Designation: D3493 – 21

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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 oil, 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 oil, 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 oil 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).

5.4 *Absorptometer,* 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 *Tablespoon,* stainless steel.

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

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

5.9 *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.[4] 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.[5]

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.[6]

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 10.2 of Test Method D2414.

9.3 Compress the sample using a carbon black press per the manufacturer instructions.

9.4 A general procedure for compressing carbon black involves the following steps:

9.4.1 Ensure the cylinder and piston tips are clean.

9.4.2 Open the press by moving the pistons is a proper position to add carbon black.

9.4.3 Add the desired amount of carbon black to the cylinder using a funnel.

9.4.4 Compress the carbon black to approximately 165 MPa (24 000 psi), hold for about 1 s, then release.

9.4.5 Open the press to release the carbon black in the funnel.

9.4.6 Break up the carbon black using a spatula.

9.4.7 Return the sample to the cylinder.

9.4.8 Repeat 9.4.4 – 9.4.7 three additional times for a total of four compressions.

9.4.9 Open the press to release the sample from the cylinder.

9.4.10 Thoroughly break up the plug of carbon black after the 4th compression to a lump free powder. This is achieved by transferring the compressed carbon black to a clean container of suitable size, or an 850-µm sieve (sieve #20), then manually working the compressed carbon black using a spoon or a spatula until it's visually lump free, or until all the material has passed through the sieve screen.

NOTE 2—For samples where lumps remain and cannot be broken up to a visually lump free material, it is recommended to pass the carbon black sample through a clean 850-µm sieve (sieve #20).

9.4.11 Retain the sample from 9.4.10 for testing. Proceed to 9.5.

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.

9.5 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.

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:

[4] *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.

[5] 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.

[6] 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.

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

where:

A = volume of oil used, cm^3,
B = mass of tested sample, g,
C = slope of normalization curve (see Test Method D2414 Section 9), and
D = y-intercept of normalization curve (see Test Method D2414 Section 9).

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 Results obtained with DBP, paraffin oil or EFA oil, 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 D4483-99. 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 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 1.

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 repeatability**, r, of this test has been established as 1.15 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 reproducibility**, R, of this test method has been established as 3.31 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.

TABLE 1 Precision Parameters for Test Method D3493, Method N/A (Type 1 Precision)[A]

		Units		10^{-5} m^3/kg ($cm^3/100$ g)					
Material	Period	Number of Laboratories (M/H/L)[B]	Mean Level	Sr	**r**	(r)	SR	**R**	(R)
SRB-9A	Mar 2013	70(0/1/0)	67.5	0.35	**1.00**	1.5	1.10	**3.12**	4.6
SRB-9B	Mar 2016	75(0/2/0)	90.1	0.45	**1.27**	1.4	1.24	**3.52**	3.9
SRB-9D	Mar 2018	67(1/1/0)	34.6	0.21	**0.61**	1.8	0.84	**2.37**	6.9
SRB-9E	Aug 2016	71(0/3/1)	73.2	0.43	**1.22**	1.7	0.91	**2.58**	3.5
SRB-9F	Mar 2015	67(1/1/0)	85.4	0.34	**0.97**	1.1	1.19	**3.37**	3.9
SRB-9G	Aug 2017	64(1/2/1)	38.0	0.24	**0.67**	1.8	1.27	**3.59**	9.4
SRB-9B2	Mar 2019	68(0/2/0)	90.1	0.46	**1.30**	1.4	1.23	**3.47**	3.9
SRB-9C	Aug 2019	68(1/2/0)	102.5	0.46	**1.30**	1.3	1.07	**3.03**	3.0
SRB-9H	Mar/Apr 2020	65(0/1/0)	135.9	0.57	**1.62**	1.2	1.68	**4.77**	3.5
Average			78.6						
Pooled Values				0.41	**1.15**	1.5	1.17	**3.31**	4.2

[A] Preferred precision shown in bold text.
[B] M = number of outliers for Mean; H = number of outliers for High variation; L = number of outliers for Low variation.