



Standard Test Methods for Silicone Fluids Used for Electrical Insulation¹

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1. Scope

1.1 These test methods cover the testing of silicone fluids for use in transformers, capacitors, and electronic assemblies as an insulating or cooling medium, or both. These methods are generally suitable for specification acceptance, factory control, referee testing, and research.

1.2 Although some of the test methods listed here apply primarily to petroleum-based fluids, they are, with minor revisions, equally applicable to silicone fluids.

1.3 Silicone fluids are used for electrical insulating purposes because of their stable properties at high and low temperatures and their relative environmental inertness.

1.4 A list of the properties and standards are as follows:

| Property Measured | Section | ASTM Test Method |
|----------------------------------|---------|----------------------|
| <i>Physical:</i> | | |
| Color | 6 | D 2129 |
| Flash point | 7 | D 92 |
| Fire point | 7 | D 92 |
| Polychlorinated biphenyl content | 8 | D 4059 |
| Pour point | 9 | D 97 |
| Refractive index | 10 | D 1807 |
| Specific gravity | 11 | D 1298, D1481, D4052 |
| Volatility | 12 | D 4559 |
| Viscosity | 13 | D 445, D2161 |
| <i>Chemical:</i> | | |
| Neutralization number | 14 | D 974 |
| Water content | 15 | D 1533 |
| <i>Electrical:</i> | | |
| Relative permittivity | 16 | D 924 ² |
| Dielectric breakdown voltage | 17 | D 877 ³ |
| Dissipation factor | 18 | D 924 ² |
| Specific resistance | 19 | D 1169 ² |
| Compatibility | 20 | D 5282 |

1.5 *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:

¹ These test methods are under the jurisdiction of ASTM Committee D-27 on Electrical Insulating Liquids and Gases and are the direct responsibility of Subcommittee D27.02 on Synthetics.

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² A modified cell cleaning procedure is given for Test Methods D 924 and D 1169.

³ A modified cell cleaning procedure is recommended for Test Method D 877.

D 92 Test Method for Flash and Fire Points by Cleveland Open Cup⁴

D 97 Test Method for Pour Point of Petroleum Products⁴

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)⁴

D 877 Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using Disk Electrodes⁵

D 923 Test Methods for Sampling Electrical Insulating Liquids⁵

D 924 Test Method for Dissipation Factor (or Power Factor) and Relative Permittivity (Dielectric Constant) of Electrical Insulating Liquids⁵

D 974 Test Method for Acid and Base Number by Color-Indicator Titration⁴

D 1169 Test Method for Specific Resistance (Resistivity) of Electrical Insulating Liquids⁵

D 1298 Practice for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method⁴

D 1481 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer⁴

D 1533 Test Methods for Water in Insulating Liquids (Karl Fischer Reaction Method)⁵

D 1807 Test Methods for Refractive Index and Specific Optical Dispersion of Electrical Insulating Liquids⁵

D 2129 Test Method for Color of Water White Electrical Insulating Liquids⁵

D 2161 Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity⁴

D 2864 Terminology Relating to Electrical Insulating Liquids and Gases⁵

D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter⁶

D 4059 Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography⁵

D 4559 Test Method for Volatile Matter in Silicone Fluid⁵

D 4652 Specification for Silicone Fluid Used for Electrical Insulation⁵

D 5282 Test Methods for Compatibility of Construction

⁴ *Annual Book of ASTM Standards*, Vol 05.01.

⁵ *Annual Book of ASTM Standards*, Vol 10.03.

⁶ *Annual Book of ASTM Standards*, Vol 05.02.

Material with Silicone Fluid Used for Electrical Insulation⁵

3. Terminology

3.1 Definitions:

3.1.1 *fire point*—the temperature at which oil first ignites and burns for at least 5 s when a small test flame is passed across the surface under specified conditions.

3.1.2 *flash point*—the temperature at which vapors above the oil surface first ignite when a small test flame is passed across the surface under specified conditions.

3.1.3 *refractive index*—the ratio of the velocity of light (of a specified wavelength) in air at 25°C to its velocity in the substance under test.

3.1.4 *specific gravity*—the ratio of weight of a given volume of material to the weight of an equal volume of water. In this method, both weights are corrected to weight in vacuum, and the material is at 25°C using hydrometers calibrated at 60/60°F.

3.1.5 *volatility*—the weight of liquid lost when a specified weight of liquid is held at a specified elevated temperature for a specific period of time.

3.1.6 *water content*—the amount of water (mg/kg) dissolved in the liquid.

3.1.7 For additional terms refer to Terminology D 2864.

4. Significance and Use

4.1 Tests covered in this standard may be used for quality control and design considerations.

4.2 Included in each test method is a brief statement describing its significance.

5. Sampling

5.1 Accurate sampling, whether of the complete contents or only parts thereof, is extremely important from the standpoint of elevation of the quality of the product sampled. Obviously, examination of a sample that because of careless sampling procedure or contamination in the sampling equipment is not directly representative, leads to erroneous conclusions concerning quality.

5.2 Sample the silicone fluid in accordance with Test Methods D 923.

PHYSICAL METHODS

6. Color

6.1 *Significance*—The chief significance of color as applied to silicone fluid lies in the fact that if the fluid is colored, some degree of contamination exists that may affect the physical, chemical, and electrical properties of the fluid.

6.2 *Procedure*—Determine the color in accordance with Test Method D 2129.

7. Flash and Fire Points

7.1 *Significance*—The flash and fire points of a silicone insulating fluid indicates the limit to which the material may be heated, under the specified test conditions, before the emitted vapors form a flammable mixture in air. Unusually low flash or fire points for a given product may indicate contamination.

7.2 *Procedure*—Determine the flash and fire points in accordance with Test Method D 92.

8. Polychlorinated Biphenyl Content

8.1 Scope:

8.1.1 *Test Method D 4059*—describes a quantitative technique for determining the concentration of polychlorinated biphenyls (PCB's) in electrical insulating liquids.

8.2 Definition:

8.2.1 *PCB concentration*—is normally expressed in units of parts per million (PPM) on a weight by weight basis. Standard chromatograms of Aroclors 1242, 1254, and 1260 are used to determine the concentration of PCB in the sample.

8.3 *Summary of Test Method*—Following dilution of the sample in a suitable solvent, the solution is treated to remove interfering substances. A small portion is then injected into a packed gas chromatographic column where the components are separated and their presence measured by an electron capture or halogen-specific electrolytic conductivity detection. The method is made quantitative by comparing the response of a sample to that of a known quantity of one or more standard Aroclors obtained under the same conditions.

8.4 *Significance and Use*—United States regulations require that electrical apparatus and electrical insulating fluids containing PCB be handled and disposed of through the use of specific procedures as determined by the PCB content of the fluid. The results of this test method can be useful in selecting appropriate handling and disposal procedures.

9. Pour Point

9.1 *Significance*—The pour point is important as an index of the lowest temperature to which the material may be cooled without seriously limiting the degree of circulation of the fluid.

9.2 *Procedure*—Determine the pour point in accordance with Test Method D 97.

10. Refractive Index

10.1 *Significance*—The refractive index is often useful for the detection of some types of contamination and for the identification of the molecular makeup of the various types of silicone insulating fluids.

10.2 *Procedure*—Determine the refractive index in accordance with Test Methods D 1807.

11. Specific Gravity

11.1 *Significance*—Silicone insulating fluids are usually sold on a weight basis. The values for the specific gravities must frequently be known to calculate the volume of fluid present at any given temperature.

11.2 *Procedure*—Determine the specific gravity in accordance with Test Methods D 1481 or D 4052 and Practice D 1298.

12. Volatility

12.1 *Significance*—High values may indicate contamination of the silicone with other organic materials, inadequate removal of volatile components, or contamination with a depolymerization catalyst.

12.2 *Procedure*—Determine volatility in accordance with Test Method D 4559.

13. Viscosity

13.1 *Significance*—The viscosity of a silicone fluid is

important during the process of impregnation.

13.1.1 At operating temperatures the viscosity of a silicone fluid is a principal factor affecting heat transfer by convection flow of the fluid.

13.2 *Procedure*—Determine the viscosity in accordance with Test Method D 445. The kinematic viscosity may be converted to absolute viscosity in accordance with Test Method D 2161.

CHEMICAL METHODS

14. Neutralization Number

14.1 *Significance*— In the inspection of unused silicone fluids, the neutralization number is of importance as a quality index of purity. Properly refined silicone fluids are free from mineral acids and alkalies.

14.1.1 Since final oxidation products of silicone fluids are not acidic, small changes in the neutralization number of used silicone fluids may indicate the solution of basic or acidic materials from the various solid materials in contact with the silicone or the deterioration of such soluble materials to form basic or acidic materials.

14.2 *Procedure*—Determine the neutralization number in accordance with Test Method D 974.

15. Water Content

15.1 *Significance*— Under high humidity conditions, polydimethylsiloxane fluids can absorb moisture up to about 250 ppm by weight at 25°C. High levels of water content will significantly lower the resistivity and dielectric breakdown voltage of the fluid.

15.2 *Procedure*—Determine water content in accordance with Test Method D 1533.

ELECTRICAL METHODS

16. Relative Permittivity (Dielectric Constant)

16.1 *Significance*— Silicone insulating fluids are used to insulate components of an electrical network from each other and from ground. For this use, it is generally desirable to have the capacitance as small as possible, consistent with acceptable chemical properties and design considerations.

16.2 *Procedure*—Determine the relative permittivity in accordance with Test Method D 924. An alternative method of cleaning the test cells is to use multiple rinses of isopropanol followed by a thorough rinsing with methylene chloride and dry with clean, dry, warm air.

17. Dielectric Breakdown Voltage

17.1 *Significance*— The importance of the dielectric breakdown voltage of a silicone liquid is as a measure of its ability to withstand electrical stress without failure. It may also indicate the presence of contaminating materials, such as water, conducting solid particles, dissolved contaminants, or the decomposition products resulting from an electric arc. A high dielectric breakdown voltage, however, is not a certain indication of the absence of all contaminants.

17.2 *Procedure*—Determine the dielectric breakdown voltage in accordance with Method D 877, with the following modification:

17.2.1 Fill the test cup by tilting it at a 45° angle. As the liquid approaches the tilted top edge of the cup, slowly rotate the cup to an upright position while continuing to pour sample into the cup. This will reduce the amount of air bubbles in the sample and prevent bubbles from being trapped under the electrodes.

17.2.2 Make one breakdown on each of the specified fillings of the test cup.

17.2.3 Clean the electrode surfaces after each breakdown by one of the following methods:

17.2.3.1 *Method A*—After each breakdown and before the cup is emptied, pass the electrode-spacing gage through the electrode gap twice. Then empty the cup. This will clean the electrodes of any semisolid breakdown products and they will flow out when the cup is emptied.

17.2.3.2 *Method B*—Empty the cup. Wipe the electrode surfaces with a lintless paper or cloth. This is best accomplished by folding the lintless paper over a clinical tongue depressor. Flush the test cup with clean silicone fluid and empty the cup.

18. Dissipation (Power) Factor

18.1 *Significance*— Dissipation (power) factor of a silicone fluid is an indication of the energy dissipated as heat in the fluid. It is useful as a means for quality control and as an indication of changes in the fluid resulting from deteriorating and contaminating influences.

18.2 *Procedures*—Determine the dissipation factor in accordance with Test Method D 924. An alternative method of cleaning the test cells is to use multiple rinses of isopropanol followed by a thorough rinsing with methylene chloride and dry with clean, dry, warm air.

19. Specific Resistance (Resistivity)

19.1 *Significance*— The specific resistance of a silicone insulating fluid is a measure of its electrical insulating capability in d-c apparatus. High resistivity reflects low content of free ions and ion-forming particles, and normally indicates a low concentration of conductive contaminants.

19.2 *Procedure*—With the exception of the procedure for cleaning the test cells, determine the specific resistance in accordance with Test Method D 1169. An alternative method of cleaning the test cells is to use multiple rinses of isopropanol followed by a thorough rinsing with methylene chloride and dry with clean, dry, warm air.

COMPATIBILITY TESTING

20. Compatibility

20.1 *Significance*— It is very important to know how other construction materials will affect silicone liquid or the converse.

20.1.1 Incompatibility of the silicone fluid with the materials of construction can affect the usable life and operation of major apparatus and equipment, such as transformers and capacitors.

20.1.2 Compatibility tests are usually made at high temperatures, and for specific time periods. They may consist of physical methods, chemical methods, electrical methods, or all types, depending upon the particular application.

20.2 *Procedure*—Determine compatibility of silicone fluids with materials of construction in accordance with Test Methods D 5282.

21. Report

21.1 The report shall consist of the separate reports detailed in the specific methods used.

22. Precision and Bias

22.1 Use the precision and bias statements for each referenced method.

23. Keywords

23.1 electrical insulating fluid; silicone fluid

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