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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 Extraction¹

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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 Industries²

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 cm³ 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-cm³ 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 cm³ 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.