



Standard Test Method for Silica—*n*-Dibutyl Phthalate Absorption Number¹

This standard is issued under the fixed designation D 6854; 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 *n*-dibutyl phthalate (DBP) absorption number 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:

D 1799 Practice for Carbon Black—Sampling Packaged Shipments²

D 1900 Practice for Carbon Black—Sampling Bulk Shipments²

D 2414 Test Method for Carbon Black—Oil Absorption Number²

3. Summary of Test Method

3.1 In this test method, DBP is added by means of a constant-rate buret to a sample of silica in the mixer chamber of an absorptometer. As the sample absorbs the DBP, the mixture changes from a free-flowing state to one of a semi-plastic 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 predetermined torque level has been reached. Preferably the torque versus volume of DBP is recorded by a penwriter or by a data acquisition system allowing a reliable determination of the endpoint. The volume of DBP per unit mass of silica is the DBP absorption number.

4. Significance and Use

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

5. Apparatus³

5.1 *Balance*, analytical, with 0.001-g sensitivity.

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

5.3 *Bottom Receiver Pan*.

5.4 *Oven*, gravity-convection type, capable of temperature regulation within ±1°C at 125°C and temperature uniformity within ±5°C.

5.5 *Spatula*, rubber, 100-mm.

5.6 *Absorptometer*,⁴ equipped with a constant-rate buret that delivers 4 ± 0.024 cm³/min.

5.7 *Desiccator*, with silica gel as desiccant.

6. Reagents and Standards

6.1 *n*-Dibutyl Phthalate,⁵ having a density of 1.042 to 1.047 mg/m³ (g/cm³) at 25°C.

6.2 *Silica*, commercial grade with a nitrogen surface area of 175 ± 10 m²/g.

7. Sampling

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

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 E and Type 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.

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 l'Eglise, 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 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).

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 *Annual Book of ASTM Standards*, Vol 09.01.