Designation: D6854 – 15a

Standard Test Method for Silica—Oil Absorption Number (OAN)[1]

This standard is issued under the fixed designation D6854; 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 (OAN) of silica.

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]

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)
D6738 Test Method for Precipitated Silica—Volatile Content
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 In this test method, oil is added by means of a constant-rate burette to a sample of silica 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. The test is stopped when a torque level has been reached. Preferably the torque versus volume of oil is recorded by a penwriter or by a data acquisition system allowing a reliable determination of the endpoint. The volume of oil per unit mass of silica is the oil absorption number (OAN).

4. Significance and Use

4.1 The oil absorption number of a specific silica is related to the processing and vulcanizate properties of rubber compounds containing the silica.

5. Apparatus[3]

5.1 *Balance,* analytical, with a sensitivity of 0.001 g.

5.2 *Sieve,* 500 µm (U.S. standard No. 35), having a diameter of 200 mm (8 in.) and a height of 25 mm (1 in.).

5.3 *Bottom Receiver Pan.*

5.4 *Oven,* gravity-convection type, capable of temperature regulation within ±1°C at 105°C and temperature uniformity within ±5°C.

5.5 *Spatula,* rubber, 100-mm.

5.6 *Absorptometer,*[4] equipped with a constant-rate burette that delivers 4 ± 0.024 cm^3/min.

5.7 *Desiccator,* with silica gel as desiccant.

6. Reagents and Standards

6.1 *Oil:*

6.1.1 *n-Dibutyl Phthalate,*[5] having a density of 1.042 to 1.047 mg/m^3 (g/cm^3) at 25°C.

6.1.2 *di-octyl-adipate (DOA),* having a density of 0.9255 g/cm^3 at 20°C, a refractive index of 1.447 at 20 °C, and a kinematic viscosity of 10 to 34 mm^2/s (cSt) at 40°C.

6.1.3 *Epoxidized fatty acid ester (EFA),* meeting the specifications listed in Test Method D2414, Annex A4.

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[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.20 on Compounding Materials and Procedures.

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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] 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., Hackensack, NJ 07606, website: www.cwbrabender.com, and HITEC Luxembourg, 5 rue de lEglise, L-1458 Luxembourg, website: www.hitec.lu.

[5] Technical grade has turned out to be suitable for the test, provided that the density is in the specified range.

6.2 *Silica,* commercial grade with a nitrogen surface area of 175 ± 10 m^2/g.

7. Sampling

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

8. Calibration

8.1 *Absorptometer*[6]—The absorptometer is composed of components that influence calibration: the dynamometer torque spring or the load cell, the torque-limit or the indicator set point, the oil damper (absorptometers Type C, E, and H are equipped with electronic damping), and the mixer-measuring head.[7] It is necessary that each of the components be in good condition or proper adjustment to achieve acceptable calibration.

NOTE 1—Stainless steel mixing chambers[8] have been found satisfactory for this test when they are manufactured to a roughness average (Ra) of 2.5 ± 0.4 µm (100 ± 15 µin.) based upon eight 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 to minimize the bowl surface effects on calibration (see Annex A1).

8.1.1 The torque indicator is the primary component used to correct calibration. The load cell tension is adjusted by varying the alarm shut-off set point. Proper adjustment on the torque indicator should provide repeatable values for a silica sample dedicated to internal reference.

8.1.2 The maximum torque span is set at 10 000 mNm (10 000 units) torque value. The torque-limit alarm is initially set at 5000 mNm (5000 units), but for testing silicas it will be necessary to adjust this setting to a lower value in order to obtain reproducible results. Use an internal silica sample with a nitrogen surface area of 175 ± 10 m^2 /g to set the torque limit alarm which should correspond to approximately 70 % of the maximum torque developed during the test. After calibration, this setting should not be changed.

NOTE 2—It is generally recommended to use the absorptometer in conjunction with a penwriter or preferably with a data acquisition system (see 9.10 for further details).

8.1.3 All digital signals are preset at 3 s damping for the torque sensing system.

8.1.4 Properly maintain the surface finish of the mixing chamber. If a new mixer chamber is installed, frequently monitor the instrument for any drift in calibration.

8.2 *Constant-Rate Burette*—The delivery rate of the burette is to be 4 cm^3/min. See Annex A1 for detailed instructions on the procedure for calibration check of the constant-rate burette.

9. Procedure

9.1 Pass a suitable amount of the sample through Sieve No. 35 (500 µm), using a brush in order to deagglomerate larger particles. Use 2 g of the sieved material to test the moisture content (see 9.3) as volatile matter according to Test Method D6738.

9.2 Determine the amount of moisture in the silica under test by weighing 2 g of the sieved silica (see 9.1) into a dish to the nearest 0.001 g. Place the dish into an oven set at 105°C, leave it inside for 2 h, cool in a desiccator and weigh to the nearest 0.001 g. See Section 10 (Calculation) for details of moisture calculation.

9.3 Weigh 12.5 g of the sample to the nearest 0.01 g.

NOTE 3—For silicas with an extraordinary high pour density it may be necessary to increase the sample mass used for the test. This modification has to be mentioned in the test report.

9.4 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 4 while running the samples.

NOTE 4—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.

9.5 Transfer the sample to the absorptometer mixer chamber and replace the cover.

9.6 Place a waste receptacle under the delivery tube. Make sure that the tube is free of air bubbles by delivering approximately 1 cm^3 of oil into the waste receptacle.

9.7 Verify the drive speed is set to 1.31 rad/s (125 r/min).

9.8 Position the burette delivery tube over the hole in the mixer chamber cover or use the accessory funnel. Set the burette digital counter to zero.

9.9 Activate the "start" buttons simultaneously or use the start procedure given in the software. The apparatus will operate until sufficient torque has developed to activate the torque-limit switch, which will halt the absorptometer and burette.

9.10 Record the volume of oil used as indicated by the burette digital counter.

NOTE 5—If a penwriter is used to record the torque curve, deactivate the automatic cut-off by setting the torque limit to 10 000. Stop the test when the torque maximum has been recorded unequivocally. Mark on the curve the oil volume corresponding to the maximum torque and measure the height (in mm or in.) of the maximum. At the left side of the maximum, identify the point corresponding to a height of 70 % of the maximum of the curve. Measure the distance on the *x*-axis from the start point to this point and convert the value to volume of oil as follows:

$$\text{Volume oil} = \text{delivery rate of burette} \cdot \text{distance} \,/\, \text{speed of penwriter}$$

NOTE 6—If a data acquisition system[9] is used, the absorptometer will stop after having recorded the torque maximum, and the test result (in

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[6] Mechanical absorptometers (type A or type B) can be used for the test; however, they are no longer commercially available. Refer to the instructions of the supplier for calibration procedure.

[7] The rotor motor speed is 1.31 rad/s (125 r/min).

[8] Replacement stainless steel bowls which have been found to be satisfactory are available from Titan Specialties, Inc., P.O. Box 2316, Pampa, TX 79066-2316, and C. W. Brabender Instruments, Inc., 50 E. Wesley St., S. Hackensack, NJ 07606, website: www.cwbrabender.com, HITEC Luxembourg, 5 rue de lEglise, L-1458 Luxembourg, website: www.hitec.lu, and Titan Specialties, Inc. P.O. Box 2316, Pampa, TX 79066–2316.

[9] OAN Data Acquisition Systems are available from C.W. Brabender Instruments, Inc., 50 E. Wesley St., S. Hackensack, NJ 07606, website: www.cwbrabender.com, and HITEC Luxembourg, 5 rue de lEglise, L-1458 Luxembourg, website: www.hitec.lu.

TABLE 1 Absorption Number (mL – 100g)

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	s_R	r	R
Silica A	207.18	0.81	2.48	2.26	6.94
Silica B	230.40	1.81	5.64	5.07	15.78

[A] The average of the laboratories' calculated averages.

cm^3/100 g) will be reported automatically.

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

10. Calculation

10.1 Calculate the moisture content as follows:

$$\text{Moisture, } \% = 100 \cdot (m_0 - m)/m_0 \quad (1)$$

where:

m_0 = mass of the silica before drying, g, and
m = mass of the silica after drying, g.

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

$$\text{OAN}, 10^{-5}\text{m}^3/\text{kg} = \quad (2)$$

$$A/B \cdot (100/(100 - \text{Moisture})) \cdot 100$$

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, and of the oil used;

11.1.2 Sample mass, if different than shown in 9.5; and

11.1.3 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[10]

12.1 The precision of this test method is based on an interlaboratory study conducted in 2010. Five laboratories tested two types of silica samples. Every "test result" represents an individual determination. Each laboratory was instructed to report four replicate test results for each material. Except for the limited number of participating laboratories, Practice E691 was followed for the design and analysis of the data.

12.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

12.1.1.1 Repeatability limits are listed in Table 1.

12.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

12.1.2.1 Reproducibility limits are listed in Table 1.

12.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

12.1.4 Any judgment in accordance with statements 12.1.1 and 12.1.2 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of laboratories reporting replicate results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Consider the repeatability limit and the reproducibility limit as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected.

12.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

12.3 The precision statement was determined through statistical examination of 40 results, from five laboratories, on two different precipitated silica samples using *n*-dibutyl phthalate as oil.

13. Keywords

13.1 di-octyl-adipate; epoxidized fatty acid ester; *n*-dibutyl phthalate; *n*-dibutyl phthalate absorption number; silica, oil absorption number

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[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1109.

ANNEX

(Mandatory Information)

A1. CALIBRATION CHECK OF CONSTANT-RATE BURETTE

A1.1 Scope

A1.1.1 The constant-rate burette is an integral part of the absorption measuring system. Failure of the burette to deliver the deliver the specified amount of reagent to the silica will result in erroneous absorption readings. This annex provides a method for checking the delivery rate of the constant-rate burette. One of the reasons for the incorrect absorption values (caused by incorrect reagent delivery by the automatic burette) 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 *Stopwatch.*

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 burette and delivery tubes with *n*-dibutyl phthalate. Ensure that all air is removed from the system.

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

A1.3.4 Stop the burette 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 burette and stopwatch.

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

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

A1.3.9 Refill the burette.

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 DBP from the delivered mass and density as follows:

$$\text{Delivery, cm}^3 = \text{mass delivered/DBP density} \tag{A1.1}$$

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	±0.05
8	±0.05