Designation: D6740 – 15

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

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 and health practices and determine the applicability of regulatory limitations prior to use.*

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.

4. Significance and Use

4.1 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.

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 cm^3.

5.5 *Beaker,* 500 cm^3.

5.6 *Graduated Pipet,* 20 cm^3.

5.7 *Graduated Cylinder,* 250 cm^3.

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 cm^3 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.

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.

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[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] 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.

TABLE 1 Residue on Ignition (%)

Material	Average[A]		Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	$S_{\bar{x}}$	S_r	S_R	r	R
A	22.86	0.17	0.12	0.20	0.35	0.57

[A] The average of the laboratories calculated averages.

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

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

NOTE 3—The 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. Precision and Bias[3]

10.1 The precision of this test method is based on an interlaboratory study conducted in 2008. Seven laboratories participated in this study. Each of the labs reported four replicate test results for a single material. Every "test result" reported represents an individual determination. Except for the use of only a single material, Practice E691 was followed for the design and analysis of the data.

10.1.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

10.1.1.1 Repeatability limits are listed in Table 1.

10.1.2 *Reproducibility limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

10.1.2.1 Reproducibility limits are listed in Table 1.

10.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

10.1.4 Any judgment in accordance with statement 10.1.1 or 10.1.2 would have an approximate 95 % probability of being correct.

10.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

10.3 The precision statement was determined through statistical examination of 28 results, from six laboratories, on one material. Due to the small number of participating labs, no outliers were removed. This material was described as follows: Material A is a commercially available bis-(triethoxysilylpropyl)tetra sulfane.

11. Keywords

11.1 ash; organosilanes; silanes

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[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1102.

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