Designation: D7854 – 18

Standard Test Method for Carbon Black-Void Volume at Mean Pressure[1]

This standard is issued under the fixed designation D7854; 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 a procedure to measure a carbon black structure property by Void Volume at mean pressure. Compressed void volumes are obtained by measuring the compressed volume of a weighed sample in a cylindrical chamber as a function of pressure exerted by a movable piston. A profile of void volume as a function of pressure provides a means to assess carbon black structure at varying levels of density and aggregate reduction. For the purposes of standardized testing a single value of void volume is reported at 50 MPa mean pressure.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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]

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)

D3493 Test Method for Carbon Black—Oil Absorption Number of Compressed Sample (COAN)

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Terminology

3.1 Refer to Sections 4 and 9 for a more complete understanding of the use of these terms in this test method.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *applied pressure, n*—the pressure exerted on a sample mass by a movable piston in a cylindrical chamber, where the load cell or force measuring system is in contact with the movable piston.

3.2.2 *transmitted pressure, n*—the resulting pressure transmitted through a sample in a cylindrical chamber, where the load cell or force measuring system is in contact with the sample opposite the movable piston, typically via a stationary second piston.

3.2.3 *compressed volume (carbon black), n*—the apparent volume that a specified mass of carbon black occupies when it is contained in a specified cylindrical chamber and subjected to a single uniaxial compression at a specified pressure by means of a movable piston.

3.2.4 *geometric mean pressure, n*—the geometric mean of the applied and transmitted pressures at a specific void volume; the geometric mean pressure is defined in Eq 1:

$$Geometric\ Mean\ P_{GM} = (P_a \times P_t)^{0.5} \quad (1)$$

3.2.5 *theoretical volume (carbon black), n*—the volume that a specific mass of carbon black would occupy if there were no void space within the carbon black, and is given by the ratio of mass to skeletal density, where the skeletal density is determined by an accepted test method.

3.2.6 *void volume (carbon black), n*—a measure of the intra-aggregate void space or occluded volume within the primary structure of carbon black, characterized by the irregularity and non-sphericity of carbon black aggregate particles, and expressed as the difference (compressed volume minus theoretical volume) as a function of specified uniaxial compression pressure, and normalized to 100 g mass. pressure, and normalized to 100 g mass. The void volume of a 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.

expressed as a function of geometric mean pressure, VV, is a carbon black structure property.

3.2.6.1 *Discussion*—Carbon blacks resist packing, compression, and fracture due to aggregate irregularities and entanglements, size distribution, and aggregate strength resulting from particle to-particle necks within aggregate branches. Compressed void volume is also affected by reacting forces to the cylinder wall and the piston tip, which in turn depend on factors including sample shape (that is, the ratio of sample height to cylinder diameter) or interfacial area, which can influence the uniformity of the compaction density. Since compressed void volumes as a function of applied pressure are known to be specific to sample mass and cylinder geometry, such a compressed void volume is biased due to error in the applied pressure relationship. The applied pressure bias is a result of force losses due to friction between the sample and cylinder wall interface. There is presently no known technique to properly correct applied pressure measurements for an instrument design using a single load cell since friction coefficients (μ) are not constant for carbon black products or applied pressures. For this reason, the most useful technique for comparing compressed void volumes is based on a numerical technique known as mean compaction force or mean pressure. The mean pressure technique requires an instrument design consisting of two load cells to enable the measurement of compressed void volume as a function of applied and transmitted force or pressure. Such a design allows the computation of void volumes at mean pressures, a method which has been demonstrated to minimize the effects of carbon black sample mass and cylinder geometry.

4. Summary of Test Method

4.1 The measured compressed volume (apparent volume) of a weighed dry test sample is obtained in a void volume instrument as a function of specified pressure. The instrument consists of an apparatus which can apply uniaxial compression to a test sample in a cylindrical sample chamber where applied and transmitted forces (or pressures) are measured. The compressed void volume is obtained by subtracting the theoretical volume from the apparent volume, then expressing the result unitized to 100 g mass, as a function of specified mean pressure.

5. Significance and Use

5.1 The greater a carbon black resists compression by having substantial aggregate irregularity and non-sphericity, the greater the compressed volume and void volume. Also, the more that a carbon black resists compression, the greater the energy required to compress the sample per unit void volume.

5.2 Structure is a property that strongly influences the physical properties developed in carbon black-elastomer compounds for use in tires, mechanical rubber goods, and other manufactured rubber products. Structure by void volume is based on compression while structure measurements by OAN (Test Method D2414) and COAN (Test Method D3493) are based on oil absorption.

6. Apparatus

6.1 *Analytical Balance,* or equivalent, capable of a weighing sensitivity of 0.1 mg.

6.2 *Gravity Convection Drying, Oven,* capable of maintaining 125 ± 5°C.

6.3 *Weighing Dish, Camel Hair or Similar Brush,* to be used for weighing and transferring samples.

6.4 *Void Volume Instrument,* to be used to measure the compressed volume (apparent volume) of carbon blacks as a function of applied pressure, from which the void volume is calculated at specified intervals of geometric mean pressure (that is, geometric mean of applied and transmitted pressures). The void volume instrument or device shall conform to the following generic specifications and be capable of operating as outlined in Section 9.

6.4.1 The instrument shall have a rigid framework that contains a cylindrical sample chamber. Hysteresis and elasticity in the framework under the range of applied forces should be accounted for in the displacement measurement.

6.4.2 The cylinder shall have a uniform diameter.

6.4.3 By means of a suitable mechanism with sufficient power for the compression forces as required for testing, the piston shall be capable of being moved to compress the sample. A device to record the movement of the piston and measure displacement shall be provided. The compressed volume of any sample is determined by the distance from the end of the piston to the end of the cylinder; this is designated as a "height" in the calculations discussed in Section 10. The sample height and cylinder diameter are used to calculate an apparent sample volume.

6.4.4 Load cells or other suitable force or pressure measurement devices are used to measure the applied and transmitted pressures.

6.4.5 The instrument design shall provide continuous compression at a controlled and constant rate thereby allowing continuous measurements of apparent volume and pressures at specified data intervals.

6.4.6 The instrument uses an electric motor or hydraulic fluid to operate a linear actuator attached to a piston.

6.4.7 The instrument incorporates two load cells to directly measure applied and transmitted forces or pressures.

6.4.8 The instrument design shall provide a means to save and store the compression data up to a minimum of 50 MPa geometric mean pressure for subsequent analysis.

7. Sampling

7.1 Samples of candidate carbon blacks shall be taken in accordance with Practice D1799 or D1900.

8. Calibration

8.1 *Calibration*—The manufacturer will typically calibrate the instrument measurement systems. The height measurement system is typically calibrated using a physical standard such as a calibrated steel plug. Load cells are typically calibrated or verified using a reference load cell. Traceability is recommended for all calibration devices. Follow the manufacturer's recommendations for calibration frequency and verification.