Designation: D 2414 – 05a

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

D 1799 Practice for Carbon Black—Sampling Packaged Shipments

D 1900 Practice for Carbon Black—Sampling Bulk Shipments

D 4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D 4821 Guide for Carbon Black—Validation of Test Method Precision and Bias

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.

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,* [4] equipped with a constant-rate buret that delivers 4 ± 0.024 cm^3/min.

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,

[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.

[3] All apparatus is to be operated and maintained in accordance with the manufacturer's directions for optimum performance.

[4] Available from C. W. Brabender Instruments, Inc., 50 E. Wesley St., Sout Hackensack, NJ 07606 and from HITEC Luxembourg, 5 Rue de l'Eglise, L-1458 Luxembourg.

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 and Marcol 9 from Exxon and Sunpar LW107 from SUNOCO.

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*—Two different types of absorptometers are in use: older models based on springs and mechanical indication of torque (Type A and B) and the current absorptometers equipped with load cells and digital torque display (Type E and Type H). 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 a velvetized stainless steel mixing bowl. Other chamber materials like aluminum, soft- or hard-anodized, are also acceptable provided they give the correct reading for the SRB F-6 after calibration. 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 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 *Spring Tension (Type A and B)*—It is recommended that the torque spring is adjusted so that the SRB F-5 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 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 for SRB F–6. 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 8.2.4.

8.2.3 *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 Type H has an appropriate software damping. Make sure that these damping options are activated.

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

8.2.4.1 *Procedure A: End-Point at Fixed Torque Level*—This "classical" method is well suited for tread blacks but often leads to problems when low-torque carcass blacks are to be tested. Adjust the torque limit switch or the indicator set point in such way that the SRB F-6 gives a value of 133.6 ± 3.3 $cm^3/100$ g.

8.2.4.2 *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 torque limit switch or the indicator set point to full scale in order to disable the automatic shut-off of the absorptometer.

8.2.4.3 *Procedure C: End-Point at a Fixed, But Reduced Torque Level*—The reduced value on SRB F-6 still needs to be established. For now, if Procedure C is desirable, use SRB-5 series standards.

8.2.5 *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.

8.3 *Standardization*:

8.3.1 Physically calibrate the test apparatus 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 4—When only tread or carcass-type carbon blacks are to be tested, the calibration can be limited to either the three tread (A-6, B-6,

[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,[1] which you may attend.

C-6) or the three carcass (D-6, E-6, F-6) type carbon black standards. Even if only tread blacks are being tested, F-6 must be used to set the torque limit switch.

8.3.3 Perform a regression analysis using the standard value of the standard (y value) and the rolling average measured value (x value). It is recommended that separate carcass and tread calibration curves be maintained.

NOTE 5—Note that the standard value of SRB F-6 (133.6 cm^3/100 g) has to be used as the y value in the regression for the absorptometer calibration Procedures A and B used in 8.2.5.

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 Alternatively, a table of numbers may be generated, based on the regression equation, to find the correspondence between a measured and calibrated value.

8.3.6 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.

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

8.3.8 In most instances, if proper calibration cannot be achieved by following 8.2 or 8.3.2-8.3.5, 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	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 and Note 7 while running the samples.

NOTE 6—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 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 the event that an endpoint is not obtained (maximum torque < TLS) when using an absorptometer with a fixed TLS such as Type B or E, it is acceptable to mill pelleted carbon blacks using a coarse grinder such as a coffee mill. The carbon black should be milled for only a few seconds to allow sufficient grind time to change the pellets to powder form. High-speed micronizing mills and air-jet mills are not acceptable, as they can reduce the carbon black structure.

9.4 Transfer the sample to the absorptometer mixer chamber and replace the cover.

9.5 Place the instrument main-power switch in the "on" position and the automatic-manual switch in the "automatic" position. For the Type E absorptometer, turn the on–off switch to the "on" position.

9.6 Turn the stopcock to connect the buret to the oil supply and activate the return switch. The plunger will be drawn down, filling the buret barrel with oil.

9.7 When the lower-limit switch stops the plunger, reverse the stopcock and place a waste receptacle under the delivery tube. Activate the up switch and purge the delivery line to make certain the buret delivery tube is free of air bubbles. Deliver approximately 1 cm^3 of oil into a waste receptacle, to ensure that the delivery tube is filled to capacity.

9.8 Position the buret delivery tube over the hole in the mixer chamber cover, and set the buret digital counter to zero.

9.9 Activate the "start" button. On the Type E absorptometer, activate both "start" buttons simultaneously. The apparatus will operate until sufficient torque has developed to activate the torque-limit switch, which will halt the absorptometer and buret.

9.10 Record the volume of oil used as indicated by the buret digital counter.

9.11 Dismantle the mixer chamber and clean the mixing blades and chamber with a rubber spatula and reassemble.

NOTE 9—It is not necessary to clean and polish the mixing blades and chamber with a solvent.

10. Calculation

10.1 Calculate the oil absorption number of the sample to the nearest 0.1 $10^{-5}m^3$/kg (cm^3/100 g) as follows:

$$\text{Oil absorption number, } 10^{-5}m^3/\text{kg} = \frac{A}{B} \times 100 \quad (2)$$

where:

A = volume of oil used, cm^3, and
B = mass of tested sample, g.

11. Report

11.1 Report the following information:

11.1.1 Proper identification of the sample,

11.1.2 Oil (DBP or paraffin,)

11.1.3 Method for end-point determination (Procedure A, B or C in 8.2),

11.1.4 Sample mass, if different than shown in 9.2, and

11.1.5 The result obtained from the individual determination is reported to the nearest 0.1 $10^{-5}m^3$/kg (cm^3/100 g).

12. Precision and Bias

12.1 These precision statements have been prepared in accordance with Practice D 4483. Refer to this practice for terminology and other statistical details.

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 interlaboratory 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.