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: F2466 – 10 (Reapproved 2018)

Standard Practice for Determining Silicone Volatiles in Silicone Rubber for Transportation Applications[1]

This standard is issued under the fixed designation F2466; 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 practice covers a means to determine the percent silicone-producing volatiles present in heat-cured silicone rubber and room temperature-cured silicones (RTV).

1.2 Silicone-producing volatiles contribute to fouling of oxygen sensor systems used in the control of vehicle emissions.

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]

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

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 Practice

3.1 This practice consists of four (4) basic steps: (*1*) the silicone is cured to its elastomeric form, (*2*) the volatiles are extracted from the cured material, (*3*) the extract is separated and measured by gas chromatography (GC), and (*4*) the GC results are quantified using a siloxane calibration.

4. Significance and Use

4.1 Use of this practice in conjunction with realistic maximum volatility tolerance level can help minimize the risk of oxygen sensor dysfunction from formed-in-place-sealants in transportation applications. This practice provides a method for determination of percentage volatiles in silicone elastomers. The volatile silicones from a commercial silicone are primarily cyclo dimethyl-siloxane. Other species present having GC retention times similar to those of the cyclics are assumed to be silicone as well.

5. Apparatus

5.1 *Gas Chromatograph,* fused silica capillary column system equipped with a flame ionization detector, split-type capillary column injector, temperature programming capability and an appropriate data recording system. An alternative unit may be an equivalent instrument equipped with a thermal conductivity detector, or as agreed upon between producer and user. Specific column and operating conditions should be selected to optimize instrument response and chromatographic resolution, particularly separation of the internal standard from extracted sample components.

5.2 *Column,* suggested to be used is 30 to 60 m by 0.25 mm with 0.25 to 1.5 µm DB-1 or DB-5 fused silica capillary column or equivalent.

5.3 Operating conditions are:

5.3.1 *Column*—50 to 320°C at 10°C/min (a post-analysis period may be required to elute higher boiling components prior to subsequent analyses).

5.3.2 *Injector*—290°C.

5.3.3 *Detector*—325°C.

5.3.4 *Sample Size*—1 µL.

5.3.5 *Injector Split Ratio*—2:1 to 50:1 (adjusted as needed).

5.3.6 *Helium or Nitrogen,* for the carrier gas.

5.3.7 *Carrier Gas Flow Velocity*—1 to 2 mL/min (adjusted as needed for column dimensions).

5.4 *Humidity Chamber,* or controlled lab environment.

5.5 *Wrist-Action Mechanical Shaker.*

[1] This practice is under the jurisdiction of ASTM Committee F03 on Gaskets and is the direct responsibility of Subcommittee F03.50 on Analytical Test Methods.

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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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5.6 *Analytical Balance,* with glass draft shield capable of 0.0001 g accuracy.

5.7 *30-mL Vials,* flint glass, with screw cap (polyethylene lined).

5.8 *Syringe,* capable of accurately delivering 20 ± 0.1 µL (no plastic elements used due to solvents used).

5.9 Solvents and standards used are pentane (99 %) and dodecane (99 %), both spectral grade.

5.10 *Rigid Plates (Glass or Aluminum),* 0.90 mm thick, for cutting the wet formed-in-place sealant.

5.11 Automated devices shall be used for measuring and calculating peaks.

6. Test Specimens

6.1 Heat-cured silicone rubber samples shall be procured from either actual production parts, or shall be compression-molded ASTM tensile plaques (Practice D3182, 2.0 ± 0.2 mm thick). Cure conditions of the tensile plaques shall mirror cure conditions used on the production parts. If actual production parts are used to obtain test samples, best practice would be to cut sample so that it is not thicker than the above stated tensile plaque thickness.

6.2 Room temperature-vulcanized (RTV) samples shall be prepared by spreading the liquid using a suitable device, into consistent 0.90 ± 0.20 mm plaques. Avoid entrapped air and knit lines when preparing the sample.

6.3 Three 1-g samples shall be cut from the plaque. These samples shall be taken from near one corner, at the center of the plaque, and near the corner at a diagonal from the first.

7. Standard Solutions[3]

7.1 Add 0.1 g (weighed to the nearest 0.1 mg) of each pure cyclic (>98 %) to 1.0 g of dodecane (99 %) (weighed to the nearest 0.1 mg). Ten millilitres ± 0.1 mL pentane is added and the container is sealed to prevent leakage/evaporation. New standard mixtures should be prepared if existing one is more than seven (7) days old.

7.2 Calibration of the standard solution is achieved by injecting 1 µL (need verify use with SE 30 column – will need to attenuate response or dilute solution) standard solution sample. Response factors for the individual cyclics are calculated using the following equation:

$$RfDn = \frac{Wt\,Dn}{ADn} * \frac{ADoD}{WtDoD} \quad (1)$$

where:

Rf = response factor
Dn = the cyclic siloxane species from a 4 member to a 10 member ring
RfDn = the response factor for each siloxane species from 4 to 10
WtDn = the weight of each siloxane species from 4 to 10 used in the standard solution
ADn = the area under the curve for each siloxane species from 4 to 10
DoD = the dodecane standard, which is arbitrarily given a response factor of "1" (one), and is used as the basis for calculating the response factors of the various know and unknow siloxane species
ADoD = the area under the curve for the dodecane standard
WtDoD = the weight of the dodecane used in the standard solutions

7.3 Response factors for cyclic species vary in a relatively linear manner from D_5 through D_{10}, so that response factors for cyclics not in the standard solution can be calculated from the known response factors of the cyclics in the standard solution. A sample calculation for response factors of standards available, and a Linear Least Squares Analysis to determine response factors of cyclics that are unavailable can be found in Appendix X1.

7.4 All of the unknowns that appear in the analysis (between D_4 and D_{10}) are assumed to be dimethyl siloxanes. All unknowns are given, as response factors, the average response factor calculated for the difunctional cyclosiloxane monomers D_4 through D_{10}.

8. Conditioning

8.1 Allow RTV samples to cure for 24 h, but not to exceed 72 h at 25°C and 50 ± 10 % relative humidity.

9. Procedure

9.1 *Extraction*—Pre-weigh each cured sample to 1.0 ± 0.2 g (record weight to the nearest 0.0001 g) and set aside.

9.2 Weigh 0.010 ± 0.005 g of dodecane (record weight to the nearest 0.0001 g) and place sample into the 30-mL vial. To this add 10 mL of pentane. Immediately place the pre-weighed sample into the vial, and seal the container to prevent leakage/evaporation. Weight precision of the dodecane and test sample are extremely important for reproducible results. The sample vial is placed on a wrist shaker for 16 h.

NOTE 1—The sequence is important due to the volatility of the solvents. used.

NOTE 2—See 10.1.1 regarding dodecane measurement.

9.3 Inject 1 to 5 µL into the GC injection port. (Injection volume is dependant on the injector split ratio).

9.4 After the elution is complete (about 35 min) identify the peaks and quantify them by integration using the following equations (sample calculations are shown in Appendix X2):

$$\%Dn = \frac{RfDn*ADn}{ADoD} * \frac{WtDoD}{SaWt} * 100 \quad (2)$$

where

SaWt = the weight of the silicone part

9.4.1 Perform Eq 2 for D_4 through D_{10}.

[3] The sole source of supply of the standards solutions known to the committee at this time is Ohio Valley Specialty Chemicals, 115 Industrial Road, Marietta, OH, 45750, 1-800-729-6972, Catalog number 34569/Cyclic Standard Kit D3 through D10. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

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