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StandardTest Method for Carbon Black—Void Volume (VV)[1]

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1. Scope

1.1 This test method covers a procedure to measure a carbon black structure property known as Void Volume. Compressed void volumes are obtained by measuring the compressed volume of a weighed sample as a function of applied pressure in a cylindrical chamber by a movable piston with a displacement transducer on the piston mechanism. A profile of void volume as a function of applied pressure provides a means to assess carbon black structure at varying levels of density and aggregate reduction.

1.2 Void volume is an important carbon black structure property that relates to the compounded physical properties for carbon black-filled elastomers including viscosity, modulus, and die swell.

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

1.4 *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)

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

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard*—Refer to Sections 4 and 9 for a more complete understanding of the use of these terms in this test method.

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

3.3 *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.

3.4 *void volume (carbon black), n*—a measure of the occluded pore 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 applied pressure, and normalized to 100 g mass.

3.4.1 *Discussion*—Carbon blacks resist packing, compression, and fracture due to aggregate irregularities and entanglements, size distribution, and aggregate strength or particle-to-particle necks within aggregate branches. Carbon black 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 the compressed void volumes are specific to the cylinder geometry and possibly to the cylinder wall surface (that is, friction effects), a measured compressed volume is not a true compressed volume unless these factors are corrected or sufficiently minimized.

4. Summary of Test Method

4.1 The measured compressed volume (apparent volume) of a weighed dry test sample as a function of applied pressure is obtained in a void volume instrument appropriately calibrated by the manufacturer or user. From the measured compressed

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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.

volume, the measured void volume is obtained by subtracting the theoretical volume from the apparent volume then expressing the result normalized to 100 g mass. A true void volume is obtained by correcting the measured void volume for instrument geometry, sample mass, and possible friction effects.

5. Significance and Use

5.1 The void volume of a carbon black expressed as a function of applied pressure, VV, is a carbon black structure property. Structure is a generic term that is a function of the shape irregularity and deviation from sphericity of carbon black aggregates. The greater a carbon black resists compression by having substantial aggregate irregularity and nonsphericity, 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, traditionally measured by OAN (Test Method D2414) and COAN (Test Method D3493), 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. Several studies within D24 have demonstrated that void volume data can be used to estimate OAN and COAN numbers of carbon blacks using mathematical models derived from void volume-pressure data and oil absorption data. The models may vary depending on whether dynamic or static void volume measurements are used and the number and types of carbon blacks included within a modeling data set. If necessary, OAN and COAN estimates from void volume models can also be normalized using current SRBs (as practiced in Guide D4821). Any estimates of OAN or COAN derived from prediction models using void volume-pressure data should be labeled appropriately (that is, Test Method D6086) to avoid confusion with OAN or COAN data obtained directly from oil absorption methods.

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 the 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. The void volume instrument or device shall conform to the following generic specifications and be capable of operating as outlined in 6.4.1-6.4.3.

6.4.1 The instrument shall have a rigid framework that contains a cylindrical sample chamber (see an example in Fig. X1.1). Hysteresis 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 procedure discussed in Section 9. The sample height and cylinder diameter are used to calculate an apparent sample volume.

6.4.4 A load cell or other suitable force or pressure measurement device is used to measure the pressure applied to the sample.

6.4.5 Two types of void volume instruments are commercially available:

6.4.5.1 A static or equilibrium measurement instrument which is designed to target one or more target pressures; an option to step from one target pressure to another is available allowing the collection of several data points. This type instrument uses an air-powered control cylinder to move the piston and does not control the rate of piston movement. The pressure applied to the sample is not directly measured, but calculated based on air pressure applied to the control cylinder. See Appendix X1 for a brief description of a commercial void volume instrument that meets these specifications.

6.4.5.2 A dynamic void volume instrument is also available which is designed to dynamically scan a pressure range at a controlled rate thereby allowing continuous measurements of apparent volume and pressure at specified data intervals. The dynamic instrument uses an electric motor to operate a linear actuator attached to the piston. The instrument incorporates a load cell to directly measure force or pressure applied to the sample. See Appendix X2 for a brief description of a commercial void volume instrument that meets these specifications.

7. Sampling

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

8. Calibration and Normalization

8.1 *Calibration*—Follow the manufacturer's instructions to set up the instrument and to calibrate the measurement systems. The use of a physical standard such as a calibrated steel plug with traceability is recommended to calibrate or verify the height displacement transducer. A reference load cell with traceability is recommended to calibrate or verify the force transducer.

8.2 *Normalization (non-mandatory; user should follow recommendations from manufacturer):*

8.2.1 Test the 6 current ASTM Standard Reference Blacks (SRBs) four times each to establish the average measured value for each SRB over the range of compression pressures of interest. Additional values are added periodically.

8.2.2 Perform a regression analysis using target SRB values (y value) and the rolling average of the last 4 measured values (x value) across the range of applied pressures specified by the manufacturer. If target SRB curves represent measured void