Designation: F2524 – 06 (Reapproved 2013)

Standard Practice for Determination of Volatile Content for Formed-in-Place Gaskets (FIPG) Silicone Adhesives and Sealants for Transportation Applications[1]

This standard is issued under the fixed designation F2524; 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 the quantitative determination of the volatile matter evolved during the curing process of silicone adhesives and sealants for transportation applications.

1.2 The values stated in SI units are to be regarded as the standard. The values 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]

D4230 Test Method of Measuring Humidity with Cooled-Surface Condensation (Dew-Point) Hygrometer

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Terminology

3.1 *Definitions:*

3.1.1 *FIPG by-products, n*—chemicals that are released during the curing process.

3.1.2 *formed-in-place gaskets (FIPG), n*— one- or two-component adhesive or sealant applied wet, uncured, to a joint surface where the mating parts are assembled before the curing process is complete.

3.1.2.1 *Discussion*—When fully cured, it forms a barrier to media migration across the joint.

3.1.3 *multicomponent FIPG, n*—FIPG that is packaged in two or more parts, which are combined before application, and upon combination, a coreactant from one part of the adhesive chemically reacts, at ambient conditions, with a coreactant from another part of the FIPG.

3.1.4 *radiation-cured FIPG, n*—FIPG that contains unreacted monomers or oligomers that are polymerized by exposure to radiation such as ultraviolet (UV) or microwave.

3.1.4.1 *Discussion*—Cure conditions and equipment must be specified by FIPG manufacturer and shall be used in place of the humidity cure activation. Conditions and equipment shall be detailed in the final report.

3.1.5 *volatile content, n*—Low molecular weight chemicals, left unbound by the cured sealant system, which are released into the ambient atmosphere.

4. Summary of Practice

4.1 This practice is used to determine the volatile content of silicone adhesives and sealants upon curing for transportation applications.

5. Significance and Use

5.1 The quantity of volatile components in FIPG silicone adhesive and sealant by-products can be established by this test method. This test method does not identify the components.

6. Apparatus

6.1 *Humidity Chamber with Temperature Controller*—A forced ventilation oven conforming to the requirements for Type IIA in Specification E145 with humidity control capability. The oven should be capable of maintaining a temperature of 40 ± 1°C.

6.1.1 The oven shall be equipped with a National Institute of Standards and Technology (NIST) traceable calibrated thermometer or thermocouple.

6.1.2 The oven temperature shall be controlled by an accurate, reliable thermoregulator, maintaining the set point within ±1.0°C or better.

6.1.3 The inside of the oven shall be free of contamination or surface deposits. Stainless steel oven liner should be used to reduce corrosion caused by continued exposure to decomposition gases.

[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.

6.2 *Sample Container*—A weighing vessel of low shape design in glass, aluminum, or stainless steel of sufficient size to hold a 10-g sample. An aluminum weighing dish, 57 mm in diameter and 18 mm in depth, is suitable.

6.3 *Balance*—A calibrate weighing balance with a precision to within ±0.001 g.

7. Preparation of Apparatus

7.1 Maintain the humidity chamber at the temperature and humidity of the test for at least 1 h before insertion of the specimens.

7.2 Before test operation, verify the uniformity of temperature within the oven according to Specification E145. The airflow in the oven should be > 0.3 m^3/min (10 ft^3/min).

7.3 Before test operation, verify the relative humidity of the environmental chamber using a measurement device in accordance with Test Method D4230.

8. Procedure

8.1 Dry the weighing vessel in the oven at 110 ± 1°C for 30 min and cool to room temperature in a desiccator. Tare the weighing vessel to the nearest 0.001 g.

8.2 Spread approximately 10 g of FIPG sample evenly over the bottom of the weighing vessel and weigh to the nearest 0.001 g. Record the initial sample weight, *M*.

8.3 Place the sample-filled weighing dish in the humidity chamber controlled at 40 ± 1°C and 90 ± 3 % relative humidity.

8.4 After 24 h, remove the weighing vessels, cool to room temperature in a desiccator, and weigh to the nearest 0.001 g. Return the weighing dish to the humidity chamber for an additional 4 h, cool to room temperature in the desiccator, and reweigh. Constant sample weight is reached when the successive weighing differs by 0.001 g or less; if the weight variation is greater than 0.001 g, continue the 4-h heating-weighing cycle until constant weight is obtained. Record this weight as *M'*.

8.5 Test a minimum of three samples and average the results.

9. Calculation

9.1 For each determination, calculate the weight percent of volatile matter by-products from FIPG as follows:

$$\% \text{ Volatile} = [(M - M')/M] \times 100 \quad (1)$$

where:

M = initial sample weight as recorded in 8.2, and
M' = final sample weight as recorded in 8.4.

10. Report

10.1 Report the following information:

10.1.1 Percent volatile to the nearest 0.01 weight percent.

11. Keywords

11.1 by-products; curing process; FIPG; humidity chamber; silicone adhesives and sealants; volatile matter