally present in these aliquots.

5.2.4 Additional treatment of the aliquots (prior to detection) depends on the detection scheme utilized for the instrument (see Note 1).

NOTE 1—These additional treatments can be provided by the instrumental components utilized to satisfy 5.2.2.

5.2.5 The detection system (in its full scope) must determine the analytical gases individually and without interference. Additionally, for each analyte, either:

5.2.5.1 The detectors must provide linear responses with respect to concentration over the full range of possible concentrations from the applicable samples, or

5.2.5.2 The system must include provisions for appropriately evaluating nonlinear responses so that they can be accurately correlated with these concentrations.

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

5.2.7 Lastly, except for those systems where the concentration data are output directly, the instrument must include an appropriate readout device for the detector responses.

5.3 Additionally consumables needed for the analyses include:

5.3.1 *Tin Capsules*, large and small,

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[3] The sole source of supply of the Leco CHN-600 instrument known to the committee at this time is Leco Corporation, 3000 Lakeview Ave., St. Joseph, MI 49085.

[4] If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

[5] The sole source of supply of the Perkin Elmer 240C, 2400 series and CEC 240XA and 440 instruments known to the committee at this time is Perkin Elmer Corporation, Main Ave., Norwalk, CT 06856.

[6] The sole source of supply of the Carlo Erba 1106, 1108, and 1500 instruments known to the committee at this time is Carlo Erba Strumentazione, Strada Rivoltana, 20090 Rodano, Milan, Italy.

TABLE 1 Calibration Standards for CHN Instrumental Analysis[A,B]

Compound	Molecular Formula	Carbon, Mass%	Hydrogen Mass %	Nitrogen Mass %
Acetanilide	C_8H_9NO	71.09	6.71	10.36
Atropine	$C_{17}H_{23}NO_3$	70.56	8.01	4.84
Benzoic acid	$C_7H_6O_2$	68.84	4.95	. . .
Cyclohexanone-2,4-dinitrophenylhydrazone	$C_{12}H_{14}N_4O_4$	51.79	5.07	20.14
Cystine	$C_6H_{12}N_2O_4S_2$	29.99	5.03	11.66
Diphenyl	$C_{12}H_{10}$	93.46	6.54	. .
EDTA	$C_{10}H_{16}N_2O_8$	41.10	5.52	9.59
Imidazol	$C_3H_4N_2$	52.92	5.92	41.15
Nicotinic acid	$C_6H_5NO_2$	58.53	4.09	11.38
Stearic acid	$C_{18}H_{36}O_2$	75.99	12.76	. .
Succinamide	$C_4H_8N_2O_2$	41.37	6.94	24.13
Sucrose	$C_{12}H_{22}O_{11}$	42.10	6.48	. .
Sulphanilamide	$C_6H_8N_2O_2S$	41.84	4.68	16.27
Triethanol amine	$C_6H_{15}NO_3$	48.30	10.13	9.39

[A] The Merck Index, 10th Edition, Merck and Company, Inc., Rahway, New Jersey, 1983.

[B] Many of these compounds can be obtained from the National Institute of Standards and Technology as well as other commercial chemical manufacturers, such as Aldrich, Alfa, Kodak and others. See 6.1 for the purity of these reagents.

5.3.2 *Ceramic Crucibles*,
5.3.3 *Copper Capsules*,
5.3.4 *Tin Plugs*,
5.3.5 *Tin Boats*,
5.3.6 *Copper Plugs*,
5.3.7 *Aluminum Capsules*,
5.3.8 *Combustion Tubes*,
5.3.9 *Adsorption Tubes*,
5.3.10 *Nickel Capsules*, and
5.3.11 *Reduction Tubes*.

5.4 *Analytical Balance*, capable of weighing to the nearest 0.00001 g.

5.5 *Syringes or Pipettes*, to transfer the test specimens to capsules.

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.[7] 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 *Calibration Standards*—Table 1 lists the pure organic compounds most commonly used to calibrate the instruments operated according to 3.4.1-3.4.3; other suitable pure compounds can also be used.

6.3 *Carrier and Combustion Gases*:

6.3.1 *Oxygen*, high purity (99.998 %),
6.3.2 *Helium*, high purity (99.995 %),
6.3.3 *Compressed Air*, *Nitrogen*, *or Argon*, for operating pneumatic valves, if needed, and
6.3.4 *Carbon Dioxide*.

6.4 *Additional Reagents (as Specified by the Instrument Manufacturer)*—This specification covers the reagents utilized to provide for the functional requirements cited in 5.2.2 and 5.2.3. These reagents can vary substantially for different instruments. Consequently, these reagents shall be those recommended by the manufacturer. Specifically, these reagents will be for:

6.4.1 *Test Method A*[3,4]:
6.4.1.1 *Sodium Hydroxide Coated Silica*,
6.4.1.2 *Quartz Wool*,
6.4.1.3 *Magnesium Perchlorate*,
6.4.1.4 *Copper Turnings*,
6.4.1.5 *Coated Calcium Oxide (Furnace Reagent)*,
6.4.1.6 *Nitrogen Catalyst*, and
6.4.1.7 *Magnesium Oxide,* [4,8] for liquids.

6.4.2 *Test Method B*[4,5]:
6.4.2.1 *EA 1000 Reagent,* [4,9]
6.4.2.2 *Silver Tungstate on MgO*,
6.4.2.3 *Silver Vanadate*,
6.4.2.4 *Quartz Wool*,
6.4.2.5 *Silver Gauze*,
6.4.2.6 *Copper Oxide,* [10]
6.4.2.7 *Tungstic Oxide*,
6.4.2.8 *Cobalt Oxide*,
6.4.2.9 *Copper Powder*,
6.4.2.10 *Sodium Hydroxide Coated Silica*,
6.4.2.11 *Alumina*,
6.4.2.12 *Magnesium Perchlorate*, and
6.4.2.13 *Platinum Gauze*.

6.4.3 *Test Method C*[4,6]:
6.4.3.1 *Quartz Wool*,
6.4.3.2 *Chromic Oxide* (oxidation catalyst),
6.4.3.3 *Silver Coated Cobalt Oxide*,
6.4.3.4 *Reduced Copper* (reduction catalyst),
6.4.3.5 *Magnesium Perchlorate*,
6.4.3.6 *Molecular Sieve*, 3A 1⁄16 in. (1.6 mm),
6.4.3.7 *Sodium Hydroxide Coated Silica*,
6.4.3.8 Chromosorb, (Absorber,[11] for liquid samples; calcined silica), and
6.4.3.9 *Copper Grains*.

6.5 *Quality Control (QC) Samples*, preferably are portions of one or more liquid petroleum materials that are stable and representative of the samples of interest. These QC samples

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[7] *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[8] The sole source of supply of Com-aid, a registered trademark of Leco, known to the committee at this time is Leco Corporation, 3000 Lakeview Ave., St Joseph, MI 49085.

[9] The sole source of supply of the EA 1000 Reagent, a registered trademark of Perkin Elmer, known to the committee at this time is Perkin Elmer Corporation, Main Ave., Norwalk, CT 06856.

[10] The sole source of supply of Cuprox, a registered trademark of Perkin Elmer, known to the committee at this time is Perkin Elmer Corporation, Main Ave., Norwalk, CT 06856.

[11] The sole source of supply of Chromosorb, a registered trademark of Carlo Erba Strumentazione, known to the committee at this time is Carlo Erba Strumentazione, Strada Rivoltana, 20090 Rodano, Milan, Italy.

can be used to check the validity of the testing process as described in Section 12.

7. Sampling, Test Specimens, and Test Units

7.1 *Laboratory Sample*—Take a representative sample as specified in Practices D 4057 or D 4177.

7.2 *Test Specimen*—Take an aliquot from the laboratory sample for analysis as follows:

7.2.1 *Preparation*—Warm viscous samples until they are fluid, and shake for 5 s.

7.2.2 *Transfer*—Use any convenient, clean syringe or pipet to transfer test specimens to the capsules as described in Section 9.

8. Preparation of Apparatus

8.1 Prepare the instrumental system (in its entirety) in strict accordance to the manufacturer's instructions.

8.2 Calibrate the system using acetanilide or other suitable calibration standard mentioned in Table 1, using the standardization procedure specified by the manufacturer. This procedure, with respect to the actual analytical process involved, must not differ from that specified for the samples.

8.3 *Tubes/Columns Preparation (see* Note 2*)*—Clean all quartz and glass parts prior to use with soap and water followed by acetone, and dry fingerprints must be removed with a grease solvent such as acetone, prior to insertion of the tubes into the furnace. Handle the tubes using appropriate gloves, such as lint-free cotton gloves, that will not leave fingerprints.

NOTE 2—All combustion tubes and absorption tubes need to be periodically replaced after 50 to 300 sample runs. The exact intervals of change should be determined as recommended by the manufacturer.

8.3.1 *Test Method A*[3,4]:

8.3.1.1 *Combustion Tube*—Pack 5 cm of quartz wool in the bottom of the tube on the primary (inlet) side. Set a ceramic crucible on top of the quartz wool on the primary side. Pack 3.1 cm of quartz wool in the bottom of the tube on the secondary (outlet) side. Fill 6.3 cm of furnace reagent on top of the quartz wool. Pack 11.9 cm of quartz wool on top of the furnace reagent.

8.3.1.2 *Reduction Tube*—Insert a small copper plug in the bottom. Fill up to 8.8 cm of *N*-catalyst on top of the copper plug. Add 13.8 cm of copper turnings on top of the *N*-catalyst.

8.3.2 *Test Method B*[4,5]:

8.3.2.1 *Combustion Tube*—Precondition EA 1000[4,9] and silver tungstate on magnesium oxide by preheating it at 900°C for 10 to 30 min. Roll a strip of silver gauze to fit into the combustion tube. Clean the rolled gauze by either ultrasonically washing it in detergent and water, and drying it, or by passing it rapidly over a flame several times until the smoke disappears. Slide 2.5 cm of quartz wool into the tube from the outlet so that the end of the wool meets with the indentations in the tube. Add 5 cm of EA 1000[4,9] reagent. Top with a small wad of quartz wool. Add 5 cm of silver tungstate on magnesium oxide. Top with a small wad of quartz wool. Add 2.5 cm of silver vanadate. Top with a small wad of quartz wool. Slide a roll of silver gauze into the combustion tube, so that there is about 1.2 cm empty space left in the tube. Insert 1.2 cm of quartz wool into the inlet end of the combustion tube, and another into the vial receptacle, and place the receptacle into the combustion tube.

8.3.2.2 *Reduction Tube*—Insert a small wad of quartz wool into the inlet end of the tube to where the tube widens. Then insert a conditioned roll of silver gauze. Fill the tube from the outlet end. Fill with copper powder, occasionally tapping till the copper column is 23.8 cm deep. Insert a small wad of quartz wool, then add 1.2 cm of cuprox (copper oxide), and add another small wad of quartz wool. Insert the copper plug into the outlet end of the reduction tube.

8.3.3 *Test Method C*[4,6]:

8.3.3.1 *Combustion Reactor*—First place a 2 mm plug of quartz wool in the bottom of the tube. Fill the tube with 50 mm of silver coated cobalt oxide. Insert a 10 mm thick plug of quartz wool. Add 120 mm of chromic oxide. Inset a 10 mm thick plug of quartz wool. Slide the combustion tube into the combustion furnace and secure with the O-ring fittings.

8.3.3.2 *Reduction Reactor*—First place a 5 mm plug of quartz wool in the bottom of the tube. Fill the tube with reduced copper. Insert a 10 mm thick plug of quartz wool. Slide the reduction tube into the reduction furnace and secure with the O-ring fittings.

8.3.3.3 *Water Trap*—First place a 10 mm plug of quartz wool in the bottom of the tube. Fill the tube with 3A 1⁄16 in. (1.6 mm) molecular sieve or magnesium perchlorate.[4,9] Insert a 10 mm thick plug of quartz wool. Secure the tube in the water trap bracket.

8.3.3.4 *Carbon Dioxide Trap*—First place a 10 mm plug of quartz wool in the bottom of the tube. Fill the tube with 50 mm of magnesium perchlorate. Add 130 mm of sodium hydroxide coated silica.[4,8] Add 50 mm of magnesium perchlorate. Insert a 10 mm thick plug of quartz wool. Secure the tube in the carbon dioxide trap bracket.

9. Procedure

9.1 *Test Method A*[3,4]:

9.1.1 Accurately to within ±1 mg weigh out about 50 to 200 mg of homogenous test specimen taken according to Practices D 4057 or D 4177, in a capsule. Crimp the capsule with a forceps.

9.1.1.1 Weigh the solid samples in tin capsules.

9.1.1.2 Weigh the liquid samples in copper capsules and saturate with an absorbent (magnesium oxide[10]) within the copper capsules. Seal the copper capsules with tin plugs. These precautions will induce a slower combustion to prevent any back flashes with the light samples, or incomplete sample combustion.

9.1.2 The following typical settings may be used.

9.1.2.1 *Temperatures*:

Combustion furnace; primary zone	950°C
Combustion furnace; secondary zone	950°C
Catalyst heater	750°C
Oven chamber	53°C

9.1.2.2 Oxygen and helium carrier gas pressures should be 40 psi each.

9.1.2.3 Typical settings for gas flows are:

Helium	400 cm^3/min (normal flow) 70 cm^3/min (conservation flow)
Oxygen	7 dm^3/min
Air	6 dm^3/min

9.1.3 To start the sequence of analysis, run two to four blank capsules, followed by five calibration standards. The results must agree within ±10 % for blanks, and ±1 % of the theoretical value for calibration standards.

9.1.4 Hold all the results in the instrument memory, and recall the best values for the calibration. Enter these results and the known calibrant concentrations into the microprocessor to generate a one point calibration curve.

9.1.5 Analyze the calibration standard again to check the new curve. The results should differ by less than 1 %, or repeat the calibration.

9.1.6 Combust the encapsulated sample in a manner similar to the calibrant. Depending on the sample matrices, set varying oxygen flow rates and combustion times to overcome incomplete combustion.

9.2 *Test Method B*[4,5]:

9.2.1 Accurately to within ±0.02 mg weigh out 2 to 4 mg of a homogeneous test specimen taken according to Practices D 4057 or D 4177, into the vial. Pinch the center of the vial with a forceps and fold it in half. Flatten the vial with the tweezers, and fold the vial in thirds.

9.2.2 The following typical settings may be used.

Combustion temperature	975°C
Reduction temperature	640°C
Detector oven temperature	80 to 84°C
Helium	137.9 kPa
Oxygen	110.3 kPa
Air, nitrogen, or argon	413.7 kPa

9.2.3 Run two to four blanks through the system. Run a conditioning sample followed by another blank. Repeat this sequence.

9.2.4 Run a calibration standard and obtain a response factor, (K), that is calculated as: detector counts for standard/mass of nitrogen, carbon, or hydrogen in the standard.

9.2.5 If the response factors obtained are within manufacturer's specifications, run the samples.

9.3 *Test Method C*[4,6]:

9.3.1 Accurately to within ±0.02 mg weigh out about 5 mg of homogeneous test specimen taken according to Practices D 4057 or D 4177, in a tin capsule. Crimp the capsule closed with a forceps.

9.3.2 For liquid samples, add about 30 mg of Chromosorb[11] to the tin capsule after weighing the sample.

9.3.3 The following typical settings may be used.

Combustion temperature	1020°C
Reduction temperature	650°C
Detector oven temperature	60 to 100°C
Detector filament temperature	190°C
Analysis cycle	420 s
Autosample inject	Start 20 s
Autosample return	Stop 70 s
Oxygen inject time	70 s
Integration window delay	2 s

9.3.4 Typical settings for gas flows are:

	Line Pressure	Flow Rate
Oxygen	100 kPa	20 cm^3/min
Helium	200 kPa	Reference detector—40 cm^3/min Measure detector—80 cm^3/min Sample purge—60 cm^3/min
Air	500 kPa	3.5 kg/cm^2

9.3.5 To start the sequence of analysis, run one unweighed sample to determine retention times, followed by one to two blank capsules, two to three calibration samples for a response factor calculation or three to six calibration samples for a linear regression calculation, then unknown samples.

9.3.6 Randomly insert standards during the analysis of samples to check and update the calibration. The placement and frequency of standards is dependent on sample type and change in analytical conditions, however, a good rule of thumb is one standard for every ten samples (see Note 3).

NOTE 3—Since these test methods were originally developed in 1992, all three instrument manufacturers included here have newer models available, which follow the same original technical principles but differ in instrumental operation parameters described here. Hence, for newer instrument models, follow the manufacturers' instructions for operating the instruments. The precision statements in Section 13 refer to the original instrument models used in the 1992 round-robin study.

10. Calculation

10.1 Calculate the concentrations of carbon, hydrogen, and nitrogen, on the appropriate sample basis, as follows:

$$A = \frac{B \times E \times F}{C \times D} \tag{1}$$

where:

A = the percent of the carbon, hydrogen or nitrogen in the sample, mass %,
B = the detector response for carbon, hydrogen or nitrogen in the sample minus response from blank,
C = the detector response for carbon, hydrogen or nitrogen in the standard minus response from blank,
D = the mass of the sample, mg,
E = the mass of the standard, mg, and
F = the percent of the carbon, hydrogen or nitrogen in the standard, mass %.

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

11. Report

11.1 Report the carbon, hydrogen, and nitrogen results as mass percent of the sample.

12. Quality Control

12.1 Confirm the performance of the instrument or the test procedure by analyzing a quality control (QC) sample (6.10).

12.1.1 When QC/Quality Assurance (QA) protocols are already established in the testing facility, these may be used when they confirm the reliability of the test result.

12.1.2 When there is no QC/QA protocol established in the testing facility, Appendix X1 can be used as the QC/QA system.

13. Precision and Bias [12]

13.1 *Precision*:

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[12] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1289.

13.1.1 *Repeatability and Reproducibility*—The repeatability and reproducibility of these test methods (D 5291) for measuring carbon, hydrogen, and nitrogen were determined in a round robin that involved 26 laboratories and fourteen petroleum based samples. A research report based on this round robin is available.[12] Based on this round robin, the following repeatability and reproducibility for these test methods (D 5291) can be expected. This is a joint precision based on all three test methods. No relative bias was found among these test methods.

	Concentration Range (mass %)	Repeatability	Reproducibility
Carbon	75 to 87	$(x + 48.48)0.0072$	$(x + 48.48)0.018$
Hydrogen	9 to16	$(x^{0.5})0.1162$	$(x^{0.5})0.2314$
Nitrogen	0.75 to 2.5	0.1670	0.4456

where: x = the mean value.

13.2 *Bias*—Since there is no accepted petroleum based reference material suitable for determining the bias for the procedure in these test methods (D 5291) for measuring carbon, hydrogen and nitrogen in petroleum products and lubricants, bias cannot be determined.

14. Keywords

14.1 carbon content; CHN analysis; CHN instruments; hydrogen content; nitrogen content

APPENDIX

(Nonmandatory Information)

X1. QUALITY CONTROL MONITORING

X1.1 Confirm the performance of the instrument or the test procedure by analyzing a quality control (QC) sample(s).

X1.2 Prior to monitoring the measurement process, the user of the method needs to determine the average value and control limits of the QC sample (see Practice D 6299 and *ASTM MNL 7*).[13]

X1.3 Record the QC results and analyze by control charts or other statistically equivalent techniques to ascertain the statistical control status of the total testing process (see Practice D 6299 and *ASTM MNL 7*[13]).[14] Investigate any out-of-control data for root cause(s). The results of this investigation may, but not necessarily, result in instrument recalibration.

X1.4 The frequency of QC testing is dependent on the criticality of the quality being measured, the demonstrated stability of the testing process, and customer requirements. Generally, a QC sample should be analyzed each testing day with routine samples. The QC frequency should be increased if a large number of samples are routinely analyzed. However, when it is demonstrated that the testing is under statistical control, the QC testing frequency may be reduced. The QC sample testing precision should be periodically checked against the ASTM method precision to ensure data quality (see Practice D 6299 and *ASTM MNL 7*[13]).

X1.5 It is recommended that, if possible, the type of QC sample that is regularly tested be representative of the material routinely analyzed. An ample supply of QC sample material should be available for the intended period of use, and must be homogenous and stable under the anticipated storage conditions.

X1.6 See Practice D 6299 and *ASTM MNL 7* for further guidance on QC and Control Charting techniques.

SUMMARY OF CHANGES

Subcommittee D02.03 has identified the location of selected changes to this standard since the last issue (D 5291–01) that may impact the use of this standard.

(1) Added Note 3.

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[13] *ASTM MNL 7*, "Manual on Presentation of Data Control Chart Analysis," 6th ed., available from ASTM International Headquarters, W. Conshohocken, PA.

[14] In the absence of explicit requirements given in the test method, this clause provides guidance on QC testing frequency.

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