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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Rubber Chemicals—Determination of Acidity in Sulfur¹

This standard is issued under the fixed designation D 4569; 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 acid material, which disassociates in distilled water, that is present in sulfur. The acidity is determined by an electrometric or visual titration.

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. Significance and Use

3.1 This test method provides a means of determining the acidity of sulfur and may be used for quality control, research and development, or for any other reason for which such a determination is required. A low acidity value indicates to the user that the sulfur used should not adversely affect the vulcanization system.

4. Apparatus

4.1 *pH Meter*, equipped with a glass measuring electrode and calomel reference electrode, operated according to the manufacturer's directions for optimum performance.

4.2 *Ten-cm³ Burette*, graduated to 0.05 cm³.

4.3 *Laboratory Glassware*, suitable for carrying out the procedure as written.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that

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.

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.

5.2 *Bromothymol Blue*—Dissolve 0.1 g of 3',3"-dibromothymolsulfonphthalein in 100 cm³ of 20 % methanol or use commercial preparations.

5.3 *Sodium Hydroxide*, 0.01 N.

6. Procedure

6.1 Weigh 10.0 g of sulfur into a 500-cm² beaker, and wet the sample with 25 cm³ of alcohol.

6.2 Add 200 cm³ of distilled water, and stir the wetted sulfur thoroughly.

6.3 Place the pH meter electrode in the solution and titrate with 0.01 N NaOH to a pH of 7.0. Stir the mixture constantly while titrating. When nearing the end of the titration, wait until the pH reading has reached equilibrium before adding additional NaOH solution to prevent overshooting the end point.

6.4 If a pH meter is unavailable, add 10 drops of bromothymol blue indicator and titrate as above.

6.5 **Important:** Titrate a blank (no sulfur) using the same amounts of alcohol and water to a pH 7.0 end point. Subtract the amount used for the blank from the amount used to titrate a sulfur sample in order to determine the acidity of the sulfur sample.

7. Calculation

7.1 Calculate the percent acidity, A, (as H₂SO₄) in accordance with the following equation:

$$A = 4.9 \times V \times N/W \quad (1)$$

where:

V = volume of NaOH for sample-volume NaOH for blank, cm³,

N = normality of NaOH solution, and

W = sample mass, g.

8. Report

8.1 Report the following information:

8.1.1 Percent acidity as H₂SO₄.

8.1.2 If the pH of the sulfur solution is greater than 7.0 and no titrant is needed, report the result as basic.