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Standard Test Method for Carbon Black—Oil Absorption Number (OAN)[1]

This standard is issued under the fixed designation D 2414; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the oil absorption number of carbon black.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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:* [2]

D 1765~~Classification System for Carbon Blacks Used in Rubber Products~~ Test Method for Measuring Friction and Wear Properties of Lubricating Grease Using a High-Frequency, Linear-Oscillation (SRV) Test Machine

D 1799~~Practice for Carbon BlackSampling Packaged Shipments~~ Test Method for Measuring Friction and Wear Properties of Lubricating Grease Using a High-Frequency, Linear-Oscillation (SRV) Test Machine

D 1900~~Practice for Carbon BlackSampling Bulk Shipments~~ Test Method for Measuring Friction and Wear Properties of Lubricating Grease Using a High-Frequency, Linear-Oscillation (SRV) Test Machine

D 4483~~Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries~~ Test Method for Measuring Friction and Wear Properties of Lubricating Grease Using a High-Frequency, Linear-Oscillation (SRV) Test Machine

D 4821~~Guide for Carbon BlackValidation of Test Method Precision and Bias~~ Test Method for Measuring Friction and Wear Properties of Lubricating Grease Using a High-Frequency, Linear-Oscillation (SRV) Test Machine

3. Summary of Test Method

3.1 In this test method, oil is added by means of a constant-rate buret to a sample of carbon black in the mixer chamber of an absorptometer. As the sample absorbs the oil, the mixture changes from a free-flowing state to one of a semiplastic agglomeration, with an accompanying increase in viscosity. This increased viscosity is transmitted to the torque-sensing system of the absorptometer. When the viscosity of the mixture reaches a predetermined torque level, the absorptometer and buret will shut off simultaneously. The volume of oil added is read from the direct-reading buret. The volume of oil per unit mass of carbon black is the oil absorption number.

3.2 Either DBP or paraffin oil is acceptable for use with standard pelleted grades including N-series carbon blacks found in Classification D 1765. OAN testing using paraffin oil on some specialty blacks and powder blacks may result in unacceptable differences as compared to OAN testing using DBP oil. While studies have shown either oil to exhibit comparable precision, paraffin oil offers the advantage of being non-hazardous; even FDA-approved grades are available. For either oil, Sections 8-11 (Calibration, Procedure, Calculation, and Report) are to be consistent with the oil selected for use. Referee testing between suppliers and users should use DBP oil until such time that precision data are available for paraffin oil.

4. Significance and Use

4.1 The oil absorption number of a carbon black is related to the processing and vulcanizate properties of rubber compounds containing the carbon black.

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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 D24.11 on Carbon Black Structure .

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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. Apparatus [3]

5.1 *Balance*, analytical, with an 0.01-g sensitivity.
5.2 *Oven*, gravity-convection type, capable of maintaining 125° ± 5°C.
5.3 *Spatula*, rubber, 100-mm.
5.4 *Absorptometer,* equipped with a constant-rate buret that delivers 4 ± 0.024 cm ^{3}/min.[4]
5.5 *Desiccator*.

6. Reagent and Standards

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.[5] 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 *n-Dibutyl Phthalate*, having a density of 1.042 to 1.047 Mg/m^3 at 25°C and a relative density of 1.045 to 1.050 at 25°C.
6.3 *Paraffin Oil*, having a kinematic viscosity of 10 to 34 mm^2/s (cSt) at 40°C.

NOTE 1—Three paraffin oils have been found suitable including Marcol 82 from Exxon, 80/90 White Oil from Conoco-Phillips, and LC1 oil from Lab Chemicals, Germany. All three oils are pharmaceutical or food grade oil, or both, based on available data.

6.4 ASTM D24 Standard Reference Blacks, SRB-6.[6]

7. Sampling

7.1 Samples shall be taken in accordance with Practices D 1799 and D 1900.

8. Calibration and Standardization

8.1 *Absorptometer*:
8.1.1 *Model*—Three different types of absorptometers are in use: 1) early models based on springs and mechanical indication of torque (Type A and B), 2) second generation absorptometers equipped with load cells and digital torque display (Type E), and 3) current model absorptometers controlled via microcomputer software (Type C and H) and Type E absorptometers which utilize add-on data acquisition and control systems. Several components influence the calibration: the dynamometer torque spring or the load cell, the torque limit switch or the indicator set-point, the damper (oil damper or electronic damping), and the mixing head consisting of two counter-rotating blades and a mixing bowl. It is necessary that all of these components are in good condition and are properly adjusted to achieve acceptable calibration.
8.1.2 *Mixing Bowl*— Typically the absorptometer is delivered with either a surface-treated stainless steel or anodized aluminum mixing bowl. These bowls are considered acceptable provided they give the correct reading for the appropriate SRB reference standards. The surface finish of the mixer chamber is critical for maintaining proper calibration, and the bowl should not be modified to achieve calibration.

NOTE 2—Stainless steel chambers have been found satisfactory for the test when they are manufactured to a roughness value (Ra) of 2.5 ± 0.4 µm (100 ± 15 µin.) based upon 8 measurements. No single measurement should be greater than 3.6 µm (140 µin.) or less than 1.5 µm (60 µin.). Stainless steel bowls purchased with an absorptometer have been pre-polished for 16 h to minimize bowl surface changes affecting calibration during their initial use. It is recommended that new replacement stainless steel bowls should also be pre-polished in the same manner (see Annex A3).

8.2 *Calibration*:
8.2.1 *Rotor Blades*— The speed of the motor driving the rotor blades is either fixed (Type A and B) or has to be set (Type E, C, and H) to 125 r/min. Due to a gear, one blade spins at 125 r/min, the other blade at 250 r/min.
8.2.2 *Constant-Rate Buret*—The delivery rate of the buret is to be 4 cm^3/min. See Annex A1 for detailed instructions on the procedure for calibration check of the constant-rate buret.
8.2.3 *Spring Tension (Type A and B)*—It is recommended that the torque spring is adjusted so that the SRB F standard will develop a maximum torque between 70 % and full-scale deflection. This is achieved by selecting the appropriate spring strength and adjusting the spring tension in accordance with the instructions of the manufacturer.

NOTE 3—The absorptometers Type E, C, and H are calibrated by the manufacturer to give a direct reading of torque in mNm; this calibration should not be modified in order to achieve a desired level of torque. If calibration is necessary, refer to the instrument manufacturer's recommendations. The instrument torque calibration should not be confused with the torque limit switch described in 8.2.5.

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[3] All apparatus are to be operated and maintained in accordance with the manufacturers' directions for optimum performance.
[4] Available from C. W. Brabender Instruments, Inc., 50 E. Wesley St., Sout Hackensack, NJ 07606 (www.brabender.com) and from HITEC Luxembourg, 5 Rue de l'Eglise, L-1458 Luxembourg (www.hitec.lu).
[5] *Reagent Chemicals, American Chemical Society Specifications* , American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K. and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.
[6] The sole source of supply of the apparatus known to the committee at this time is Laboratory Standards and Technologies, 227 Somerset, Borger, TX 79007. 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, which you may attend.

8.2.4 *Damper*—For the Type A absorptometer, it is recommended to keep the valve of the oil damper fully closed. The Type B absorptometer shall provide a full-scale recovery of 3 ± 0.5 s; the valve has to be adjusted accordingly. The Type E absorptometer has an electronic damping option and Types C and H have appropriate software damping. Make sure that these damping options are activated.

8.2.5 *Torque Limit Switch (TLS) or the Indicator Set Point*—If the end-point of the test is determined by a fixed torque limit, the setting of the TLS, also called indicator set-point, has to be selected using one of the following three procedures:

8.2.5.1 *Procedure A: End-Point at Fixed Torque Level*—This "classical" method is well suited for hard or tread blacks but often leads to problems when low-torque carcass blacks are to be tested. For Type A and B absorptometers, adjust the TLS or the indicator set point in such way that the current SRB F standard gives the correct target values within the limits as defined in Guide D 4821. For Type E absorptometers dedicated to testing carcass blacks only, set the TLS in such a way that the current SRB F standard gives the correct target values within the limits as defined in Guide D 4821. For Type E, C, and H absorptometers dedicated to testing tread blacks only, there is no advantage to setting the TLS with current SRB F standard; for these absorptometers set the TLS between 3000 and 4000 mNm (preferred TLS is 3500 mNm when using DBP oil, or 4000 mNm if using paraffin oil).

8.2.5.2 *Procedure B: End-Point at 70 % of the Maximum Torque*—Certain carcass blacks and thermal blacks may fail to give an end-point due to insufficient torque level. Therefore, the preferred method for testing soft blacks is to record the torque curve using a chart-recorder or a data acquisition system and to read the end-point at 70 % of the maximum of the torque achieved. Set the TLS or the indicator set point to full scale in order to disable the automatic shut-off of the absorptometer.

8.2.5.3 *Procedure C: End-Point at a Fixed, But Reduced Torque Level*—Requires use of SRB-5 series standards. See Test Method D 2414 – 00. ~~Note4—For Type E, C, and H absorptometers dedicated to testing tread blacks only, or for Type E, C, and H absorptometers in which procedure A is used for testing tread blacks and procedure B is used for testing carcass blacks, there is no advantage to setting the TLS with the current SRB F standard; for these absorptometers set the TLS between 3000 and 4000 mNm (preferred TLS is 3500 mNm when using DBP oil, or 4000 mNm if using paraffin oil).~~

8.3 *Standardization*:

8.3.1 Physically calibrate the test apparatus including TLS adjustment using the instructions in 8.2.

8.3.2 Test the six ASTM Standard Reference Blacks (SRBs) in duplicate to establish the average measured value. Additional values are added periodically, typically on a weekly basis. The rolling average of the measured values is computed from the latest four values.

NOTE ~~5—When~~ 4—When only tread- or carcass-type carbon blacks are to be tested, the calibration can be limited to either the three tread- (A, B, C) or the three carcass-type (D, E, F) carbon black standards.

8.3.3 Perform a regression analysis using the standard value of the standard (*y* value) and the rolling average measured value (*x* value). Separate carcass and tread calibration curves should be maintained.

8.3.4 Normalize the values of all subsequent samples as follows:

$$\text{Normalized value} = (\text{measured value} \times \text{slope}) + y\text{–intercept} \quad (1)$$

8.3.5 For measured values on the SRBs that are consistently outside the expected variability listed in Guide D 4821, the test apparatus should be recalibrated in accordance with 8.2.

8.3.6 When any absorptometer or calibration changes occur, a new calibration curve must be initiated as described in 8.3.2.

8.3.7 In most instances, if proper calibration cannot be achieved by following 8.2 or 8.3.2-8.3.4, it will be necessary to replace the mixer chamber with one of proper surface finish.

9. Procedure

9.1 Dry an adequate sample for 1 h in the specified oven set at 125°C. Prior to testing, cool the sample in a desiccator for a minimum of 30 min.

9.2 Weigh the sample to the nearest 0.01 g. The recommended masses are as follows:

Carbon Black	Mass, g
N630, N642, and N700 series, except N765	25
N800 and N900 series, SRB D-7	40
All others	20

9.3 It is recommended that a testing temperature of 23 ± 5°C be maintained, as measured by a thermocouple in the mixing bowl. If a temperature controllable mixing bowl is not available, keep the bowl temperature below 30°C and comply with ~~Note 6~~ Note 5 and ~~Note 7~~ Note 6 while running the samples.

NOTE ~~6—If~~ 5—If the absorptometer has remained idle for more than 15 min and a temperature controllable bowl is not being used, a 10-min warm-up sample must be run before beginning a test. It is important that the mixer chamber temperature be kept uniform. Preferably, allow 5 min between the end of one test and the start of another.

NOTE ~~7—It~~ 6—It is important that the temperature of the bowl be the same for machine calibration as for oil absorption testing. ASTM task group work has shown that an increase in bowl temperature can cause higher values that increased variability in bowl temperatures cause increased test variability.

NOTE ~~8—In~~ 7—In the event that an endpoint is not obtained (maximum torque < TLS) when using an absorptometer with a fixed TLS such as Type