Designation: D 4572 – 89 (Reapproved 1999)

Standard Test Method for Rubber Chemicals—Wet Sieve Analysis of Sulfur[1]

This standard is issued under the fixed designation D 4572; 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 the evaluation of the particle size distribution of the coarse fraction of sulfur. It is limited to the measurement of those particles greater than 45 µm (No. 325 sieve).

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:*

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

E 11 Specification for Wire-Cloth Sieve for Testing Purposes[3]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[3]

3. Significance and Use

3.1 This test method is used to evaluate sulfur for suitability as a rubber vulcanizing agent. Sulfur particles must be significantly small enough to dissolve in rubber during cure and produce a uniform network of cross-links. This test method is used as a quality control method to ensure that no excessively large sulfur particles are present and to see if the product follows a typical pattern of size distribution.

3.2 This procedure is necessary when problems of the sulfur caking occur with the use of the dry sieving procedure for particle size.

4. Apparatus

4.1 *Standard Sieves*, stainless steel, 76 mm diameter containing selected stainless steel wire cloth in the range of 45 to 250 µm mesh count.

4.2 *No. 6 Stiff Bristle Artist Brush*, having 10 to 15 mm long bristles.

4.3 *Balance*, with a minimum capacity of 150 g sensitive to 0.001 g.

4.4 *Oven*, circulating air type, controlled to 70 ± 2°C.

5. Materials

5.1 *Liquid Detergent*.

6. Procedure

6.1 Weigh 10.0 g of the sulfur sample into a 250-cm ^{3} beaker. Wet the sulfur with 25 mL of water. Mix thoroughly with a glass stirring rod to guarantee wetting all the sulfur.

NOTE 1—A 1 % solution of a liquid detergent may be used if the sulfur sample does not wet out properly.

6.2 Weigh each cleaned and dried sieve to the nearest 0.001 g. Assemble preweighed stainless steel sieves in order to descending fineness with the coarsest screen on top. Carefully transfer the wetted sulfur to top screen using additional water to wash all of the sulfur out of the beaker. Wash the sulfur through the top sieve with a gentle stream of water from a nozzle and detergent (see 5.1) as needed. Tap or vibrate the screen while washing. Finally, break up all agglomerated sulfur particles using the brush. Clean sulfur from the bristles of the brush using wash water. Be careful that the wash water does not back up on the finer sieves causing the sulfur slurry to overflow the sieve sides.

6.3 Remove the top sieve. Wash off any residual sulfur slurry on the underside of the sieve. Repeat the washing procedure described in 6.2 ensuring total transport of the fine sulfur particles through each successive sieve.

6.4 Carefully dry the sides of the sieves with a lint free towel, and place each sieve separately in a drying oven at 70°C for 1 h or until the sulfur is dry. Remove the sieves and cool at least ½ h in a dessicator.

6.5 Weigh each sieve to the nearest 0.001 g. When the amount of sulfur on the coarser sieves is very small (a few particles), it may be appropriate to brush the particles on to a preweighed glassine weighing paper and weigh the sulfur directly.

7. Calculation

7.1 Calculate the percent retained on each sieve, *P*, as follows:

$$P = \frac{M_1}{M_2} \times 100 \quad (1)$$

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

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

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

TABLE 1 Type 1 Precision Results—Sulfur, Percent Through Wet-Sieve (250–75 µm Mesh)

Material	Mean Test Level	250 µm mesh		180 µm mesh		150 µm mesh		75 µm mesh	
		r^A	R^B	r	R	r	R	r	R
Oil Treated, 90 % Insoluble Sulfur—A	[C]	0.014	0.014	0.022	0.179	0.044	0.626	0.213	2.26
Oil Treated, 90 % Insoluble Sulfur—B	[C]	0.034	0.034	0.028	0.104	0.058	0.150	0.218	5.53
General Purpose Ground Sulfur	[C]	0.01	0.01	0.030	0.030	0.105	0.146	0.457	6.06

[A] r = repeatability = 2.83 (S_r) (within laboratories).
[B] R = reproducibility = 2.83 (S_r) (between laboratories).
[C] Material test levels for all three materials are essentially equal (99.93 − 100.0) for the 250–100 µm mesh tests. For 75 µm mesh, the test levels vary from 94.08 to 96.39.

where:
M_1 = mass residue, g, and
M_2 = mass sample, g.

7.1.1 In order to determine how much would be retained on a finer sieve, it is necessary to accumulate the mass of material collected on all the sieves and add it to the mass of material from the selected sieve.

7.2 The amount of material passing through each sieve is determined by subtracting the percent of the material collected on the sieve from 100.

8. Report

8.1 Report the following information:
8.1.1 Proper identification of samples,
8.1.2 Identification of each sieve used,
8.1.3 Percent retained on each sieve, and
8.1.4 Percent material passing through each sieve.

9. Precision and Bias [4]

9.1 This precision and bias section has been prepared in accordance with Practice D 4483. Refer to Practice D 4483 for terminology and other statistical details.

9.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers) used in the particular interlaboratory programs as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

9.3 A Type 1 (interlaboratory) precision was evaluated in 1986. Both repeatability and reproducibility are short term. A period of a few days separates replicate test results. A test result is the mean value, as specified by this test method, obtained on two determinations or measurements of the property or parameter in question.

9.4 Three different materials were used in the interlaboratory program. These were tested in seven laboratories on two different days.

9.5 The results of the precision calculations for repeatability and reproducibility are given in Table 1. Values for *r* and *R* are given for 250, 180, 150, and 75 µm mesh wet sieve tests.

9.6 *Repeatability*— The repeatability, *r*, of this test method has been established as the *appropriate value* tabulated in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or nonidentical sample populations.

9.7 *Reproducibility*— The reproducibility, *R*, of this test method has been established as the *appropriate value* tabulated in Table 1. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* (for any given level) must be considered to have come from different or nonidentical sample populations.

9.8 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values have not been evaluated for this test method. Bias, therefore, cannot be determined.

10. Keywords

10.1 sulfur; wet sieve

[4] Supporting data are available from ASTM Headquarters. Request RR: D11–1048.