



Designation: D6741 – 10 (Reapproved 2023)

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

This standard is issued under the fixed designation D6741; 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 These test methods cover 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 standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D1193 Specification for Reagent Water

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

¹ These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and are the direct responsibility of Subcommittee D11.20 on Compounding Materials and Procedures.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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 mm by 35 mm by 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