Designation: D 5644 – ~~96~~01

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 D 5644; 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 Th~~i~~ese test ~~method covers~~ methods describe the ~~determination of the~~ procedures for determining average particle size distribution of recycled vulcanizate particulate.

1.2 Method A descr~~u~~ib~~b~~es 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 D 3849.

1.2~~4~~ The values stated in SI units are to be regarded as the ~~standard. The values in parentheses are for information only.~~

~~1.3~~ 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:*

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[1] This test method is under the jurisdiction of ASTM Committee D~~-~~11 on Rubber and is the direct responsibility of Subcommittee D11.26 on Recycled Rubber.
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D ~~1566 Terminology Relating to~~ 297 Test Methods for Rubber Products—Chemical Analysis[2]
D 1416 Test Methods for Rubber from Synthetic Sources—Chemical Analysis[2]
D 1566 Terminology Relating to Rubber[2]
D 3182 Practice for Rubber-Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[2]
D 3191 Test Methods for Carbon Black in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures[2]
D 3192 Test Methods for Carbon Black Evaluation in NR (Natural Rubber)[2]
D 3849 Test Method for Carbon Black—Primary Aggregate Dimensions from Electron Micorscope Image Analysis[2]
D 5603 Classification for Rubber Compounding Materials—Recycled Vulcanizate Particulate Rubber[2]
E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]
E 105 Practice for Probability Sampling of Materials[3]

3. Terminology

3.1 *Definitions:*

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

3.1.2 *recycled vulcanizate ~~particulate~~ rubber, n*—~~recyclable vulcanizate~~ 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 D 1566.

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 ~~and~~, maximum 10 % for ~~finer than 850 µm.~~ 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 ~~top (zero)~~ zero screen (see Table 1, Classification D 5603).

4.3 For Method A, ~~6~~ 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.

4~~,~~.4 Method B addresses problems that may be caused by tackiness and ~~Note 1).~~ 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. ~~Apparatus~~ 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 ~~5~~ 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.

5~~6~~.2 *Standard Sieves*, stainless steel or brass, 200 mm (7.9 in.) in diameter ~~conforming to~~ in accordance with Specification E 11. The sieve set should include a lid and a bottom pan.

5~~6~~.3 *Balance*, with a sensitivity of 0.1 g.

5~~6~~.4 *Brush*.

[2] *Annual Book of ASTM Standards*, Vol 09.01.
[3] *Annual Book of ASTM Standards*, Vol 14.02.
[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.

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

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

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

56.6 *Rubber Balls*, [5] with a diameter from 25 ~~mm (1 in.)~~ 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.

56.7 *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.

67. Procedure

67.1 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.

67.2 Clean each screen with brush (see ~~5~~ 6.4), making sure all particles are removed from both sides of screen.

67.3 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.

67.4 Add bottom receiver pan to stack.

67.5 Obtain approximately 150 to 200 g of vulcanizate particulate rubber from the lot (refer to Practice E 105).

67.6 Prepare a 100-g specimen as follows:

67.6.1 Weigh 100 g of specimen to the nearest gram.

67.6.2 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.

67.6.3 Add talc to specimen.

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

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

67.8 Place the stack in the shaker.

67.9 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.

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

67.11 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.

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

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

67.14 Repeat ~~6~~ 7.11 to ~~6~~ 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.

78. Calculation

78.1 The sum of the masses of each fraction from the sieves and the bottom pan shall not be less than the original mass of the specimen plus mass of talc less 2 g, or greater than the original mass of the ~~sample~~ specimen plus 100 % of talc added. Repeat test if either of these conditions occurs.

78.2 To adjust for the addition of talc to the specimen, the mass of the contents of the bottom pan is adjusted by the following ~~calculation:~~ equation:

$$x = y - (z - 100) \tag{1}$$

where:

x = mass of rubber in bottom pan,
y = total mass of contents in bottom pan, and
z = total mass of contents of all six sieves plus bottom pan.

78.3 The top screen (zero screen) selected shall be one in which no rubber particles are retained. This "zero percent retained screen" is designated in Classification D 5603 Table 1 (see Note 1).

78.4 The second screen in the sieve deck is for product designation and can contain a maximum 5 % rubber particles for up to 850 µm (20 mesh) and a maximum 10 % for finer than 850 µm (20 mesh) (see Note 1).

NOTE 1—An example of ~~7.3~~ 8.3 and ~~7.4~~ 8.4 is if an 850-µm (20 mesh) sieve contains zero rubber particles and a 600-µm (30-mesh) sieve contains 3 % rubber particles, then the proper product designation for this material is 600-µm (30-mesh) particulate rubber product.

[5] Available from various sieve manufacturing suppliers.

~~8. Report~~

~~8.1 Report the following information:~~
~~8.1.1 Date of test,~~
~~8.1.2 Proper identification of samples,~~
~~8.1.3 Identification of each sieve used,~~
~~8.1.4 The residue mass on each sieve,~~
~~8.1.5 The mass on the bottom pan and its adjusted mass, and~~
~~8.1.6 The product mesh size determined for the sample.~~

9. Report

9.1 Report the following information:
9.1.1 Date of test,
9.1.2 Proper identification of specimens,
9.1.3 Identification of each sieve used,
9.1.4 The residue mass on each sieve,
9.1.5 The mass on the bottom pan and ~~Bias~~
~~9.1 Round-robin testing will be conducted and precision and bias statements will be balloted~~ its adjusted mass, and
9.1.6 The product mesh size determined for ~~inclusion when testing is completed.~~ the specimen.

10. Precision and Bias

10.1 Round-robin testing will be conducted and precision and bias statements will be balloted for inclusion when testing is completed.

11. Keywords

1~~0~~1.1 particle size distribution; recycled vulcanizate particulate rubber

12. Method B—Ultrasonic and Light Microscopy Technique

12.1 *Summary of Test Method*
12.1.1 *Method B—Ultrasonic Technique*
12.1.1.1 A 25 ± 1 mg specimen of recycled rubber is ultrasonically dispersed in acetone, diluted appropriately, and placed on a microscope slide for subsequent light optical microscopic analysis. Photomicrographs are taken of the specimen and then computer software is used to automatically determine area measurements of the particles. The area measurements are mathematically converted to diameter data, which then can be used to produce statistical data and histograms of the particle size distribution.

13. Apparatus

13.1 *Test tubes*, 75 by 10 mm glass, with corks.
13.2 A benchtop tank-type ultrasonic dispersion bath with an output of 40 to 80 KHz and 105 to 270 W power.
13.3 *Glass microscope slides*, 25 by 75 mm.
13.4 *Transfer pipettes*, disposable polyethylene type.
13.5 *Light Optical Microscope*, requirements for a microscope for this test are that it should have a transmitted light accessory, and also a port to attach a camera. The microscope should be capable of magnifications of 30×.
13.6 *Image Analysis System*, which could consist of a camera, a video TV camera, or a digital camera system.
13.6.1 If a film camera is used, a photograph scanner will be needed to be able to scan the images and transfer them to a computer for subsequent image analysis.
13.6.2 If a video rate TV camera system is used, it must be connected to a computer frame-grabber card that will enable the user to directly transfer images to the computer for subsequent image analysis.
13.6.3 If a digital camera is used, the images can be directly captured and transferred to a computer.
13.7 *Computer*, with adequate speed and memory to be able to do image analysis. This may vary depending on what camera system is used for the analysis.
13.7.1 Computer software capable of dimensional image analysis.
13.7.2 Computer program to determine statistics and histogram information.
13.7.3 Stage micrometer.

14. Reagents and Materials

14.1 Acetone, commercial grade.
14.2 Recycled rubber crumb.

15. Sampling

15.1 Put 100 ± 1 g of the recycled rubber in a capped container and shake it ten times to ensure that the specimen is well-mixed

and the fine particles present have not settled, before testing.

16. Test Procedures

16.1 Place 25 ± 1 mg of recycled rubber in a 3 ml glass test tube.

16.2 Add 1 ml of acetone to the test tube and place a cork stopper in the test tube. At the same time, prepare another test tube with 1 ml of acetone only and put in a cork stopper.

16.3 Sonicate the specimen at 105 to 270 W for 5 min ± 15 s in an ultrasonic bath filled to 55 ± 2 mm with the surfactant solution recommended by the manufacturer of the ultrasonic bath or use a suitable substitute solution. The test tube should not be touching the bottom of the ultrasonic bath, but should be suspended in the bath by suspending a beaker. Many manufacturers have available, as an option, a basket in which a beaker can be suspended above the bottom of the bath.

16.4 Dilute the specimen by transferring 1 to 6 drops of the sonicated solution to the other test tube containing 1 cm^3 of acetone and sonicate for an additional 5 min.

16.4.1 The number of drops used for the dilution depends on the mesh grade of the specimen being analyzed and will vary from 1 drop for 170 mesh grades and finer up to 6 drops for the larger particle size grades. The optimum number of drops for best dispersion for each individual mesh grade must be determined by experimentation.

16.4.2 After the final 5 min sonification, immediately place 3 sequential drops on a clean glass microscope slide and allow the solvent to evaporate. For some grades, it may be desirable to swirl the solvent on the microscope slide gently while the solvent is evaporating to get an even distribution of the particles on the slide.

17. Specimen Analysis and Instrument Calibration

17.1 Using transmitted light, analyze the particles at 30× magnification and take photomicrographs of the specimens and also a stage micrometer (see 13.7.3) at that exact magnification.

17.2 Transfer the images into a computer by either scanning the micrographs, directly transferring video images by using the frame grabber, or directly store the images from a digital camera using the computer software supplied by the manufacturer of the digital camera.

17.3 Using the appropriate image analysis computer software, follow the computer software instructions for calibration using the stored image of the stage micrometer. Individual calibrations must be used for each magnification used for analyzing the unknown specimens of recycled rubber.

17.4 Measure the areas of the individual particles by using fill option or by allowing the computer to trace the circumference of all of the individual particles automatically (for software with this capability). The computer software will automatically calculate the areas of the particles for either computer program.

17.4.1 If the particles appear to be touching each other, they can be manually erased before the measurements are made, using the eraser tool.

17.5 Transfer the area data to a spreadsheet and determine the diameters of the non-spherical particles by mathematically transforming the area data into diameter data by using the following formula:

$$\sqrt{\frac{4\,(a1)}{3.14}} \tag{2}$$

where:

a1 = area data in the first column of the spreadsheet.

NOTE 2—This formula can be directly entered in the formula bar of the spreadsheet. The result for the first data point is calculated and placed in the "B" column of the spreadsheet, and in this case, the B1 location.

17.6 After determining the diameter of the first particle, use the "copy" command on the B1 data and "paste" it into the rest of the B column to determine the diameters of the remaining particles.

17.7 From the "tools" dropdown file, choose "data analysis" and perform the descriptive statistics and histogram functions on the diameter data in column B.

18. Report

18.1 Report the following information:

18.1.1 Descriptive statistics, including the average particle size as determined by using the units of measure desired. The average particle size can be reported separately, if desired.

18.1.2 Histogram plot as determined by spreadsheet program.

19. Keywords

19.1 particle size distribution; ultrasonic light optical microscopic technique