



Designation: D 4573 – 98

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Rubber Chemicals—Determination of Oil Content in Oil-Treated Sulfur¹

This standard is issued under the fixed designation D 4573; 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 determination of the amount of hydrocarbon oils added to oil-treated sulfurs. The test method is employed when the amount of oil added is 1 % or more of the total sample.

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 Industries²

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *lot sample*—a production sample representative of a standard production unit, normally referred to as “the sample.”

3.1.2 *specimen*—also known as the “test portion;” it is the actual material used in the analysis; it must be representative of the lot sample.

4. Significance and Use

4.1 This test method measures the hydrocarbon oils added to sulfur to help control dusting of the sulfur. It can be used for research, development, and quality control to measure the level of oil added to the sulfur to help maintain the oil content at required levels.

5. Apparatus

5.1 *Filtering Crucible*, 30 cm³, medium porosity.

5.2 *Filter Flask*, and adapter for crucible.

5.3 *Circulating Air Oven*, capable of 70 ± 2°C explosion proof, vented.

5.4 *Desiccator*.

5.5 *Precision Balance*, or scale sensitive to 0.001 g.

5.6 *Conical Flask*, 250 cm³.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Fine Particle Size Pure Sulfur* (45 μm or smaller).

6.3 *Hexane Saturated With Sulfur:*

6.3.1 *Method of Preparation*—In a suitable size container equipped with an air stirrer and external heater, mix hexane and sulfur for 2 h while heating to 40°C maximum. Continue to stir while the mixture cools to room temperature. Allow to settle for 12 h. Decant off the sulfur saturated hexane, and filter just prior to use in the test. Store in a capped bottle.

7. Procedure

7.1 Weigh a 30 cm³ filtering crucible to the nearest 0.001 g.

7.2 Weigh 5 + 0.5 g of the oil treated sulfur specimen into a 250 cm³ conical flask. Add 20 cm³ of sulfur-saturated hexane. Swirl the flask gently to disperse the sulfur specimen. Filter this mixture through the previously tared 30 cm³ filtering crucible contained in the holder on a vacuum filter flask in a well-ventilated hood, adjusting the suction to pull the filtrate through the crucible at a rate forming drops and not a stream of liquid. Wash the residue in the flask with 100 cm³ sulfur-saturated hexane and quantitatively transfer the washings to the filtering crucible with an additional 50 cm³ sulfur-saturated hexane. Use 100 to 150 cm³ of solution for the total test. The extraction time should take between 8 and 15 min to complete. Never allow the sulfur cake in the crucible to dry and crack prior to completely adding all of the extracting solvent.

7.3 After adding all the solvent, increase the suction and dry the cake. Leave the vacuum on for 30 s after the last drop of solvent drips off the cake.

7.4 Remove the crucible from the flask and place in the

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 *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.