



Designation: **D2414 – 13** **D2414 – 13a**

## Standard Test Method for Carbon Black—Oil Absorption Number (OAN)<sup>1</sup>

This standard is issued under the fixed designation D2414; 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.

*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:<sup>2</sup>

- D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
- D1218 Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids
- D1765 Classification System for Carbon Blacks Used in Rubber Products
- D1799 Practice for Carbon Black—Sampling Packaged Shipments
- D1900 Practice for Carbon Black—Sampling Bulk Shipments
- D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
- D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries
- D4821 Guide for Carbon Black—Validation of Test Method Precision and Bias
- D5554 Test Method for Determination of the Iodine Value of Fats and Oils

#### 2.2 DIN Standards:<sup>3</sup>

- DIN 16945 Testing of resins, hardeners and accelerators, and catalyzed resins
- DIN EN ISO 660 Animal and vegetable fats and oils - Determination of acid value and acidity

### 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, paraffin or epoxidized sunflower oils are acceptable for use with most standard pelleted grades of carbon black including N-series carbon blacks found in Classification D1765. OAN testing using paraffin oils or epoxidized sunflower oils on some standard blacks and specialty blacks including powder products may result in unacceptable differences as compared to OAN testing with DBP oil. Paraffin and epoxidized sunflower oils are considered non-hazardous; some paraffin oils are FDA approved. For any of the oils, 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 alternate oils.

<sup>1</sup> 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. Current edition approved July 1, 2013. Published August 1, 2013. Originally approved in 1965. Last previous edition approved in 2012 as D2414 – 12; D2414 – 13. DOI: 10.1520/D2414-13.10.1520/D2414-13A.

<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 Deutsches Institut für Normung e.V. (DIN), Burggrafenstrasse 6, 10787 Berlin, Germany, <http://www.din.de>.

#### 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<sup>4</sup>

5.1 *Balance*, analytical, with an 0.01-g sensitivity.

5.2 *Oven*, gravity-convection type, capable of maintaining  $125^{\circ} \pm 5^{\circ}\text{C}$ .

5.3 *Spatula*, rubber, 100-mm.

5.4 *Absorptometer*, equipped with a constant-rate buret that delivers  $4 \pm 0.024 \text{ cm}^3/\text{min}$ .<sup>5</sup>

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.<sup>6</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 *n-Dibutyl Phthalate*, having a density of 1.042 to 1.047 Mg/m<sup>3</sup> 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<sup>2</sup>/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 *Epoxidized Fatty Acid Ester (EFA)*, meeting the specifications listed in **Annex A4**. It is recommended to store the product at temperatures between 7 and 30°C. If stored in sealed original containers, the product is stable for at least 12 months. For handling and safety, please refer to safety data sheet.

6.5 ASTM D24 Standard Reference Blacks, SRB.<sup>7</sup>

#### 7. Sampling

7.1 Samples shall be taken in accordance with Practices **D1799** and **D1900**.

#### 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<sup>8</sup>), and (3) current model absorptometers which are designed with a torque measuring system that includes a micro-computer and software to continuously record torque and oil volume with time (Types H and C and modified Type E<sup>8</sup>). Types A, B, and E<sup>8</sup> are designed to stop mixing at a predetermined, fixed torque level, which is the recommended procedure for measuring hard or tread blacks (calibration Procedure A). The computer controlled models (Types H and C and modified Type E<sup>8</sup>) are required for running calibration Procedure B, the recommended torque curve analysis for the determination of the end-point of soft or carcass blacks. The Type H and C and modified Type E<sup>8</sup> absorptometers can also provide an end-point at a fixed or predetermined torque level such that these types of absorptometers are well-suited for measuring OAN of both hard and soft carbon blacks. 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.

<sup>4</sup> All apparatus are to be operated and maintained in accordance with the manufacturers' directions for optimum performance.

<sup>5</sup> Available from C. W. Brabender Instruments, Inc., 50 E. Wesley St., Sout Hackensack, NJ 07606 ([www.brabender.com](http://www.brabender.com)) and from HITEC Luxembourg, 5 Rue de l'Eglise, L-1458 Luxembourg ([www.hitec.lu](http://www.hitec.lu)).

<sup>6</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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

<sup>7</sup> The sole source of supply of ASTM Standard Reference Blacks known to the committee at this time is Laboratory Standards and Technologies, 227 Somerset, Borger, TX 79007, <http://carbonstandard.com/>. 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,<sup>1</sup> which you may attend.

<sup>8</sup> Type E absorptometers can be modified with additional hardware and micro-computer system.

NOTE 2—Stainless steel chambers have been found satisfactory for the test when they are manufactured to a roughness value (Ra) of  $2.5 \pm 0.4 \mu\text{m}$  ( $100 \pm 15 \mu\text{in.}$ ) based upon 8 measurements. No single measurement should be greater than  $3.6 \mu\text{m}$  ( $140 \mu\text{in.}$ ) or less than  $1.5 \mu\text{m}$  ( $60 \mu\text{in.}$ ). 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  $4 \text{ cm}^3/\text{min}$ . 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](#).

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  $3 \pm 0.5 \text{ s}$ ; 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 most hard or tread blacks but may lead to problems when low-torque carcass blacks are to be tested; proceed to Procedure B for low-torque carbon blacks. For Type A, B, and E absorptometers, adjust the TLS or the indicator set point in such way that the current SRB F standard gives the correct target value within the limits as defined in [Guide D4821](#). For Type E, C, and H absorptometers dedicated to testing tread blacks only, there is no advantage to setting the TLS based on the SRB F standard; for these absorptometers, set the TLS to 3500 mNm for DBP oil, or 4000 mNm for 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 D2414 – 00.

## 8.3 Normalization:

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 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 normalized values of the SRBs that are consistently outside the x-chart limits listed in [Guide D4821](#), 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. [Review Appendix X1](#).

## 9. Procedure

9.1 Dry an adequate sample for 1 h in the specified oven set at  $125^\circ\text{C}$ . 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 and D-8	40
All others	20

9.3 It is recommended that a testing temperature of  $23 \pm 5^{\circ}\text{C}$  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  $30^{\circ}\text{C}$  and comply with **Note 5** and **Note 6** while running the samples.

**NOTE 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 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 7**—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 chamber cover. For Type H, close the safety door surrounding the mixing chamber.

9.5 Position the buret delivery tube over the hole in the mixer chamber cover, and for Types A, B, or E set the buret digital counter to zero (Types C and H have automatic reset). Insure the buret delivery tubes have no air bubbles.

9.6 Activate the “start” button. On the Type E absorptometer, activate both “start” buttons simultaneously. The apparatus will operate until one of the following conditions are met: 1) sufficient torque has developed to activate the torque-limit switch, which will halt the absorptometer and buret; 2) the sample torque has reached a maximum and then dropped below maximum torque for a preset period of time (using Procedure B).

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

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

9.9 Mixing chamber cleanup can be aided by the addition of dry carbon black to the mixing chamber prior to disassembly, and the use of the preset cleanup cycle for Types E, C, and H (use of water to aid cleanup is not recommended).

**NOTE 8**—It is not necessary to clean and polish the mixing blades and chamber with a solvent, but it is recommended to remove all visible residues by wiping the chamber and mixing blade surfaces.

## 10. Calculation

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

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

where:

- A = volume of oil used,  $\text{cm}^3$ , 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, paraffin, or epoxidized sunflower oil),
- 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  $0.1 \cdot 10^{-5}\text{m}^3 / \text{kg}$  ( $\text{cm}^3/100 \text{ g}$ ).

## 12. Precision and Bias

12.1 These precision statements have been prepared in accordance with Practice **D4483**. 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**.

12.3 A type 1 interlaboratory precision program was conducted as detailed in **Table 1**. Both repeatability and reproducibility represent short-term (daily) testing conditions. The testing was performed using two operators in each laboratory performing the

**TABLE 1 Precision Parameters for D2414, Carbon Black—Oil Absorption, (Type 1 Precision)<sup>A</sup>**

Units	Number of Laboratories	10 <sup>-5</sup> m <sup>3</sup> /kg (cm <sup>3</sup> /100 g)				
Material		Mean Level	<i>Sr</i>	<i>r</i>	<i>SR</i>	<i>R</i>
SRB D6 (N762)	15	67.4	0.50	1.40	0.77	2.17
SRB C6 (N326)	17	70.3	0.35	0.99	0.86	2.44
SRB E6 (N660)	15	88.2	0.60	1.69	0.72	2.03
SRB B6 (N220)	18	114.3	0.37	1.04	0.54	1.54
SRB A6 (N134)	18	123.7	0.61	1.73	1.05	2.97
SRB F6 (N683)	14	133.6	1.11	3.14	2.98	8.43
Averaged Pooled Values		99.6	0.64	1.82	1.42	4.02

<sup>A</sup> Precision data in Table 1 were obtained with DBP oil. ASTM taskforce studies with paraffin oil have shown similar precision as DBP oil. Future precision studies will include paraffin oil, such that similar data will become available.

test once 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. The between operator component of variation is included in the calculated values for *r* and *R*.

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 1.82 10<sup>-5</sup> m<sup>3</sup>/kg (cm<sup>3</sup>/100 g). 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 9—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, and so forth, which generated the two test results.

12.6 *Reproducibility*—The pooled absolute reproducibility, *R*, of this test method has been established as 4.02 10<sup>-5</sup> m<sup>3</sup>/kg (cm<sup>3</sup>/100 g). 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 two 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 or technical/commercial 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; n–dibutyl phthalate; oil absorption number; paraffin oil

## ANNEXES

### (Mandatory Information)

#### A1. CALIBRATION CHECK OF CONSTANT-RATE BURET

##### A1.1 Scope

A1.1.1 The constant-rate buret is an integral part of the absorption-measuring system. Failure of the buret to deliver the specified amount of reagent to the carbon black will result in erroneous absorption readings. This annex provides a method for checking the delivery rate of the constant-rate buret. One of the reasons for the incorrect absorption values (caused by incorrect reagent delivery by the automatic buret) is entrapped air in the plastic tubing or the delivery tube, especially above the nozzle. This trouble source should be checked first.



## A1.2 Apparatus

A1.2.1 *Stop Watch.*

A1.2.2 *Beaker, 150-cm<sup>3</sup>.*

## A1.3 Procedure

A1.3.1 Ensure that all seals and tubing are in good condition.

A1.3.2 Fill the buret and delivery tubes with oil. Ensure that all air is removed from the system.

A1.3.3 With the buret completely full, set the stopcock to the delivery position. Run the buret on “deliver” until a constant flow is obtained from the delivery tube.

A1.3.4 Stop the buret and set the digital counter to zero.

A1.3.5 Position a tared 150-cm<sup>3</sup> beaker under the delivery tube.

A1.3.6 Simultaneously start the buret and stop watch.

A1.3.7 After 2 min, stop the buret and record the digital counter reading.

A1.3.8 Weigh and record the amount of reagent delivered.

A1.3.9 Refill the buret.

A1.3.10 Repeat A1.3.3 – A1.3.9, changing the delivery time in A1.3.7 to 4 and 8 min.

## A1.4 Calculation

A1.4.1 Calculate the volume of oil from the delivered mass and density (A1.3.8) as follows:

$$\text{Delivery, cm}^3 = \frac{\text{mass delivered}}{\text{oil density}} \quad (\text{A1.1})$$

## A1.5 Acceptable Results

A1.5.1 The calculated delivery should be within the following limits of the digital counter reading:

Time, min	Volume, cm <sup>3</sup>	Tolerance, cm <sup>3</sup>
2	8	±0.05
4	16	±0.10
8	32	±0.20

## A1.6 Oil Density

A1.6.1 Oil density is necessary to calculate the volume of oil delivered from a buret as described in A1.4. Oil densities may be analyzed using calibrated hydrometers or density meters at specified temperatures. Typical densities for both DBP and paraffin oil