



# Standard Test Method for Silanes Used in Rubber Formulations (bis-(triethoxysilylpropyl)sulfanes): Characterization by High Performance Liquid Chromatography (HPLC)<sup>1</sup>

This standard is issued under the fixed designation D 6844; 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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

## 1. Scope

1.1 This test method covers the characterization of silanes, or of admixtures of silane and carbon black (see 10.4), of the type bis-(triethoxysilylpropyl)sulfane by high performance liquid chromatography.

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 5297 Test Methods for Rubber Chemical Accelerator-Purity by High Performance Liquid Chromatography<sup>2</sup>

E 682 Practice for Liquid Chromatography Terms and Relationships<sup>3</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1  $S_x$ —Bis-(triethoxysilylpropyl)polysulfane or polysulfide,  $(EtO)_3SiC_3H_6S_xC_3H_6Si(OEt)_3$

3.1.2  $S_2$ —Bis-(triethoxysilylpropyl)disulfane or disulfide,  $(EtO)_3SiC_3H_6S_2C_3H_6Si(OEt)_3$

3.1.3  $S_3$ —Bis-(triethoxysilylpropyl)trisulfane or trisulfide,  $(EtO)_3SiC_3H_6S_3C_3H_6Si(OEt)_3$

3.1.4  $S_3$ —Bis-(triethoxysilylpropyl)tetrasulfane or tetrasulfide,  $(EtO)_3SiC_3H_6S_4C_3H_6Si(OEt)_3$

3.1.5  $S_3$ —Bis-(triethoxysilylpropyl)pentasulfane or pentasulfide,  $(EtO)_3SiC_3H_6S_5C_3H_6Si(OEt)_3$

3.1.6  $S_3$ —Bis-(triethoxysilylpropyl)hexasulfane or hexasulfide,  $(EtO)_3SiC_3H_6S_6C_3H_6Si(OEt)_3$

3.1.7  $S_3$ —Bis-(triethoxysilylpropyl)heptasulfane or heptasulfide,  $(EtO)_3SiC_3H_6S_7C_3H_6Si(OEt)_3$

3.1.8  $S_3$ —Bis-(triethoxysilylpropyl)octasulfane or octasulfide,  $(EtO)_3SiC_3H_6S_8C_3H_6Si(OEt)_3$

3.1.9  $S_3$ —Bis-(triethoxysilylpropyl)nonasulfane or nonasulfide,  $(EtO)_3SiC_3H_6S_9C_3H_6Si(OEt)_3$

3.1.10  $S_3$ —Bis-(triethoxysilylpropyl)decasulfane or decasulfide,  $(EtO)_3SiC_3H_6S_{10}C_3H_6Si(OEt)_3$

3.1.11 *average sulfur chain length*—the weighted average of the sulfur bridge in the polysulfide mixture. Includes  $S_2$  to  $S_{10}$  species.

## 4. Summary of Test Method

4.1 A sample of the silane is analyzed by high performance liquid chromatography to determine amounts of each component, the average chain length and the amount of dissolved elemental sulfur.

4.2 Two methods are described: Method A with a constant composition of the mobile phase (isocratic), and Method B using a gradient. Both methods will give similar chromatograms.

## 5. Significance and Use

5.1 The average sulfur chain length is an important parameter in determining the behavior of the silane in a rubber mixture.

## 6. Apparatus

6.1 *HPLC with UV Detector*, operating at 254 nm, Inlet Valve with 5 mm<sup>3</sup> (μL) loop, integrator or data system.

6.2 *Column C18*, 5 μm, 4.6 × 250 mm.

6.3 *Column Oven*.

6.4 *Analytical Balance*, accuracy ±0.1 mg.

6.5 *Hamilton Syringe*, 100 mm<sup>3</sup> (μL).

6.6 *Volumetric Pipet*, 5 cm<sup>3</sup>.

6.7 *Volumetric Flasks*, 50 and 2000 cm<sup>3</sup>.

6.8 *Syringe*, 3 cm<sup>3</sup> or 5 cm<sup>3</sup>.

6.9 *Glass Bottles*, 5 cm<sup>3</sup>.

6.10 *Disposable PTFE Filters*, 0.20 μm, d = 25 mm.

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 09.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.06.