



Designation: **D 5603 – 9601**

## **Standard Classification for Rubber Compounding Materials—Recycled Vulcanizate Particulate Rubber<sup>1</sup>**

This standard is issued under the fixed designation D 5603; 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 classification covers the compounding material commercially known as recycled vulcanizate particulate rubber. Recycled vulcanizate particulate rubber is the product that results when vulcanizate rubber has been processed by some means to obtain a desired particle size distribution.

1.2 *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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<sup>1</sup> This classification is under the jurisdiction of Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.26 on Rubber Recycling. Current edition approved Nov. 10, ~~1996~~, 2001. Published January ~~1997~~, 2002. Originally published as D 5603 - 96. Last previous edition D 5603 - 96.

- D 297 Test Methods for Rubber Products—Chemical Analysis<sup>2</sup>  
 D 1418 Practice for Rubber and Rubber Latices—Nomenclature<sup>2</sup>  
 D 1509 Test Methods for Carbon Black—Heating Loss<sup>2</sup>  
 D 1566 Terminology Relating to Rubber<sup>2</sup>  
 D 1900 Practice for Carbon Black—Sampling Bulk Shipments<sup>2</sup>  
 D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets<sup>2</sup>  
 D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries<sup>2</sup>  
 D 5644 Test Method for Rubber Compounding Materials—Determination of Particle Size Distribution of Recycled Vulcanizate Particulate Rubber<sup>2</sup>  
 E 11 Specification for Wire- Cloth and Sieves for Testing Purposes<sup>3</sup>  
 E 105 Practice for Probability Sampling of Materials<sup>3</sup>  
 E 122 Practice for Choice of Calculating Sample Size to Estimate, With a Measure of Quality Specified Tolerable Error, the Average for a Characteristic of a Lot or Process<sup>3</sup>

### 3. Terminology

#### 3.1 Definitions:

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

3.1.1.1 *Discussion*—The words “vulcanizate” and “vulcanized” are interchangeable. Additional terminology associated with this classification can be found in Terminology D 1566.

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

### 4. Significance and Use

4.1 Recycled vulcanizate particulate rubber is important in rubber compounding. It allows the rubber compounder to add a certain percentage of rubber filler back into its parent or similar compounds or to use the material as substitute or stand alone compound. Its use may lower compound costs or improve performance characteristics, or both.

4.2 Many types of recycled vulcanizate particulate rubber are available, with the number of types of vulcanizate particulate rubber limited only by the number of parent compounds.

4.3 Use of recycled rubber has a positive environmental impact.

### 5. Classification of Recycled Vulcanizate Particulate Rubber

5.1 Several grades of recycled vulcanizate particulate rubber exist. Their classification is based on two major characteristics: particle size distribution and the polymer type found in the parent rubber from which the recycled vulcanizate particulate rubber was derived.

5.2 *Particle Size Distribution*—maximum particle size. (For product mesh size designations, see 5.5.)

5.2.1 Coarse rubber powders are products with designations of 425  $\mu\text{m}$  (40 mesh) or larger. These materials typically range in particle sizes from 2000  $\mu\text{m}$  (10 mesh) to 425  $\mu\text{m}$  (40 mesh) regardless of polymer type or method of processing.

5.2.2 Fine rubber powders are products with designations smaller (finer) than 425  $\mu\text{m}$  (40 mesh). These materials typically range in particle sizes from less than 300  $\mu\text{m}$  (50 mesh) to less than 75  $\mu\text{m}$  (200 mesh) regardless of polymer type or method of processing.

5.3 *Classification by Parent Compounds*:

5.3.1 Based on polymer/compound type alone, the three most common grades (Grades 1, 2, and 3) used in rubber compounding and three other grades are shown as follows:

5.3.1.1 *Grade 1*—Whole tire recycled vulcanizate particulate rubber is prepared from passenger car, truck, and bus tires from which the fiber and metal have been removed. The rubber is then processed to the desired particle size.

5.3.1.2 *Grade 2*—Car, truck, and bus tread recycled vulcanizate particulate rubber is prepared from car, bus, and truck tread only, processed to the desired particle size. This material is commonly referred to as “peel” rubber.

5.3.1.3 *Grade 3*—Tread recycled vulcanizate particulate rubber prepared by utilizing tire retread buffings only. This material is generated from car, truck, and bus tire retreading where the processing (buffing) of the tire includes the tread and tire shoulder area only. This material is then processed to the desired particle sizes. The vulcanizate particulate rubber produced from this material is usually characterized by some elongated rubber particles in the vulcanizate particulate rubber with mesh size designations coarser than 600  $\mu\text{m}$  (30 mesh). When finer mesh sizes are produced, the elongated nature of the particles is no longer present.

5.3.1.4 *Grade 4*—Whole tire recycled vulcanizate particulate rubber prepared by utilizing tire retread buffings only. This material is generated from car, truck, and bus tire retreading where the processing (buffing) of the tire includes the tread, tire

<sup>2</sup> Annual Book of ASTM Standards, Vol 09.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 14.02.

shoulder area, and the sidewalls. This material is then processed to the desired particle sizes. The vulcanizate particulate rubber produced from this material is usually characterized by elongated rubber particles in the vulcanizate particulate rubber with mesh size designation coarser than 600  $\mu\text{m}$  (30 mesh). When finer mesh sizes are produced, the elongated nature of the particles is no longer present.

5.3.1.5 *Grade 5*—Recycled vulcanizate particulate rubber prepared from off road tires, large equipment tires, industrial tires, forklift tires, farm implement tires, and others. This does not include car, bus, and truck tires.

5.3.1.6 *Grade 6*—Recycled vulcanizate particulate rubbers are prepared from rubber vulcanizates from non-tire rubber products. This is not to imply that all polymer types can be used interchangeably. Actual classification according to polymer types shall be agreed upon between vendor and customer.

5.4 *Classification by Particle Size:*

5.4.1 The particle size designation portion of the classification is based on physical characteristics (that is, particle distributions using dry material sieve testing). Test Method D 5644 is used to determine the particle size distribution. The product size designation is the mesh size based on the size designation sieve-screen which allows a range for the upper limit retained of zero to some maximum value depending on the nominal mesh size designation (see Table 1). Each product will also designate a sieve on which no product is retained. This sieve (zero screen) will have a larger nominal opening than the product size designation screen. Additional sieves can be specified as agreed upon between the vendor and the customer to obtain a particular size distribution.

5.5 *Overall Classification Designations:*

5.5.1 The overall classification of recycled vulcanizate particulate rubber products is based on two designations: Particle Size Distribution and Origin of the Rubber (Grades 1 through 6).

5.5.2 Table 1 lists the standard particle size designations for recycled vulcanizate particulate rubber. Other product size designations not listed in Table 1 may be agreed upon between the vendor and customer following the ASTM guidelines in this classification.

**6. Sampling**

6.1 Test portions for the testing as outlined in Section 7 will be taken on approximately each 1000 to 1250 kg or as agreed upon between vendor and customer.

6.2 Test portions for sieve testing on products that are coarser than 800  $\mu\text{m}$  (20 mesh) are selected in accordance with Practices E 105 and E 122.

**7. Composition and Properties**

7.1 Table 2 shows the typical chemical properties of all the tire grade particulate rubber in Grades 1, 2, 3, and 4. Properties of particulate rubber other than those in Grades 1, 2, 3, and 4 shall be as agreed upon between supplier and customer.

7.2 The product designations portion of this classification is based on physical characteristics (that is, particle distributions). Refer to 5.4 and Table 1.

7.3 Material to be substantially free of other foreign contaminants including, but not limited to, wood and wood chips, paper, metal, sand, dirt, and glass.

7.3.1 Visually examine the material for foreign contaminants. None of these should be present.

7.3.2 To check for iron content, weigh a 100-g specimen of recycled vulcanizate particulate rubber and place on a flat nonmagnetic surface. Pass a small horseshoe magnet over and through the specimen for 60 s. Remove all metal fragments from the magnet. Weigh the iron fragments in grams and record the mass as the percentage of free iron.

**TABLE 1 Recycled Rubber Product Designation**

Nominal Product Designation	Example Classification D 5603 Designation <sup>A</sup>	Zero Screen, $\mu\text{m}$	Percent Retained on Zero Screen	Size Designation Screen, $\mu\text{m}$	Maximum Percent Retained on Designation Screen
10 Mesh	Class 10-X	2360 (8 Mesh)	0	2000 (10 Mesh)	5
20 Mesh	Class 20-X	1180 (16 Mesh)	0	850 (20 Mesh)	5
30 Mesh	Class 30-X	850 (20 Mesh)	0	600 (30 Mesh)	10
40 Mesh	Class 40-X	600 (30 Mesh)	0	425 (40 Mesh)	10
50 Mesh	Class 50-X	425 (40 Mesh)	0	300 (50 Mesh)	10
60 Mesh	Class 60-X	300 (50 Mesh)	0	250 (60 Mesh)	10
70 Mesh	Class 70-X	259 (60 Mesh)	0	212 (70 Mesh)	10
80 Mesh	Class 80-X	250 (60 Mesh)	0	180 (80 Mesh)	10
100 Mesh	<del>Class 100-X</del>	<del>180 (80 Mesh)</del>	0	<del>150 (100 Mesh)</del>	10
100 Mesh	Class 100-X	180 (80 Mesh)	0	150 (100 Mesh)	10
120 Mesh	Class 120-X	150 (100 Mesh)	0	128 (120 Mesh)	15
140 Mesh	Class 140-X	128 (120 Mesh)	0	106 (140 Mesh)	15
170 Mesh	Class 170-X	106 (140 Mesh)	0	90 (170 Mesh)	15
200 Mesh	Class 200-X	90 (170 Mesh)	0	75 (200 Mesh)	15

<sup>A</sup>When specifying materials, replace the X with the proper parent material grade designation code. For example, Class 30-2 would indicate a 600  $\mu\text{m}$  (30 mesh) product made from Grade 2 material, car, truck, and bus tread rubber. Class 100-6 would indicate a 150  $\mu\text{m}$  (100 mesh) product made from Grade 6 material, non-tire rubber.

**TABLE 2 Properties for Recycled Rubber (Grades 1–6)**

Property	Percent	Test Method
(a) Grades 1–4		
Acetone extractables	8–22	D 297, Sections 17, 18, 19
Ash, max	8	D 297, Sections 34, 35, 36, 37
Carbon black	26–38	D 297, Sections 38, 39
Loss on heating, max	1	D 1509
Natural rubber	10–35	D 297, Sections 52, 53
Rubber hydrocarbon content (RHC), min	42	D 297, Section 11
(b) Grades 1–6		
Metal content, max	0.1	see 7.3.2
Fiber content, max (Grades 1, 4, 5, 6)	0.5	see 7.4
Fiber content, max (Grades 2, 3)	nil	see 7.4

7.4 Fiber content of material may be agreed upon by supplier and customer.

7.4.1 The general fiber content of Grades 1, 4, 5, and 6 should contain no more than that specified in Table 2.

7.4.2 Grades 2 and 3 are prepared from tread rubber only and should contain no fiber.

7.4.3 The method to check for fiber content is as follows:

7.4.3.1 Perform a normal sieve analysis procedure in accordance with Test Method D 5644.

7.4.3.2 As the test screens are disassembled, there may be free fabric in the form of “fabric balls” which can be removed from each screen level.

7.4.3.3 Accumulate the “fabric balls” as the screens are disassembled.

7.4.3.4 Weigh the “fabric balls” in grams and record their mass as the percentage of free fabric.

7.4.3.5 The “fabric balls” may have a tendency to entrap very small particles of rubber. It is permissible to attempt to dislodge these particles by shaking the “fabric balls” or dropping them on a clean surface. Weighing the “fabric balls” with the entrapped rubber will tend to distort the fabric content to the high side.

## **8. Determination of the Bulk Density of Recycled Vulcanizate Particulate Rubber**

8.1 The bulk density of recycled vulcanizate particulate rubber is determined from the mass of the particulate rubber contained in a special cylindrical container that is exactly  $1000.0 \pm 0.1 \text{ cm}^3$  in volume.

### 8.2 Apparatus:

8.2.1 Cylindrical Container, 1000-cm<sup>3</sup> capacity, having uniform height and no pouring lip or deformation of the walls of the container.

8.2.2 Straightedge or Spatula, at least 150 mm in length.

8.2.3 Balance, with a sensitivity of 0.1 g.

### 8.3 Procedure:

8.3.1 Obtain approximately  $275 \pm 25 \text{ g}$  of recycled vulcanizate particulate rubber from the lot (see Practice E 105).

8.3.2 Pour the sample into the center of the tared container from a height not more than 50 mm above the rim of the container. A large enough excess should be used to form a cone of the product above the rim of the cylindrical container. Immediately level the surface with a single sweep of the straightedge or spatula held perpendicular to and in firm contact with the lip of the container. Record the mass of the recycled vulcanizate particulate rubber to the nearest gram.

### 8.4 Calculation:

8.4.1 Calculate the bulk density to the nearest  $0.1 \text{ kg/m}^3$  as follows:

$$D_R = W \quad (1)$$

where:

$D_R$  = bulk density,  $\text{kg/m}^3$ , and

$W$  = mass of recycled vulcanizate particulate rubber, g.

### 8.5 Report:

8.5.1 Report the following information:

8.5.1.1 Date of test.

8.5.1.2 Proper identification of samples.

8.5.1.3 Result obtained, reported to the nearest  $0.1 \text{ kg/m}^3$ .

### 8.6 Precision and Bias:

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

## **9. Determination of the Density of Recycled Vulcanizate Particulate Rubber<sup>4</sup>**

### 9.1 Apparatus:

9.1.1 Balance, with a bridge and minimum sensitivity of 0.1 g.

9.1.2 Two Roll Laboratory Rubber Mill.

9.1.3 Supporting Wire, for weighing sample.

9.1.4 Hydraulic Press, with electric platens

9.1.5 ASTM Tensile Mold, (Practice D 3182).

### 9.2 Procedure:

9.2.1 Mill the ground rubber sample on the two roll mill. Keep the mill tight enough to knit the ground rubber together. Layer several sheets of the sample together. Use enough sheets to completely fill the tensile mold.

9.2.2 Heat the mold with sheets at 140 ± 2°C for 30 ± 1 min at a minimum pressure of 7 MPa on the cavity area. Cool the molded sample to 23 ± 2°C, and then cut a specimen.

9.2.3 Weigh the specimen first in air.

9.2.4 Dip the specimen in alcohol or acetone, or any appropriate wetting agent to eliminate any bubbles on the surface that will cause errors in the determination.

9.2.5 Suspend supporting wire and weigh in water at 23 ± 2°C.

9.2.6 Suspend the specimen with supporting wire and weigh the specimen in water at 23 ± 2°C.

### 9.3 Calculations:

9.3.1 Calculate the density as follows:

$$\frac{0.9971 \times A}{A - (B - C)} \times \frac{1}{1000} \quad (2)$$

where:

A ≡ mass of specimen in air, g.

B ≡ mass of specimen and supporting wire in water, g.

C ≡ mass of supporting wire in water, g.

D ≡ density, kg/m<sup>3</sup>.

0.9971 ≡ density of water.

### 9.4 Report:

9.4.1 Report the following information:

9.4.1.1 Date of test.

9.4.1.2 Proper identification of samples.

9.4.1.3 Results obtained, reported to the nearest 0.1 kg/m<sup>3</sup>.

### 9.5 Precision and Bias:

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

## **10. Keywords**

810.1 recycled vulcanizate particulate rubber

<sup>4</sup> This procedure is modified for ground rubber from Test Method D 297, Hydrostatic section.

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