



Designation: D6740 – 15 (Reapproved 2023)

Standard Test Method for Silanes Used in Rubber Formulations (bis-(triethoxysilylpropyl)sulfanes): Residue on Ignition¹

This standard is issued under the fixed designation D6740; 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 residue on ignition of silanes of the type bis-(triethoxysilylpropyl)sulfane, or of admixtures of these silanes and solid carriers, such as carbon black, waxes, or organic polymers.

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, 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

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

3. Summary of Test Method

3.1 In this test method, a sample of the silane is treated with hydrochloric acid and then ignited in a muffle furnace at 1000 °C. The mass retained after this procedure is called the residue on ignition. It is expressed in percentage of the initial mass.

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

Current edition approved Nov. 1, 2023. Published December 2023. Originally approved in 2001. Last previous edition approved in 2019 as D6740 – 15 (2019). DOI: 10.1520/D6740-15R23.

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.

4. Significance and Use

4.1 The residue on ignition, which consists essentially of SiO₂, is related to the silicon content of the silane and may be used to verify the composition of the silane.

5. Apparatus

5.1 *Analytical Balance*, accuracy ± 0.1 mg.

5.2 *Hot Plate or Ventilated Oven*, placed in a fume hood.

5.3 *Muffle Oven*, (1000 °C).

5.4 *Porcelain Crucible (High-Form)*, capacity 25 cm³.

5.5 *Beaker*, 500 cm³.

5.6 *Graduated Pipet*, 20 cm³.

5.7 *Graduated Cylinder*, 250 cm³.

5.8 *Glass Rod*.

5.9 *Desiccator*.

6. Reagents

6.1 *Hydrochloric Acid*, 37 %, analytical grade.

6.2 *Sulfuric Acid*, 96 %, analytical grade.

6.3 *Deionized Water*.

6.4 *Silica Glass Wool*.

7. Procedure

NOTE 1—Preheat porcelain crucibles and silica glass wool used for the test for 2 h at 1000 °C in a muffle furnace. Cool to room temperature and store in a desiccator until needed for the test.

7.1 *Liquid Silanes:*

7.1.1 Weigh (tare) a predried crucible and glass wool plug. Remove the glass wool and tare the crucible alone.

7.1.2 Weigh approximately 1 g of the liquid silane into the pre-treated crucible to the nearest 0.1 mg.

7.1.3 Add 2 cm³ of concentrated (96 % nominal) sulfuric acid and homogenize by careful agitation.

7.1.4 Cover the mixture with pre-treated silica glass wool.

7.1.5 Place the crucible into a ventilated oven, placed in a fume hood, and raise the temperature to 125 °C.

NOTE 2—The oven may be replaced by an electrical heating plate if the former is not available.