



Test for Volatiles in Silicone Rubber

1 Introduction

Note: Nothing in this standard supercedes applicable laws and regulations.

Note: In the event of conflict between the English and domestic language, the English language shall take precedence.

1.1 Purpose. Measure the percent silica producing volatiles present in liquid injection molded silicone, heat cured silicone rubber or one component room temperature vulcanizing (RTV) silicone sealant regardless of cure system.

1.2 Applicability. Silica producing volatiles are contributors to the fouling of oxygen sensor systems used to control vehicle exhaust emissions. Encapsulating electronics in silicone liquid injected molded products may result in these low molecular weight extractable (cyclic dimethyl-siloxane) disrupting electrical function. As a contributing source of contamination, the volatile potential of silicone can be accurately monitored using this procedure. Utilizing this procedure in conjunction with a realistic maximum volatility tolerance level for end use silicones can minimize risk of component dysfunction.

1.2.1 Consistency. To avoid variation in measurements to production parts or sealant, and when comparing test results, the following must be strictly controlled:

- Time from molding to test (3.3).
- Extraction or shake time (5.2.1).
- Specific gravity (5.3.2) of the silicone part or sealant evaluated must be used to determine compliance to the maximum percent specified on the release print.

1.3 Remarks. This procedure consists of the following four (4) basic steps:

1. The silicone sealant is cured to its elastomeric form and molded parts are sampled.
2. The volatiles (cyclic dimethyl-siloxane) are extracted from the cured silicone.
3. The extract is separated and measured by gas chromatography (GC).
4. The GC results are quantified using a siloxane standard solution calibration.

2 References

Note: Only the latest approved standards are applicable unless otherwise specified.

2.1 External Standards/Specifications.

ASTM E288

2.2 GM Standards/Specifications.

9986350

2.3 Additional References.

- "Evaluating the Measurement Process", D.J. Wheeler and R.W. Layday, c. 1984
- Fisher Scientific Catalog
- International Union of Pure and Applied Chemistry (IUPAC) Nomenclature, Kirk-Othmer Encyclopedia of Chemical Technology, Volume. 20, 5th Edition, c. 2006
- Minitab, Inc., - (www.minitab.com)
- Ohio Valley Specialty Chemical, 115 Industrial Road, Marietta, Ohio, USA 45750

- Petrarch Systems Inc., 2731 Bartram Road, Bristol, Pennsylvania USA 19007
- Van Waters and Rogers Catalog - (www.vwr.com)
- Varian, Perkin-Elmer or Hewlett Packard

3 Resources

3.1 Facilities. A suitable area or room for conducting testing with a stable and known environment.

3.1.1 Calibration. The test facilities and equipment shall be in good working order and shall have a valid calibration label.

3.1.2 Alternatives. Alternative test facilities and equipment may also be used. However, all measuring variables as specified in this standard shall be determined correctly with respect to their physical definition.

3.2 Equipment.

3.2.1 GC equipped with a thermal conductivity or flame ionization detector and linear temperature programmer. Varian, Perkin-Elmer or Hewlett-Packard to provide this equipment.

3.2.1.1 Carrier Gas. Helium or Nitrogen.

3.2.2 Column. A column capable of separating cyclosiloxanes, e.g., 2440 mm × 3.2 mm stainless steel standard wall packed with 5% OV-101 on Chromosorb W, HP grade, 80/100 mesh. Inlet packing should be Chromosorb W, HP grade, 80/100 mesh. The maximum operating temperature for OV-101 is 350 °C. Use of other phase materials with maximum operating temperatures closer to the 290 °C procedure temperature may result in column bleed and a shift in the baseline. A baseline shift will affect integration of the higher cyclic and linear forms.

Note: OV is the registered trademark of Ohio Valley Specialty, the company that produces a line of silicone columns under that OV prefix. Also signified as Chromosorb WHP or W-HP.

3.2.3 Computer for GC data acquisition and analysis software.

3.2.4 Wrist action mechanical shaker. Van Waters and Rogers (VWR) Catalog number 57040-027 or equivalent.

3.2.5 Vials, Borosilicate Glass, 30 mL. VWR Catalog number 66011-165 or equivalent. Screw cap, size 22 400 with polyethylene cone liner.

3.2.6 Syringe, capable of delivering 25 µl ± 0.1 µl with no plastic elements due to solvents used.

3.2.7 Volumetric Flask. All flasks are graduated to Class A volumetric tolerances. Flasks with 5 mL to 250 mL capacity are calibrated to meet ASTM E288. VWR Catalog number 89025-840 (50 mL) -842 (100 mL) and -846 (250 mL) sizes or equivalent.

3.2.8 Volumetric Pipet, 10 mL. Class A tolerance, ± 0.02 mL. VWR Catalog number 89045-074 or equivalent.

3.2.9 Capped serum bottle, 20 mL. For standard solution storage to prevent pentane evaporation. Fisher Scientific Catalog number 06-406E or equivalent.

3.2.10 Analytical balance with draft shield capable of 0.1 mg accuracy.

3.2.11 Equipment for RTV sealant sample preparation.

3.2.11.1 Rigid plates (glass or aluminum), 0.90 mm thick for curing the wet formed-in-place sealant (Figure 1).

3.2.11.2 Tetrafluoroethylene fluorocarbon resin slab or suitable nonstick surface (Figure 1).

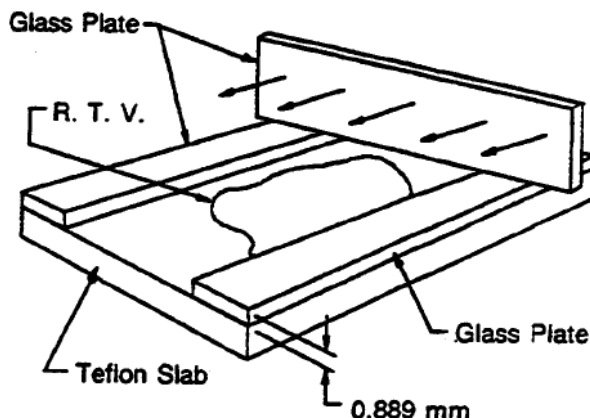


Figure 1: Wet Sealant Slab Preparation

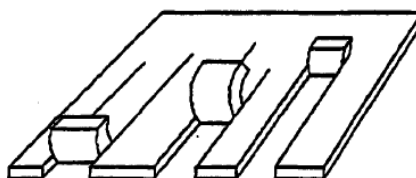
3.2.12 Test Fluids.**3.2.12.1 Pentane.** Reagent grade.**3.2.12.2 Dodecane,** 99% spectral grade.**3.2.12.3 Silicone Standards.** Cyclic monomers D₃ through D₆ and D₉ are available from Ohio Valley Specialty Chemical, 115 Industrial Road, Marietta, Ohio, U.S.A., 45750, or Petrarch Systems Inc., 2731 Bartram Road, Bristol, PA U.S.A., 19007. D₇, D₈ and D₁₀ are not commercially available.**3.3 Test Vehicle/Test Piece.** All specimens at the time of test are actual production process or intent. Because the specimen and test locations may be independent and miles apart, it is essential that samples are tested within their allotted time frame. The time frame for sealant cure is a minimum of 24 h and a maximum of 26 h. The time frame for molded part cure is a minimum of 72 h and a maximum of 74 h (for molded parts). Ship RTV sealant from the manufacturing sampling location in cartridges cap sealed at both ends (over the plunger and nozzle) so samples can be made in the test laboratory. For molded parts, express ship them directly to the test technician in sealed plastic bags. Test samples must come from the same mold and batch run. Included with each molded part are the press conditions, cure temperature (°C) and time, (minutes, seconds), mold date, time, part number, compound supplier and number, molding machine identification.**3.3.1 Sealants.** Tape two (2) 0.9 mm ± 0.2 mm thick rigid plates to the non-stick slab so they are approximately parallel and about 50 mm apart (Figure 1). Place a quantity of RTV sealant on the non-stick slab between the two (2) plates and near one (1) end. Draw down a third plate across the deposited sealant, spreading it to an even thickness (0.9 mm ± 0.2 mm) between the taped plates filling in most of the space. Avoid entrapped air and knit lines in the skived wet sealant. Allow the sample to cure for a minimum of 24 h, maximum of 26 h at 23 °C ± 3 °C and 50% ± 5% relative humidity.**3.3.1.1** Peel away the cured sealant and cut three (3) strips. (Figure 2). Take a sample from each strip and combine them to reach the desired sample weight of 1 g. Three (3) to ten pieces totaling 1 g. The smaller the cut size, the more surface area for solvent to extract.

Figure 2: Location for Sample Cut from Cured Slabs