



Standard Test Methods for Silanes Used in Rubber Formulations (bis-(triethoxysilylpropyl)sulfanes): Sulfur Content¹

This standard is issued under the fixed designation D 6741; 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 sulfur content of silanes, or of admixtures of silane and carbon black, of the type bis-(triethoxysilylpropyl)sulfane. The following test methods are included:

Test Method A: Combustion in an Oxygen Flask and Subsequent Titration of Sulfate

Test Method B: High-Temperature Combustion with Infrared Absorption Detection

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water²

3. Significance and Use

3.1 The sulfur content can be used to characterize a silane or an admixture of silane and carbon black. It depends mainly on the average length of the sulfur chain.

Test Method A: Combustion in an Oxygen Flask and Subsequent Titration of Sulfate

4. Summary of Test Method

4.1 The weighed sample is burned in an oxygen atmosphere. The resulting sulfur dioxide and trioxide is absorbed in hydrogen peroxide solution, and the sulfate ions thus generated are titrated with barium perchlorate solution employing Thorin indicator. Taking into account the known concentration of the perchlorate solution, the volume consumed, and the sample mass, the sulfur content of the sample can be calculated.

5. Apparatus

5.1 *Analytical Balance*, accuracy ± 0.1 mg.

5.2 *Combustion Apparatus*, according to Schoeniger, with 750 cm³ flask.

5.3 *Ultrasonic Bath*.

5.4 *Automatic Titrator*.

5.5 *Photoelectrical Detector*, set at 550 nm.

5.6 *Volumetric Cylinder*, 100 cm³.

5.7 *Volumetric Flask*, 100 cm³.

5.8 *Volumetric Pipet*, 10 cm³.

5.9 *Syringe*, 0.5 cm³.

5.10 *Titration Beakers*.

5.11 *Magnetic Stirrer*, with stirring rod.

5.12 *Filter Paper*, for Schoeniger combustion (35 $\times$ 35 $\times$ 70 mm).

5.13 *Glass Tube*, attached to a rubber hose.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall 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 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

6.3 *2-propanol* > 99 %.

6.4 *Hydrogen Peroxide Solution*, (5 %) as absorbing liquid.

6.5 *Barium Perchlorate Solution*, 0.005 mol/dm³ in 2-propanol/water.

6.6 *Thorin Solution*, 0.1 % in water (Thorin octahydrate).

6.7 *Sulfuric Acid*, as a standard solution (1.000 $\pm$ 0.002 g SO₄²⁻).

6.8 *Deionized Water*.

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.20 on Compounding Materials and Procedures.

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² *Annual Book of ASTM Standards*, Vol 11.01.

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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.