Designation: D 2414 – 03

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.

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:*

D 1799 Practice for Carbon Black—Sampling Packaged Shipments[2]

D 1900 Practice for Carbon Black—Sampling Bulk Shipments[2]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

D 4821 Guide for Carbon Black—Validation of Test Method Precision and Bias[2]

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. 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 is 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, 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[6]

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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 Absorptive Properties of Carbon Black.

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[2] *Annual Book of ASTM Standards*, Vol 09.01.

[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., South Hackensack, NJ 07606 and from HITEC Luxembourg, 5 Rue de l'Eglise, L-1458 Luxembourg.

[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] 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.[7]

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 (Brabender Type A and B) and the current ones (Brabender Type E and Hitec Type H) equipped with a load cell and a digital torque display. 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 1—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 2—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[8] 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 3—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, 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 4—Note that the standard value of SRB F-6 (133.6 cm^3/100g) has to be used as the y value in the regression for the absorptometer calibration procedures A and B used in 8.2.5.

[7] 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.

[8] A data acquisition system designed for recording and evaluating oil measurements is available from HITEC Luxembourg, 5 Rue de l'Eglise, L-1458 Luxembourg

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 to 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 and N785	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 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.

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 7—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:

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

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.

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 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 $10^{-5}m^3/kg$ ($cm^3/100g$). Any other value in Table 1 may be used as an estimate of

TABLE 1 Precision Parameters for D 2414, Carbon Black—Oil Absorption, (Type 1 Precision)[A]

Units	Number of Laboratories	10^{-5} m^3/kg (cm^3/100 g)				
Material		Mean Level	*Sr*	*r*	*SR*	*R*
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		99.6				
Pooled Values			0.64	1.82	1.42	4.02

[A] Precision data in Table 1 was 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.

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 8—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 has been established as 4.02 $10^{-5}m^3$/kg (cm^3/100g). 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^3.

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^3 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:

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

A1.5 Acceptable Results

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

Time, min	Tolerance, cm^3
2	±0.05
4	±0.10
8	±0.20

A2. DETERMINATION OF MAXIMUM TORQUE

A2.1 Scope

A2.1.1 On some instruments the SRB F-6 (an N683 carbon black) will not develop sufficient torque to produce acceptable test precision. This is an indication that other similar type carbon blacks may also test with poor precision.

A2.1.2 In order to obtain acceptable test precision in these situations, it is necessary that the absorptometer be adjusted mechanically or electronically for Type E absorptometers so that the F-6 SRB will develop a maximum torque of at least 70 % of full scale. This procedure gives the needed instructions to determine the maximum torque developed by a carbon black sample.

A2.2 Procedure

A2.2.1 Set the torque pointer to 10 on the Set Scale. For Type E absorptometers, move the shut–off alarm set point to maximum scale. This makes 100 % of the torque range available.

NOTE A2.1—Torque limit switch settings should always be made with the instrument stopped and the mixing chamber empty.

A2.2.2 Start the apparatus having followed 9.1 through 9.7 for testing SRB F-6.

A2.2.3 As the sample begins to develop viscosity and the torque increases, the pointer will move down the scale towards zero. The maximum % torque, T_{max}, developed by the sample is as follows:

$$T_{max} = (10 - N_{min}) \times 10 \quad \text{(A2.1)}$$

where:
N_{min} = the lowest pointer reading.

A3. PRE-POLISHING PROCEDURE FOR NEW REPLACEMENT STAINLESS STEEL BOWLS

A3.1 Scope

A3.1.1 It is recommended that new replacement stainless steel bowls manufactured with a 2.5 ± 0.4 µm (100 ± 15 µin.) roughness be pre-polished for 16 h prior to their use for oil absorption testing. This will minimize the calibration changes for the absorptometer that will probably occur without the pre-polishing.

A3.2 Reagents

A3.2.1 *Carbon Black* (SRB F-6).
A3.2.2 *n-Dibutyl phthalate or paraffin oil.*

A3.3 Procedure

A3.3.1 Weigh 25 g of SRB F-6 carbon black and transfer this sample into the absorptometer mixing chamber.

A3.3.2 Turn on the absorptometer and add 35 cc of oil.

NOTE A3.1—Relieve the torque limit switch to prevent automatic shutoff. It may be necessary to increase the spring tension.

A3.3.3 Allow the absorptometer to run continuously for 16 h.

NOTE A3.2—The absorptometer bowl must be securely covered during this time to prevent the loss of sample to be able to achieve adequate pre-polishing action.

A3.3.4 After 16 h, stop the absorptometer, empty the sample, and clean the mixing chamber and blades. Allow the chamber to cool to room temperature.

A3.3.5 Check and adjust the torque-switch setting and the spring tension before proceeding with calibration following the standard testing procedure.