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

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1. Scope

~~1.1This test method covers a procedure to measure a carbon black property called Void Volume. The void volume is obtained by measuring the compressed volume of a weighed sample contained in a cylindrical chamber when a specified compression force is applied by a movable piston with a displacement transducer on the piston mechanism.~~

~~1.2Void volume is an important carbon black property that relates to the compounded physical properties for carbon black-filled elastomers.~~

~~1.3The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.~~

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]

D 1799 Practice for Carbon Black—Sampling Packaged Shipments

D 1900~~Practice for Carbon Black—Sampling Bulk Shipments~~ Practice for Carbon BlackSampling Bulk Shipments

D 2414 Test Method for Carbon BlackOil Absorption Number (OAN)

D 3493 Test Method for Carbon BlackOil Absorption Number of Compressed Sample (COAN)

D 4821 Guide for Carbon BlackValidation 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.

3.1.1 *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 ~~compression force~~applied pressure by means of a movable piston.

3.1.2 *theoretical volume (carbon black), n*— ~~the volume that a specific mass of carbon black particles with no voids, for practical purposes, is given by the ratio of the mass to the density, when the density is determined by an accepted test.~~ — 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.

3.1.3 *void volume (carbon black), n*—~~a measure of the irregularity and non-sphericity of carbon black aggregate particles, it is expressed as the difference, (compressed volume-theoretical volume) for a mass of carbon black, the differential volume is normalized to a selected unit mass.~~ —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.

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[1] This test method is under the jurisdiction of Committee D24 on Carbon Black and are 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.

3.1.3.1 *Discussion*—The aggregate irregularity resists compression and thus the compressed volume is a function of the degree of this irregularity. The compressed void volumes are specific to the compressed volume measuring instrument and to the conditions of test. —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

4.1The compressed volume of a weighed dry test sample of a candidate carbon black is obtained in a void volume instrument under calibration test conditions. From the compressed volume, the void volume is obtained by subtracting the theoretical volume.

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 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

5.1The void volume of a carbon black expressed in absolute terms, VV, is a carbon black property that reflects differences in structure for candidate carbon blacks. Structure is a generic term that is a function of the shape irregularity and deviation from sphericity of carbon black aggregates. The more that a carbon black resists compression by having substantial aggregate irregularity and non-sphericity, the greater the compressed volume and void volume.

5.2Structure is a property that influences the physical properties developed in carbon black compounds for use in tires, mechanical rubber goods, and other manufactured rubber products.

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 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, traditionally measured by OAN (Test Method D 2414) and COAN (Test Method D 3493), 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 D 4821). Any estimates of OAN or COAN derived from prediction models using void volume-pressure data should be labeled appropriately (that is, Test Method D 6086) 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 metal block with a vertically oriented cylindrical sample chamber (see an example in Fig. X1.1). 6.4.2When opened,Hysteresis in the sample port shall allow framework under the range of applied forces should be accounted for in the loading of a sample of carbon black. 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 indicatemeasure displacement shall be provided. The compressed volume of any sample is determined at the end of a test, by the distance from the end of the piston to the end of the sample port;cylinder; this is designated as a "height" in the procedure discussed in Section 9. A test is concluded and the height is measured when a predetermined pressure is attained. See . The sample height and cylinder diameter are used to calculate an apparent sample volume.