



Designation: D5461 – 06 (Reapproved 2017)

Standard Test Method for Rubber Compounding Materials—Wet Sieve Analysis of Powdered Rubber Compounding Materials¹

This standard is issued under the fixed designation D5461; 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 test method covers a procedure for the qualitative evaluation of aggregate size and aggregate size distribution of powdered rubber accelerators and other compounding materials which are insoluble in water.

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

1.3 *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.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Terminology

2.1 Definitions:

2.1.1 *aggregate*—a cluster of individual particles making up the mass of the material.

2.1.2 *aggregate size distribution*—the distribution of aggregates obtained by passing wetted rubber chemicals through stacked sieves of known mesh which are arranged in order of decreasing size.

3. Summary of Test Method

3.1 A sample of powdered rubber chemical is wetted with a dilute aqueous solution of soap and defoamer. The sample is transported by water flow through stacked sieves arranged in order of decreasing mesh size. The material retained on the face of each sieve is dried in an oven. The dry mass of retained

material is obtained for each sieve. The percent retained material is calculated on the basis of original sample mass. Individual masses and percentages are summed to evaluate the aggregate size distribution for the sample.

4. Significance and Use

4.1 This test method is used to evaluate the suitability of powdered rubber chemicals, particularly accelerators, for use in rubber compounds that require very small particle clusters to achieve a uniform cross-linked network. This test method is intended to be used to ensure that no excessively large particles are present that would result in network “flaws” and to determine that the powdered product follows a typical pattern of size distribution.

4.2 In view of the inherent variability in this type of method, it is recommended that the results be used only in a semi-quantitative sense; that is, comparisons within a laboratory or relative comparisons of samples against reference samples, or both.

5. Apparatus

5.1 *Standard Sieves*, stainless steel, 200 mm (approximately 8 in.) diameter containing selected stainless steel wire cloths with openings in the range of 45 to 250 μm . (This corresponds to 325 to 60 mesh sieves.)

NOTE 1—If the 200 mm diameter sieves are not available, the smaller 76 mm diameter sieves can be used. In this case, the sample mass should be reduced from 50 to 10 g.

5.2 *Analytical Balance*, 150 g capacity, 1.0 mg sensitivity.

5.3 *Convection Oven*, controlled at $70 \pm 2^\circ\text{C}$. For low melting materials, the oven temperature should be set 10°C below the melting point.

5.4 *Aluminum Weighing Dishes*, disposable, 55 to 60 mm diameter, approximately 1 g mass.

5.5 *No. 6 Stiff Bristle Artist Brush*, having 10 to 45 mm long bristles.

6. Reagents and Materials

6.1 *Detergent*, sodium alkybenzenesulfonate (SABS), 40 % active or equivalent.

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

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6.2 *Silicone Defoamer*,² water soluble.

6.3 *Soap Solution*, prepared by adding 100 g of SABS and 200 cm³ silicone defoamer to 20 dm³ water.

7. Procedure

7.1 Place the soap solution in a suitable carboy on a shelf or stand over a sink and discharge the solution from the bottom of the container through 6 mm (0.25 in.) rubber tubing connected to a bottom outlet. A pinch-clamp serves as a control valve.

7.2 Assemble pre-cleaned sieves (200 or 76 mm) into a stack, with the largest sieve opening on top. For example:

	Sieve Size	Sieve Opening, μm
Top	60	250
	100	150
	200	75
	270	53
	325	45
Bottom	325	45

7.3 Place the sieve stack in the sink under the discharge line for the soap solution and under a sink tap equipped with an aerator.

7.4 Wet the surface of each screen with water and soap solution.

7.5 Tare a series of commercially available aluminum weighing dishes, corresponding to each sieve. Take care to maintain the “dish to sieve” correspondence.

7.6 Weigh 50 + 0.1 g sample for 200 mm sieve or 10.0 + 0.1 g sample for 76 mm sieve of the test material into a 1 dm³ screw cap bottle equipped with a vinyl or TFE-fluorocarbon insert.

7.7 Add 100 cm soap solution to the bottle and screw the cap onto the bottle.

7.8 Agitate and mix the contents of the bottle by gentle shaking. Make sure the contents are thoroughly wetted as determined by visual inspection. Absence of foam, bubbles, and visible chunks indicates complete wetting.

7.9 Pour the contents of the bottle onto the top sieve in the sieve stack. Ensure complete transfer of the sample by washing the bottle two to three times with 50 cm³ portions of water.

7.10 Gently wash the sample through the sieve with a small stream of soap solution. Switch to an aerated stream of water to finish washing the sample through the sieve. If necessary, alternate the use of water and soap solution to assure complete transport of sample through the sieve breaking up agglomerates using a stiff bristle brush. (Clean the brush with water over sieve.)

7.11 When transfer is complete, wash the residue to the middle of the sieve with a gentle stream of water. Take care to ensure that all soap residue is rinsed from the remaining sample.

7.12 Remove the top sieve from the stack and wipe excess water from the sieve sides and bottom with a dry lint-free absorbent towel and set aside.

7.13 Repeat 7.10 through 7.12 for all sieves in the stack.

7.14 Dry each sieve plus residue in a 70°C convection oven for 1 h. (Low melting materials should be dried at 10°C below the melting point.)

7.15 Remove the sieves from the oven and allow them to cool to ambient temperature.

7.16 Remove the residue from the sieve by gently tapping the sides while pouring the solids into a tared, properly labeled aluminum weighing dish.

NOTE 2—It may be possible to avoid this transfer when using the smaller 76 mm sieves. In that case, the sieves should be tared initially, then reweighed following drying and cooling to ambient temperature.

7.17 Record the quantity of residue contained on each sieve.

8. Calculation

8.1 Calculate the percent retained on each sieve as follows:

$$\text{Retained \%} = \frac{A}{B} \times 100 \quad (1)$$

where:

A = mass of residue, g, and
 B = mass of sample, g.

8.1.1 Residue mass should be accumulated for successive sieves. To determine the residue mass on a finer sieve, it is necessary to sum the masses of residues collected on all coarser sieves and add it to the mass from the selected sieve.

8.2 The amount of sample passing through each sieve is determined by subtracting the percent of material collected on the sieve and all coarser sieves from 100.

8.3 The amount of material passing through all sieves is determined by subtracting the total amount retained on all sieves from 100.

9. Report

9.1 Report the following information:

- 9.1.1 Proper sample identification,
- 9.1.2 Identification of each sieve used,
- 9.1.3 Mass retained on each sieve in grams,
- 9.1.4 Percent of material passing through each sieve, and
- 9.1.5 Percent of material passing through all sieves.


10. Precision and Bias

10.1 Due to the qualitative nature of this test method, classical precision testing is not appropriate. A pilot round-robin precision test gave unacceptably large between-laboratory variations.

11. Keywords

11.1 particle size; rubber compounding materials; wet sieve analysis

² The sole source of supply of the apparatus (Dow-Corning FG-10) known to the committee at this time is Dow-Corning, Midland, MI. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

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