Designation: D 4559 – 99ε1

Standard Test Method for Volatile Matter in Silicone Fluid[1]

This standard is issued under the fixed designation D 4559; 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.

ε[1] NOTE—Reference to a research report was added in February 2004.

1. Scope

1.1 This test method describes a procedure for determining the volatile matter in silicone fluids used for electrical insulation.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 923 Test Method for Sampling Electrical Insulating Liquids

D 2225 Test Methods for Silicone Fluids Used for Electrical Insulation

D 4652 Specification for Silicone Fluid Used for Electrical Insulation

D 5423 Specification for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation

3. Summary of Test Method

3.1 Specimens are weighed before and after heating for a specific time in a forced air oven to determine weight loss.

4. Significance and Use

4.1 High values may indicate contamination of the silicone with other materials, inadequate removal of volatile components by the producer, or the presence of a depolymerization catalyst.

4.2 The outcome will be affected directly by the presence of any high vapor pressure material in the sample, such as solvents or low molecular weight silicones.

4.3 A high volatile content could also indicate the presence of a depolymerization catalyst in the fluid. The time and temperature specified in this test method are ideal for detecting the effect of such a material, as the depolymerization takes place at a highly accelerated rate and the low molecular weight components are rapidly evaporated. The result is a very significant weight loss during the test period. The exact amount depends on the type and amount of catalyst present. The conditions specified in the method should not cause measureable depolymerization of silicone if such a catalyst is not present.

5. Apparatus

5.1 *Air-circulating Oven*, capable of meeting the requirements of Specification D 5423.

5.2 *Griffin Pyrex Beakers*, 50-mL, with the following dimensions:

5.2.1 *Outside Diameter*—42 ± 0.6 mm.

5.2.2 Wall Thickness—0.14 to 0.165 mm.

5.3 *Analytical Balance*, capable of measuring to 1 mg.

6. Sampling

6.1 Obtain a sample of the silicone fluid to be tested using appropriate ASTM sampling apparatus in accordance with Test Methods D 923.

7. Procedure

7.1 Clean two 50-mL beakers by washing in toluene, followed by a rinse in acetone or other appropriate cleaning method.

7.2 Bake two 50-mL beakers at 150 ± 5°C for a minimum of 1 h, transfer to a desiccator using clean, dry gloves or crucible tongs and cool the beakers to ambient temperatures. Use clean, dry gloves or crucible tongs to handle beakers from this point forward. Weigh each beaker to the nearest mg (T).

7.3 Weigh a 2.0 ± 0.2 g sample to the nearest mg in each beaker (W_1).

[1] This test method is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.07 on Physical Test.

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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.

7.4 Heat at 150 ± 5°C in an air circulating oven for 24 ± 0.25 h, cool in a desiccator, and reweigh (W_2).

8. Calculation and Report

8.1 Calculate the volatile matter in silicone fluid as follows:

$$\text{Volatile Matter, \%} = \frac{W_1 - W_2}{W_1 - T} \times 100$$

where:
W_1 = weight of silicone fluid and beaker,
W_2 = weight of silicone fluid and beaker after heating, and
T = weight of beaker.

8.2 Calculate the volatile matter, as the simple average percentage of the two tests.

8.3 Report the simple average of the two tests, rounded to the nearest 0.1 % as the volatile matter, %.

9. Precision and Bias [3]

9.1 *Repeatability*—The difference between results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, exceed the value of 0.243 only one case in twenty.

9.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the value of 0.565 only one case in twenty.

9.3 *Bias*—An estimate of bias of -0.032 was obtained for this test method using the spiked values and the measured value of the base oil.

9.4 *Limit of Detectability*—The limit of detectability was not calculated due to insufficient data.

10. Keywords

10.1 insulating liquid; silicone; volaticity

[3] A research report is available from ASTM International. Request RR:D27–1010.