Designation: D7662 – 15

Standard Test Method for
Carbon Content in Carbon Black Feedstock Oils[1]

This standard is issued under the fixed designation D7662; 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 This test method covers the instrumental determination of carbon content in samples of carbon black feedstock oils. Values obtained represent the total carbon content.

1.2 This test method is applicable to carbon black feedstock oils derived from petroleum, coal, and other sources which include fuel oils, residues, tars, pitches, reclaimed oils, and similar materials that are normally handled as liquids. This test method is applicable to products that typically contain 75 to 94 mass % carbon.

1.3 The results of these tests can be expressed as mass % carbon.

1.4 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D7633 Test Method for Carbon Black—Carbon Content

3. Summary of Test Method

3.1 In this test method, a sample of feedstock oil is weighed in a combustion boat containing a tungsten oxide based accelerator and the carbon content is determined by placing the boat in a tube furnace at 1350°C in an air- or oxygen-enriched atmosphere resulting in complete combustion. Carbon in the sample is completely oxidized to carbon dioxide. Moisture and particulates are removed from the gas stream by traps filled with anhydrous magnesium perchlorate. The gas stream is then passed through a cell in which carbon dioxide concentration is measured by an infrared (IR) absorption detector at a precise wavelength in the IR spectrum.

3.2 This test method is for use with commercially available carbon analyzers equipped to carry out the combustion and measurement operations automatically.

3.3 The carbon analyzer shall be calibrated using an appropriate calibration standard (see 6.5).

3.4 The instrument may be configured to analyze more than one element such as a carbon-sulfur analyzer. In this case, the analyzer has individual detectors for each element that are arranged in series.

4. Significance and Use

4.1 Measuring the total carbon content of feedstock oil is often a necessary component in calculations for carbon dioxide emissions, estimating yield of a process, or assessing product quality.

4.2 The carbon black industry measures carbon content of feedstock oils along with carbon content of carbon black products per Test Method D7633 for Carbon Black-Carbon Content in calculations to determine carbon dioxide emissions for compliance with governmental greenhouse-gas-reporting requirements.

5. Apparatus

5.1 There are a number of commercial instruments available that are designed to measure carbon content in organic materials including carbon and carbon-sulfur analyzers. This test method makes no specifications regarding system designs.

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

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[1] This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.66 on Environment, Health, and Safety.

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[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.

5.2.1 An analytical balance, or equivalent, that is capable of a weighing sensitivity of 0.1 mg or better resolution.

5.2.2 The combustion tube and boat are made of a suitable material such as mullite, porcelain, or zircon.

5.2.3 The sample is absorbed onto tungsten oxide based accelerator to control the sample combustion rate.

5.2.4 The sample is combusted at a temperature of 1350°C in an air- or oxygen-enriched atmosphere.

5.2.5 The combustion gas is passed through an absorbent (anhydrous magnesium perchlorate) to remove water vapor.

5.2.6 The detection system measures carbon dioxide using an IR absorption detector at a wavelength suitable for linear responses with respect to the concentration over the full range of possible concentrations.

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.[3] 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 *Magnesium Perchlorate.*

6.3 *Oxygen, high purity, 99.9 %.*

6.4 *Tungsten (VI) Oxide Accelerator containing potassium dihydrogen-phosphate.*

6.5 *Calibration Standard.* [4]

7. Sampling

7.1 Collect a feedstock oil as specified in Practices D4057 or D4177.

7.2 *Sample*—This is the portion or aliquot of the feedstock oil for use in obtaining a result.

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

7.4 *Transfer*—Use any convenient, clean syringe or pipet to transfer a sample to the combustion boat as described in Section 10.

8. Preparation of Apparatus

8.1 Assemble the apparatus according to the manufacturer's instructions.

8.2 Stabilize the furnace temperature at 1350 ± 15°C.

8.3 Make a minimum of two determinations to condition the equipment or follow the manufacturer's recommendation to condition equipment before calibrating the instrument.

9. Calibration

9.1 Calibrate the instrument per the manufacturer's recommendation using appropriate carbon calibration standards.

9.2 *Adjustment of Response of Measurement System*—Weigh approximately 0.1 g of calibration standard or use the recommended mass per manufacturer. Analyze the sample (see Section 10). Repeat this procedure. Adjust the instrument as recommended by the manufacturer until the absence of drift is indicated. Insure the minimum analysis time is sufficient for complete combustion of the sample.

9.3 *Calibration Procedure*—Follow the calibration procedure recommended by the manufacturer. Confirm the calibration by analyzing a reference material of known carbon concentration. Reference standards should be similar to feedstock oils with carbon content in the range of approximately 75 to 94 %. The measured value should be within allowable limits of the known value. If not, repeat the procedure. If acceptable results are not obtained, refer to the manufacturer's instructions for calibration.

10. Procedure

10.1 Stabilize and verify calibration of the analyzer (see 8.1 – 8.3 and 9.1 – 9.3).

10.2 Confirm the furnace temperature is 1350 ± 15°C.

10.3 Weigh approximately 0.5 to 1 g of tungsten oxide based accelerator into the combustion boat (or use a mass recommended by the manufacturer), spreading evenly.

10.4 Weigh approximately 0.1 to 0.15 g of the sample into the combustion boat (on top the accelerator).

10.5 Record the sample weight.

10.6 Cover the sample with approximately 0.5 to 1 g of tungsten oxide based accelerator.

10.7 Initiate the analysis and place the boat in the instrument using a boat puller or an auto-sampler mechanism.

10.8 When the analysis is complete, the instrument should indicate the carbon value. Refer to the manufacturer's recommended procedure.

11. Report

11.1 The percent carbon value is obtained directly from the apparatus.

11.2 Report results to the nearest 0.1 %.

12. Precision and Bias[5]

12.1 These precision statements have been prepared in accordance with Practice D4483. Refer to this practice for terminology and other statistical details.

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[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 *Annual 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..

[4] Carbon calibration and reference standards are typically available from the instrument manufacturer, and are also available from Alpha Resources, 3090 Johnson Road, Stevensville, MI 49127, www.alpharesources.com.

[5] A research report is forthcoming.

12.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials used in the particular inter-laboratory program described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method. Any appropriate value may be used from Table 1 for the High Temperature Combustion Method. A common alternate method for carbon content, CHN, was included in the inter-laboratory program used to generate this precision statement since a number labs use this instrumental method. Precision data specific to the CHN method is found in Table 2.

12.3 A type 1 inter-laboratory precision program was conducted. Both repeatability and reproducibility represent short-term (daily) testing conditions. The testing was performed in each laboratory performing the test twice on each of two days (total of four tests). A test result is the value obtained from a single determination. Acceptable difference values were not measured.

12.4 The results of the precision calculations for this test are given in Table 1. The materials are arranged in ascending "mean level" order.

12.5 *Repeatability*—The pooled absolute repeatability, r, of this test has been established as 1.80 %. Any other value in Table 1 may be used as an estimate of repeatability, as appropriate. The difference between two single test results (or determinations) found on identical test material under the repeatability conditions prescribed for this test will exceed the repeatability on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results that differ by more than the appropriate value from Table 1 must be suspected of being from different populations and some appropriate action taken.

NOTE 1—Appropriate action may be an investigation of the test method procedure or apparatus for faulty operation or the declaration of a significant difference in the two materials, samples, etc., which generated the two test results.

12.6 *Reproducibility*—The pooled absolute reproducibility, R, of this test has been established as 3.71 %. Any other value in Table 1 may be used as an estimate of reproducibility, as appropriate. The difference between two single and independent test results found by operators working under the prescribed reproducibility conditions in different laboratories on identical test material will exceed the reproducibility on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results produced in different laboratories that differ by more than the appropriate value from Table 1 must be suspected of being from different populations and some appropriate investigative action taken.

12.7 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.

13. Keywords

13.1 carbon black; carbon content; combustion; feedstock; hydrocarbon oil

TABLE 1 Precision Parameters for Test Method D7662, Type 1 Precision, High Temperature Combustion, Carbon Content of Feedstock

Material	Number of Laboratories	Units Percent Mean Level	Sr	r	(r)	SR	R	(R)
5. NA Tar Oil	5	80.10	0.843	2.39	2.94	2.240	6.34	7.83
6. Leco 502-816 (85.7 % C)	5	86.52	0.502	1.42	1.64	0.925	2.62	3.03
7. Asian Coal Tar	5	90.70	0.454	1.29	1.42	0.547	1.55	1.71
8. NA Coal Tar	5	91.75	0.671	1.90	2.07	0.841	2.38	2.59
Average		87.49						
Pooled Values			0.636	**1.80**	2.06	1.312	**3.71**	4.24

TABLE 2 Precision Parameters for Test Method D7662, Type 1 Precision, CHN, Carbon Content of Feedstock

Material	Number of Laboratories	Units Percent Mean Level	Sr	r	(r)	SR	R	(R)
5. NA Tar Oil	8	82.20	0.509	1.441	1.75	1.269	3.59	4.37
6. Leco 502-816 (85.7 % C)	8	85.99	0.346	0.978	1.14	0.426	1.21	1.40
7. Asian Coal Tar	8	90.92	0.528	1.495	1.64	0.887	2.51	2.76
8. NA Coal Tar	8	91.85	0.314	0.888	0.97	0.689	1.95	2.12
Average		87.74						
Pooled Values			0.435	**1.23**	1.40	0.874	**2.47**	2.82

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