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Standard Test Methods for Rubber Compounding Materials—Determination of Particle Size Distribution of Recycled Vulcanizate Particulate Rubber[1]

This standard is issued under the fixed designation D5644; 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 These test methods describe the procedures for determining average particle size distribution of recycled vulcanizate particulate.

1.2 Method A describes the Ro-tap sieve test method for 60 mesh or coarser particles.

1.3 Method B describes the ultrasonic technique combined with optical microscope especially suitable for 80 mesh or finer particles. This procedure is based on Test Method D3849.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *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]

D297 Test Methods for Rubber Products—Chemical Analysis

D1416 Test Methods for Rubber from Synthetic Sources—Chemical Analysis (Withdrawn 1996)[3]

D1566 Terminology Relating to Rubber

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D3191 Test Methods for Carbon Black in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures

D3192 Test Methods for Carbon Black Evaluation in NR (Natural Rubber)

D3849 Test Method for Carbon Black—Morphological Characterization of Carbon Black Using Electron Microscopy

D5603 Classification for Rubber Compounding Materials—Recycled Vulcanizate Particulate Rubber

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E105 Practice for Probability Sampling of Materials

3. Terminology

3.1 *Definitions:*

3.1.1 *parent compound, n*—original compound used in the product.

3.1.2 *recycled vulcanizate rubber, n*—vulcanized rubber that has been processed to give particulates or other forms of different shapes, sizes, and size distributions.

3.1.3 *Discussion*—The words "vulcanizate" and "vulcanized rubber" are interchangeable. Additional terminology associated with this classification can be found in Terminology D1566.

4. Significance and Use

4.1 The particulate size distribution of vulcanizate particulate rubber is used for the purpose of assigning a product mesh or average particle size designation.

4.2 The product designation for mesh size for the Ro-tap method (Method A, as follows) is based on the size designation screen which allows a range for the upper limit retained of maximum 5 % for up to 850 µm (20 mesh) particles, maximum 10 % for 600 to 150 µm (30 to 100 mesh), and maximum 15 % for 128 to 75 µm (120 to 200 mesh). No rubber particles shall be retained on the zero screen (see Table 1, Classification D5603).

4.3 For Method A, the weight percent retained on a specific screen is noted whereas in Method B (ultrasonic technique), the number of particles at a particular size is counted.

[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are 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] The last approved version of this historical standard is referenced on www.astm.org.

4.4 Method B addresses problems that may be caused by tackiness and static electrical forces that recycled rubber particles exert on each other to form agglomerates, especially for 80 mesh or finer particles. This method eliminates agglomerate formation by ultrasonically dispersing the particles.

4.5 Both methods can be used as a quality control tool.

5. Method A: The Ro-tap Method

5.1 *Summary of Test Method:*

5.1.1 *Method A—Ro-tap Method:*

5.1.1.1 A 100 ± 1 g specimen of the recycled rubber is combined with a fixed amount of talc and placed on top of a series of mesh sieves, with the coarsest sieve being on top and the finest on the bottom. The specimen is placed in a Ro-tap shaker for 10 to 20 min, depending on the grade of the recycled rubber. The weight of the rubber retained on the individual sieves is then recorded and the mesh designation of the product determined.

6. Apparatus

6.1 *Mechanical Sieve Shaker*[4]—This is a mechanically operated sieve shaker that imparts a uniform rotary and tapping motion to a stack of 200-mm (8-in.) sieves in accordance with 6.2. The sieve shaker should be adjusted to accommodate a stack of sieves, receiver pan, and cover plate. The bottom stops should be adjusted to give a clearance of 1.5 mm (0.06 in.) between the bottom plate and the screens so that the screens will be free to rotate. The sieve shaker machine shall be powered with an electric motor operating 28.75 to 29.17 Hz (1725 to 1750 rpm). This will produce 2.33 to 2.60 Hz and 280 to 320 rotary motions/min. The cover plate shall be fitted with a cork stopper that shall extend from 3.00 to 9.00 mm (0.118 to 0.354 in.) above the metal recess. At no time shall a rubber, wood, or other material other than cork be permitted.

6.2 *Standard Sieves,* stainless steel or brass, 200 mm (7.9 in.) in diameter in accordance with Specification E11. The sieve set should include a lid and a bottom pan.

6.3 *Balance,* with a sensitivity of 0.1 g.

6.4 *Brush.*

6.4.1 A Tyler Model 1778-SB soft brass wire brush for cleaning sieves 100 mesh and coarser.

6.4.2 A nylon bristle brush for cleaning sieves finer than 100 mesh.

6.5 *Jar,* capacity of 500 cm^3 (1 pint) with large opening.

6.6 *Rubber Balls,* [5] with a diameter from 25 to 50 mm (1 to 2 in.) or Plastic Rings, with a height of 20 ± 3 mm, an outside diameter of 60 ± 3 mm, and an inside diameter of 58 ± 3 mm. The height of the balls or rings must be lesss than the depth of the screens being used. Enough balls or rings are needed to have two balls or rings per sieve. Balls and rings are not to be used simultaneously.

6.7 *Talcum Powders,* usually some mixture of magnesium silicate, silica, magnesium oxide, magnesium-aluminum silicate with at least 90 % of the particles being less than 40 µm (approximately 400 mesh) in size.

7. Procedure

7.1 Select test screens appropriate to the particle size distribution of the product being tested. A set of two to six sieves and a receiver pan are normally used. The actual number of sieves is to be agreed upon by vendor and customer.

7.2 Clean each screen with brush (see 6.4), making sure all particles are removed from both sides of screen.

7.3 Stack test screens in order of increasing mesh size with smallest number on top (coarsest) and highest number on bottom (finest). For products of 425 µm (40 mesh) or finer, add two rubber balls or plastic rings per sieve. For products coarser than 425 µm (40 mesh), the use of rubber balls or plastic rings as agitation aid is optional. Rubber balls and plastic rings are not to be used simultaneously. Same size balls shall be used on any one screen.

7.4 Add bottom receiver pan to stack.

7.5 Obtain approximately 150 to 200 g of vulcanizate particulate rubber from the lot (refer to Practice E105).

7.6 Prepare a 100-g specimen as follows:

7.6.1 Weigh 100 g of specimen to the nearest gram.

7.6.2 Weigh talc according to product gradation designation. For products designated coarser than 300 µm (50 mesh), weigh 5.0 g of talc. For products designated 300 µm (50 mesh) or finer, weigh 15.0 g of talc.

7.6.3 Add talc to specimen.

7.6.4 Mix thoroughly by placing talc and specimen in a 500-cm^3 (1-pt) jar and shake the jar for a minimum of 1 min, until agglomerates are broken and talc is uniformly mixed.

7.7 Place the specimen on the top sieve and place a cover on the stack.

7.8 Place the stack in the shaker.

7.9 Activate the shaker for 10 min for products designated coarser then 300 µm (50 mesh). For products designated 300 µm (50 mesh) or finer, activate the shaker for 20 min.

7.10 After the shaker completes the appropriate cycle, remove the stack.

7.11 Starting with the top sieve, remove the screened fraction by gently tapping its contents to one side and pouring the contents on the balance and recording its mass to the nearest 0.1 g. Record any mass less than 0.1 g as trace.

7.12 Brush any material adhering to the bottom of the screen onto the next finer screen.

7.13 Zero the balance in preparation for weighing the retained contents of the next screen.

7.14 Repeat 7.11 to 7.12 until all sieves in the stack and the bottom pan have been emptied, weighed, and recorded. This gives percent retained on each screen.

[4] The Ro-Tap Sieve Shaker meets the specified conditions and has been found satisfactory for this purpose, and is available from many scientific laboratory suppliers.

[5] Available from various sieve manufacturing suppliers.