Designation: D 6740 – 01

Standard Test Method for Silanes Used in Rubber Formulations (bis-(triethoxysilylpropyl)sulfanes): Residue on Ignition[1]

This standard is issued under the fixed designation D 6740; 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Method

2.1 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.

3. Significance and Use

3.1 The residue on ignition, which consists essentially of SiO_2, is related to the silicon content of the silane and may be used to verify the composition of the silane.

4. Apparatus

4.1 *Analytical Balance*, accuracy ±0.1 mg.
4.2 *Simon-Mueller Oven*, or equivalent (850°C).
4.3 *Muffle Oven*, (1000°C).
4.4 *Porcelain Crucible (High-Form)*, capacity 25 cm^3.
4.5 *Beaker*, 500 cm^3.
4.6 *Graduated Pipet*, 20 cm^3.
4.7 *Graduated Cylinder*, 250 cm^3.
4.8 *Glass Rod*.
4.9 *Desiccator*.

5. Reagents

5.1 *Hydrochloric Acid*, 37 %, analytical grade.
5.2 *Sulfuric Acid*, 96 %, analytical grade.
5.3 *Deionized Water*.
5.4 *Silica Glass Wool*.

6. Preparation of 90 % Sulfuric Acid

6.1 Pipet 20 cm^3 of deionized water into 500 cm^3 beaker contained in an ice-water bath.

6.2 While stirring with a glass rod, add, very slowly, in several steps, 250 cm^3 of 96 % sulfuric acid.

NOTE 1—Adding acid too fast will generate excessive heat in the mixture and may result in acid splashes. Handle carefully.

6.3 Cool to room temperature.

6.4 Transfer the mixture into a suitable glass bottle.

7. Procedure

NOTE 2—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 cm^3 of 90 % 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 Simon-Mueller oven, mounted in a fume-cupboard, and raise the temperature to 125°C.

NOTE 3—The Simon-Mueller oven may be replaced by an electrical heating plate if the former is not available.

7.1.6 When most of the acid has been evaporated (usually after 1 h), transfer the crucible into a muffle furnace, previously brought to 1000°C.

7.1.7 Heat for 2 h at 1000°C.

7.1.8 If the ash is gray or black, continue heating for an additional 2 h in the muffle furnace at 1000°C.

7.1.9 Allow to cool in a desiccator and weigh to the nearest 0.1 mg.

[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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7.2 *Admixtures of Silanes and Carbon Black*:

7.2.1 Weigh (tare) a pre-dried crucible and glass wool plug. Remove the glass wool and tare the crucible alone.

7.2.2 Weigh approximately 1 g of the silane/carbon black admixture into the pre-treated crucible to the nearest 0.1 mg.

7.2.3 Add 2 cm^3 of 37 % hydrochloric acid and homogenize by careful agitation.

7.2.4 Cover the contents with the respective glass wool plug.

7.2.5 Place the crucible into a Simon-Mueller oven, mounted in a fume-cupboard, and raise the temperature to 125°C.

NOTE 4—The Simon-Mueller oven may be replaced by an electrical heating plate if the former is not available.

7.2.6 When the major part of the acid has been evaporated (usually after 1 h), transfer the crucible into a muffle furnace, previously brought to 1000°C.

7.2.7 Heat for 2 h at 1000°C.

7.2.8 If the ash is gray or black, continue to heat the crucible for an additional 2 h in the muffle furnace at 1000°C.

7.2.9 Allow to cool in a desiccator and weigh to the nearest 0.1 mg.

8. Calculation

8.1 Calculate the residue on ignition to the nearest 0.01 % as follows:

$$\text{Residue on ignition} = \frac{(C - A)}{(B - A)} \times 100 \quad (1)$$

where:

A = mass of crucible and silica glass wool, g,
B = mass of crucible, silica glass wool plus the sample, g, and
C = mass of crucible, silica glass wool plus the residue on ignition, g.

9. Report

9.1 Report the following information:

9.1.1 Proper identification of the sample, and

9.1.2 The average of two single determinations, reported to the nearest 0.01 %.

10. Keywords

10.1 ash; organosilanes; silanes