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.

Designation: D6844 – 10 (Reapproved 2023)

Standard Test Method for Silanes Used in Rubber Formulations (bis-(triethoxysilylpropyl)sulfanes): Characterization by High Performance Liquid Chromatography (HPLC)[1]

This standard is issued under the fixed designation D6844; 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 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 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]

D5297 Test Methods for Rubber Chemical Accelerator—Purity by High Performance Liquid Chromatography

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

E682 Practice for Liquid Chromatography Terms and Relationships

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

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.

[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.

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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.

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