



Standard Test Method for Silanes Used in Rubber Formulations (bis-(triethoxysilylpropyl)sulfanes): Characterization by Gas Chromatography (GC)¹

This standard is issued under the fixed designation D6843; 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 of the type bis-(triethoxysilylpropyl)sulfanes by gas 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:*²

D4626 Practice for Calculation of Gas Chromatographic Response Factors

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

E355 Practice for Gas 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 *PTES*—Propyltriethoxysilane $\text{CH}_3\text{CH}_2\text{CH}_2\text{Si}(\text{OEt})_3$

3.1.2 *Cl-PTES*—Chloropropyltriethoxysilane $\text{Cl-CH}_2\text{CH}_2\text{CH}_2\text{Si}(\text{OEt})_3$

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.

4. Summary of Test Method

4.1 In this test method, a sample of the silane is analyzed by gas chromatography in order to determine the amount of volatile components. From the peak areas in the chromatogram, the percents by weight of volatiles are totaled and designated as the total volatile impurities or volatile by-products.

5. Significance and Use

5.1 The amount of volatile components reflects the impurity level in the product, and as a consequence, its behavior in a rubber mixture.

6. Apparatus

6.1 *Gas Chromatograph*, equipped with:

6.1.1 *Flame Ionization Detector (FID)*.

6.1.2 *Capillary Column*, typical is 30 m length, 0.25 to 0.53 mm internal diameter, fused silica, 0.1 to 1.0 μm film thickness.

6.1.3 *Carrier Gas Flow Control*, with splitter.

6.1.4 *Temperature Controls*, for injector, detector and column.

6.2 *Syringe*, 1 mm³ (μL).

6.3 *Analytical Balance*, accuracy ±0.1 mg.

6.4 *Automatic Pipets*, 0.2 to 1.0 cm³, 5 cm³.

6.5 *Sample Vials*, approximately 15 cm³.

7. Reagents

7.1 *Methanol*, analytical grade (for cleaning syringe).

7.2 *Undecane*,³ analytical grade (used as internal standard).

7.3 *Optional: Cyclohexane*, analytical grade (used to dilute the sample).

7.4 *Helium*, minimum 99.99 % purity, suitable for chromatographic use, dried (carrier gas).

7.5 *Hydrogen Gas*, minimum 99.99 % purity, total hydrocarbons ≤1 ppm (for detector).

³ Similar hydrocarbons like decane, analytical grade, can be used in place of undecane, as long as they do not interfere with peaks from the sample.