Designation: D 3493 – 04

Standard Test Method for Carbon Black—Oil Absorption Number of Compressed Sample (COAN)[1]

This standard is issued under the fixed designation D 3493; 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 procedure for the mechanical compression of a carbon black sample and the determination of the oil absorption number of the compressed sample.

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 1799 Practice for Carbon Black—Sampling Packaged Shipments

D 1900 Practice for Carbon Black—Sampling Bulk Shipments

D 2414 Test Method for Carbon Black—Oil Absorption Number

D 3324 Practice for Carbon Black—Improving Test Reproducibility Using ASTM Standard Reference Blacks[3]

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

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

3. Summary of Test Method

3.1 A sample of carbon black is compressed four times in a compression cylinder at a pressure of 165 MPa (24 000 psi) and then tested in an absorptometer to determine the oil absorption number.

3.2 *n* -Dibutyl phthalate or paraffin oil is added by means of a constant-rate buret to the compressed 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 simultaneously shut off. 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. Either DBP or paraffin oil is acceptable for use with many standard pelleted grades of N-series carbon blacks found in Classification D 1765. COAN testing using paraffin oil on some carbon black products may result in unacceptable differences as compared to OAN testing using DBP oil. 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.

4.2 The difference between the regular oil absorption number and the oil absorption number of compressed sample is some measure of the stability of the structure of the carbon black.

5. Apparatus [4]

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

5.2 *Oven*, gravity-convection type, capable of maintaining 125° ± 5°C.

5.3 *Carbon Black Press*, capable of compressing a 25 g sample to 165 MPa (24 000 psi).[5]

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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] 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] Withdrawn.

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

[5] Carbon black presses from the following companies have been found satisfactory for this test method: EG&G Engineering, P.O. Box 470710, Tulsa, OK 74147–0710 and Titan Specialties, Inc., P.O. Box 2316, Pampa, TX 79066-2316.

5.4 *Absorptometer*,[6] equipped with a constant-rate buret which delivers 67 ± 0.4 mm^3/s (4 ± 0.024 cm^3/min).

5.5 *Spatula*, rubber, 100-mm.

5.6 *Sieve*, 850 µm (U.S. No. 20), approximately 125-mm (5-in.) diameter with receiver pan.

5.7 *Brush*, approximately 40-mm (1.5-in.), stiff bristle.

5.8 *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.[7] 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[8]

6.4 *ASTM D24 Standard Reference Blacks*, SRB-6.[9]

7. Sampling

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

8. Calibration and Standardization

8.1 See Test Method D 2414.

NOTE 1—If values are not obtained within the acceptable range, it will be necessary to either vary the pressure of the hydraulic press until acceptable values are obtained or follow Practice D 3324.

9. Procedure

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

9.2 Weigh 25 ± 0.1 g of the sample.

9.3 Compress the sample using either the Chandler or Titan press.

9.4 *Chandler Press*

9.4.1 Place the bottom seal plate and the compression cylinder in the hydraulic press. Move the handle of the seal plate to check its position in the support plate. Rotate the cylinder to be certain that it fits on the seal plate.

9.4.2 Place the carbon black sample in the compression cylinder and insert the piston with the nylon spacer next to the carbon black. Rotate the piston while pressing it into the cylinder as far as possible by hand.

9.4.3 Adjust the alignment of the piston, cylinder, and ram to prevent galling of the cylinder.

9.4.4 Compress the carbon black to approximately 165 MPa (24 000 psi), hold for about 1 s, then release. The exact pressure is determined by measuring the compressed oil. A value of the SRB materials and making appropriate adjustments. If the values are too high, the pressure is increased and pressure is lowered if the values are too low.

NOTE 2—165 MPa (24 000 psi) is equivalent to 131 kN (29 450 lbf) on the Enerpac gage GF-20S.

9.4.5 Raise the ram to a sufficient height to allow the bottom seal plate to be removed, then lower the ram in order to press the piston and sample through the cylinder and into a sieve screen fitted with a receiver pan.

9.4.6 Wipe the piston, cylinder, and seal in order to remove carbon black dust and reassemble the apparatus as described in 9.4.1.

9.4.7 Pass the compressed carbon black through the sieve screen into the receiver.

9.4.8 Repeat 9.4.2-9.4.7, compressing the sample a total of four times. Retain the sample from 9.4.7 after the fourth compression. Proceed to 9.6.

9.5 *Titan Press*

9.5.1 Lower the cylinder piston by pressing the left hand lever downward, then pour the carbon black sample into the cylinder.

9.5.2 Close and latch the door of the press. Compress the sample by operating the ram using a downward movement of the right hand lever, until the preset gauge pressure reaches approximately 11 MPa (1550 psi). Release immediately. The exact pressure is determined by measuring the compressed oilA value of the SRB materials and making appropriate adjustments. If the values are too high, the pressure is increased and pressure is lowered if the values are too low.

9.5.3 Raise the ram until it is level with the top of the conical collar placed on top of the cylinder.

9.5.4 Raise the cylinder piston until the compressed sample is broken by contact with the raised ram. The conical collar will retain the sample.

9.5.5 Break up the sample with a spatula, lower the cylinder piston, and allow the sample to fall back into cylinder. If necessary, brush the inside of the collar to return all of the carbon black to the cylinder.

9.5.6 Repeat steps 9.5.2-9.5.5 an additional three times, for a total of four compression cycles.

9.5.7 Remove the sample and pass it through a 850 µm sieve (sieve #20).

9.6 Weigh 20 ± 0.01g of the compressed sample into the mixing bowl of the absorptometer and measure the oil absorption value in accordance with Test Method D 2414.

NOTE 3—If the compressed sample is not to be tested within 15 min after compression, it should be stored in a desiccator or dried for 1 hour in the specified oven set at 125°C prior to testing.

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[6] 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.

[7] *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.

[8] Three paraffin oils have been found suitable including Marcol 82 and Marcol 9 from Exxon and Sunpar LW107 from SUNOCO.

[9] F-5 will be used until depleted, at which time F-6 will be used. Available from Laboratory Standards and Technologies, 227 Somerset St., Borger, TX 79007.

10. Calculation

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

$$\textit{Oil}\ \text{absorption number, compressed sample, } 10^{-5}m^3/kg = \frac{A}{B} \times 100 \quad (1)$$

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

11.1.3 Oil (DBP or paraffin), and

11.1.4 Method for end-point determination (Procedure A, B or C in D 2414, Section 8.3).

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.

TABLE 1 Precision Parameters for D 3493 Oil Absorption Number of Compressed Sample, (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)	13	60.2	0.53	1.51	1.23	3.48
SRB C6 (N326)	13	68.1	0.53	1.51	1.04	2.96
SRB E6 (N660)	13	76.0	0.83	2.34	1.95	5.51
SRB F6 (N683)	13	88.6	0.86	2.42	1.53	4.34
SRB B6 (N220)	15	98.5	0.60	1.71	1.36	3.86
SRB A6 (N134)	15	101.0	0.82	2.33	1.24	3.50
Average		82.1				
Pooled Values			0.71	2.01	1.42	4.03

[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.

12.3 A type 1 inter-laboratory 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 material 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 2.01 $10^{-5}m^3/kg$ ($cm^3/100$ 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 4—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 4.03 $10^{-5}m^3/kg$ ($cm^3/100$ 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