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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Rubber Chemicals—Determination of Oil Content in Oil-Treated Sulfur[1]

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 Industries[2]

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 cm^3, 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 cm^3.

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 cm^3 filtering crucible to the nearest 0.001 g.

7.2 Weigh 5 + 0.5 g of the oil treated sulfur specimen into a 250 cm^3 conical flask. Add 20 cm^3 of sulfur-saturated hexane. Swirl the flask gently to disperse the sulfur specimen. Filter this mixture through the previously tared 30 cm^3 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 cm^3 sulfur-saturated hexane and quantitatively transfer the washings to the filtering crucible with an additional 50 cm^3 sulfur-saturated hexane. Use 100 to 150 cm^3 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

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[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.

70°C oven for 1 h. Remove and cool in a desiccator for 0.5 h or more. Weigh it to the nearest 0.0001 g.

7.5 Repeat steps described in 7.1-7.4 on a 5.0-g sample of pure sulfur as a blank to obtain a correction factor for the oil-treated sulfur samples. The correction for the blank may be positive or negative.

8. Calculation

8.1 The formula for calculating percent oil extracted from an oil-treated sulfur sample, *A*, is as follows:

$$A = 100\,[1 - (W_2 / W_1 + C)] \quad (1)$$

$$C = 1 - \frac{B_2}{B_1} \quad (2)$$

where:

B_1 = pure sulfur weighed as blank, g,
B_2 = sulfur remaining after extracting blank, g,
C = correction factor for blank,
W_1 = oil-treated sulfur sample, g, and
W_2 = sulfur remaining after extraction, g.

9. Report

9.1 Report the following information:

9.1.1 Proper identification of samples, and

9.1.2 Percent of extracted oil from two individual determinations and their average to the nearest 0.01 %.

10. Precision and Bias [4]

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

10.2 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.

10.3 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 method, obtained on two determinations or measurements of the property or parameter in question.

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

10.5 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.

10.6 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.

10.7 *Repeatability*—The repeatability, *r*, of this test method has been established as the *appropriate value* tabulated in the Precision Table. 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.

10.8 *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.

10.9 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.

10.10 *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.

11. Keywords

11.1 oil content; oil-treated sulfur; rubber chemicals; sulfur

[4] Supporting data are available from ASTM Headquarters. Request RR: D11-1049.

TABLE 1 Type 1 Precision Results—Oil Content (Sulfur)

Material	Average	Within Laboratory[A]			Between Laboratory[A]		
		S_r	r	(r)	S_R	R	(R)
Oil Treated, 90 % Insoluble Sulfur—B	17.84	0.175	0.496	2.78	1.081	3.060	17.1
Oil Treated, 90 % Insoluble Sulfur—A	20.32	0.247	0.699	3.44	0.360	1.020	5.0
Pooled Values[B]	18.97	0.211	0.597	3.14	0.806	2.281	12.0

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

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