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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Rubber Chemicals—Determination of Percent Insoluble Sulfur by Solvent Extraction[1]

This standard is issued under the fixed designation D 4578; 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 provide for the determination of solvent insoluble materials in a sulfur sample. The two test methods available are: (*1*) Test Method A, Extraction by Carbon Disulfide, and (*2*) Test Method B, Extraction by Toluene. If there are no other solvent insoluble materials present in the sulfur, these test methods determine the insoluble sulfur content directly. If other materials are also present, additional testing is necessary to identify what portion of the insolubles is insoluble sulfur.

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

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.* For a specific hazard statement, see Note in 5.1.

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]

3. Significance and Use

3.1 Test Method A is commonly used for quality control testing because of the solvent efficiency of carbon disulfide. Test Method B employs toluene as a solvent because it is less toxic and less volatile than carbon disulfide. Test Method B is more likely to be used for research and development or for any other purpose where such information may be useful.

TEST METHOD A—EXTRACTION BY CARBON DISULFIDE

4. Apparatus

4.1 *Filtering Crucible*, 30 cm^3 medium porosity.

4.2 *Filter Flask and Adapter for Crucible*.

4.3 *Circulating Air Oven*, at 60°C, explosion proof, vented.

5. Reagent

5.1 *Carbon Disulfide*, reagent grade.

Note: **Warning**—Carbon disulfide is a highly volatile, toxic, and flammable liquid. It must be utilized in a well ventilated chemical fume hood free of ignition sources. Rubber gloves should be worn when handling this solvent.

6. Procedure

6.1 Initially weigh a 30-cm^3 filtering crucible to ±0.001 g, and then accurately weigh to ±0.001 g, a 2 to 5-g specimen into the tared crucible.

6.2 Place the crucible in a suction flask in a well-vented hood and wash with 100 to 150 cm^3 of carbon disulfide. Adjust the suction so that the extraction time is at least 8 min.

6.3 Dry the specimen as completely as possible on the suction flask by continuing suction. Place the crucible in a vented, explosion-proof oven at 60°C for 1 h.

6.4 Remove, cool in a desiccator for 1 h, or to constant mass, and reweigh.

6.5 If insoluble materials other than insoluble sulfur are known to be present or suspected, place the crucible in a loosely capped jar and heat 16 h at 80°C in a vented, explosion-proof oven. Repeat steps 6.2 through 6.4 and obtain the mass after heating. This is the insoluble material other than insoluble sulfur.

7. Calculation

7.1 If only insoluble sulfur, *S*, is found, calculate the following, in percent:

$$S = 100 \times B/A \quad (1)$$

7.2 If other insoluble materials are present, calculate the following:

$$S = 100 \times B - C/A \quad (2)$$

where:

S = insoluble sulfur, %,
A = original specimen mass, g,
B = specimen mass after first extraction, g, and

[1] These test methods are under the jurisdiction of ASTM Committee D-11 on Rubber and are the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

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

[3] Supporting data are available from ASTM Headquarters. Request RR: D11-1051.

C = specimen mass after heating 16 h at 80°C followed by repeating the extraction procedure, g.

8. Report

8.1 Report the following information:

8.1.1 The proper identification of samples, and

8.1.2 The percent insoluble sulfur determined.

9. Precision and Bias [3]

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 Two different materials were used in the interlaboratory program. These were tested in six laboratories on two different days.

9.5 The results of the precision calculations for repeatability and reproducibility are given in Table 1, in ascending order of material average or level, for each of the materials evaluated.

9.6 The precision of this test method may be expressed in the format of the following statements which use an "appropriate value'' of *r*, *R*, (*r*), or (*R*), that is, that value to be used in decisions about test results (obtained with the test method). The *appropriate value* is that value of *r* or *R* associated with a mean level in Table 1 closest to the mean level under consideration at any given time, for any given material in routine testing operations.

9.7 *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.

9.8 *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.

9.9 Repeatability and reproducibility expressed as a percent of the mean level, (*r*) and (*R*), have equivalent application statements as above for *r* and *R*. For the (*r*) and (*R*) statements, the difference in the two single test results is expressed as a percent of the arithmetic mean of the two test results.

9.10 *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.

TEST METHOD B—EXTRACTION BY TOLUENE

10. Apparatus

10.1 *400-cm^3 Beaker and 90-mm Diameter Watch Glass.*

10.2 *Magnetic Stirrer and Teflon Coated Stirring Bars.*

10.3 *Filtering Crucible*, 50-cm^3, medium porosity.

10.4 *Filter Flask, 500 cm^3, and Adaptor for Crucible.*

10.5 *Circulating Air Oven*, explosion proof, vented, set at 70°C.

11. Reagent

11.1 *Toluene*, reagent grade.

12. Procedure

12.1 Accurately weigh to ±0.001 g approximately 2 g of a sulfur specimen in a 400-cm^3 beaker. Add 200 cm^3 toluene and a magnetic stirring bar. Cover with a watch glass and stir at a moderate rate for 30 min.

12.2 Weigh a clean, dry filtering crucible to ±0.001 g. Place the crucible in the adaptor, mounted on a vacuum suction flask in a fume hood. Remove the beaker from the magnetic stirrer and quantitatively transfer all the material from the beaker into the crucible. Wash the remaining filtrate in the crucible three times with 20 cm^3 portions of toluene.

12.3 Remove as much toluene as possible, using maximum suction. Transfer the crucible to the oven and dry for one hour at 70°C. Remove the specimen from the oven, cool in a dessicator, and weigh.

TABLE 1 Type 1 Precision Results—Insoluble Sulfur Content: Test Method A (Carbon Disulfide)

Material	Average	Within Laboratory[A]			Between Laboratory[A]		
		S_r	r	(r)	S_R	R	(R)
Oil Treated, 90 % Insoluble Sulfur—A	74.39	0.1500	0.4245	0.571	0.2154	0.6096	0.820
Oil Treated, 90 % Insoluble Sulfur—B	76.14	0.0913	0.2583	0.339	0.0922	0.2609	0.343
Pooled Values[B]	75.19	0.1242	0.3514	0.467	0.1794	0.5077	0.675

[A] S_r = repeatability standard deviation.
r = repeatability = 2.83 × the square root of the repeatability variance.
(r) = repeatability (as a percentage of material average).
S_R = reproducibility standard deviation.
R = reproducibility = 2.83 × the square root of the reproducibility variance.
(R) = reproducibility (as a percentage of material average).
[B] No values omitted.

13. Calculation

13.1 If only insoluble sulfur is found, calculate the following:

$$S = 100 \times B/A \quad (3)$$

13.2 If other insoluble materials are present, calculate the following:

$$S = 100 \times B - C/A \quad (4)$$

where:

S = insoluble sulfur, %,
A = original specimen mass, g,
B = specimen mass after first extraction, g, and
C = specimen mass after heating 16 h at 80°C followed by repeating the extraction procedure, g.

14. Report

14.1 Report the following information:

14.1.1 The proper identification of samples, and

14.1.2 The percent insoluble sulfur determined.

15. Precision and Bias

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

16. Keywords

16.1 insoluble sulfur; sulfur