Designation: D3493 – 16

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

This standard is issued under the fixed designation D3493; 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]

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

D2414 Test Method for Carbon Black—Oil Absorption Number (OAN)

D4821 Guide for Carbon Black—Validation of Test Method Precision and Bias

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing 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, paraffin, or EFA 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, paraffin, or EFA oil is acceptable for use with many standard pelleted grades of N-series carbon blacks found in Classification D1765. COAN testing using paraffin or EFA oil on some specialty blacks and powder blacks may result in significant differences when compared to COAN testing using DBP oil. Referee testing between suppliers and users should use DBP oil until such time that precision data is available for paraffin and EFA oils.

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[3]

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

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

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

5.3 *Carbon Black Press,* capable of compressing a 25-g sample to 165 MPa (24 000 psi).[4]

5.4 *Absorptometer,*[5] equipped with a constant-rate buret that 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.[6] 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.[7]

6.4 *Epoxidized Fatty Acid Ester (EFA),* meeting the specifications listed in Test Method D2414, Table 1. 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.[8]

7. Sampling

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

8. Calibration and Standardization

8.1 See Test Method D2414.

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 Guide D4821.

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 a sample mass of either 25, 30, or 45 g depending on the sample mass requirement for oil absorption per Test Method D2414. The desired sample mass to be compressed will weigh 5 ± 0.1 g greater than the mass specified in subsection 9.2 of Test Method D2414.

9.3 Compress the sample using either the Chandler, Titan, or ISTCO 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 if the values are too low, the pressure is lowered.

NOTE 2—165 MPa (24 000 psi) is equivalent to 131 kN (29 450 lbf) on the Enerpac gauge 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.

[4] The carbon black press is available from two suppliers: Jaron Technologies, LLC, 2338 Duncan St., Pampa, TX 79065, http://jarontech.com/. ISTCO Engineering Works, K-1. 884/10 & 885/A, GIDC Industrial Estate, Makarpura, Vadodara-390010, Gujarat, India, http://www.indiamart.com/istcoengineering/hydraulic-testingmachine.html. 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.

[5] 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. The sole source of supply of the apparatus known to the committee at this time is listed above. 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.

[6] *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.

[7] The sole source of supply of paraffin oil (Marcol 82, which has been demonstrated to provide comparable results to DBP oil) known to the committee at this time is Exxon. 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] The sole source of supply of the ASTM standard reference blacks known to the committee at this time is Laboratory Standards and Technologies, 227 Somerset, Borger, TX 79007, http://www.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,[1] which you may attend.

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 9.7.

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 oil absorption of the compressed SRB materials and making appropriate adjustments. If the values are too high, the pressure is increased, and if the values are too low, the pressure is lowered.

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 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). Retain the sample from 9.5.7 after the fourth compression. Proceed to 9.7.

9.6 *ISTCO Press:*

9.6.1 Lower the ejector piston by pushing the appropriate button and pour the carbon black sample into the cylinder.

9.6.2 Close and latch the door of the press. Compress the sample by pushing the appropriate button to initiate the compression.

9.6.3 Raise the ejector piston by pushing the appropriate button to raise the compressed sample.

9.6.4 Break up the sample with a spatula, lower the ejector 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.

9.6.5 Repeat 9.6.2 – 9.6.4 an additional three times, for a total of four compression cycles.

9.6.6 Remove the sample and pass it through a 850-µm sieve (ASTM 20-mesh sieve). Retain the sample from 9.6.6 after the fourth compression. Proceed to 9.7.

9.7 Measure the oil absorption number of the compressed sample in accordance with Test Method D2414. Use the same sample masses as recommended in subsection 9.2 of Test Method D2414.

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 h in the specified oven set at 125°C prior to testing.

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:

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

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 on Standardization in Test Method D2414).

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 Interlaboratory precision program (ITP) information was conducted as detailed in Table 1. Both repeatability and

TABLE 1 SRB8 ITP Information

SRB8 Material	Grade	Producer	Test Period	Number of Labs (M/H/L) D3493
SRB-8A	N326	Continental	March 2008	64 (1/0/0)
SRB-8A2	N326	Continental	March 2013	72 (0/1/1)
SRB-8B	N134	Cabot	June 2009	66 (0/0/0)
SRB-8B2	N134	Cabot	March/April 2014	40 (3/4/3)
SRB-8C	HS Tread	Columbian	September 2010	66 (2/1/0)
SRB-8D	LS Carcass	Cabot	March 2009	67 (0/2/0)
SRB-8E	N660	Orion	September 2008	57 (1/0/0)
SRB-8F	N683	Orion	March 2010	67 (1/1/0)
SRB-8F2	N683	Orion	March 2015	62 (1/0/0)
SRB-8G[A]	N990	Cancarb	Last half of 1996	Unknown

[A] SRB-8G was produced and approved in the last half of 1996 as SRB-5G and has continued to be included in the current SRB sets since that time. At the time it was produced and approved it was D24's practice to only publish the within-laboratory standard deviation, Sr, and associated limits. The between-laboratory standard deviation, SR, was never published and since the data is no longer available it is not possible to calculate or publish the SR values and corresponding limits. The SRB G material was only tested for NSA, STSA, and OAN per the test method version available in 1996.

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*.

12.3 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 programs described in 12.2. 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 2.

12.4 The results of the precision calculations for this test are given in Table 2. The materials are arranged in ascending "mean level" order.

12.5 *Repeatability*—The **pooled relative** repeatability, *(r)*, of this test has been established as 1.4 %. Any other value in Table 2 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 2 must be suspected of being from different populations and some appropriate action taken.

TABLE 2 Precision Parameters for Test Method D3493, COAN Method (Type 1 Precision)[A]

Units	10^{-5} m^3/kg ($cm^3/100$ g)						
Material	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SRB-8C	130.6	0.54	1.52	**1.2**	1.47	4.17	**3.2**
SRB-8B2	103.1	0.50	1.42	**1.4**	1.03	2.92	**2.8**
SRB-8B	99.4	0.47	1.32	**1.3**	1.03	2.91	**2.9**
SRB-8A2	67.5	0.35	0.98	**1.5**	1.08	3.05	**4.5**
SRB-8A	66.7	0.42	1.20	**1.8**	0.87	2.46	**3.7**
SRB-8F	88.6	0.40	1.12	**1.3**	0.91	2.58	**2.9**
SRB-8E	74.7	0.36	1.01	**1.3**	0.99	2.82	**3.8**
SRB-8D	36.9	0.26	0.74	**1.9**	0.96	2.72	**7.1**
Average	83.7						
Pooled Values		0.42	1.19	**1.4**	1.06	2.99	**3.6**

[A] The preferred precision values are shown in bold text.

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 relative** reproducibility, *(R)*, of this test method has been established as 3.6 %. Any other value in Table 2 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 2 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