Designation: D2225 − 20

Standard Test Methods for Silicone Liquids Used for Electrical Insulation[1]

This standard is issued under the fixed designation D2225; 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 These test methods cover the testing of silicone liquids for use in transformers, capacitors, and electronic assemblies as an insulating or cooling medium, or both. These methods are generally suitable for specification acceptance (Specification D4652), 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 liquids.

1.3 Silicone liquids 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
Physical:		
Color	6	D2129
Flash point	7	D92
Fire point	7	D92
Polychlorinated biphenyl content	8	D4059
Pour point	9	D97
Refractive index	10	D1807
Specific gravity	11	D1298, D1481, D4052
Volatility	12	D4559
Viscosity	13	D445
Chemical:		
Acid number	14	D974
Water content	15	D1533
Electrical:		
Relative permittivity	16	D924[2]
Dielectric breakdown voltage	17	D877[3]
Dielectric breakdown voltage	17	D1816
Dissipation factor	18	D924[2]
Specific resistance	19	D1169[2]
Compatibility	20	D5282

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *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:*[4]

D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester
D97 Test Method for Pour Point of Petroleum Products
D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
D877 Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using Disk Electrodes
D923 Practices for Sampling Electrical Insulating Liquids
D924 Test Method for Dissipation Factor (or Power Factor) and Relative Permittivity (Dielectric Constant) of Electrical Insulating Liquids
D974 Test Method for Acid and Base Number by Color-Indicator Titration
D1169 Test Method for Specific Resistance (Resistivity) of Electrical Insulating Liquids
D1298 Test Method for Density, Relative Density, or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
D1481 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer
D1533 Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration
D1807 Test Methods for Refractive Index and Specific Optical Dispersion of Electrical Insulating Liquids (Withdrawn 2014)[5]

[1] These test methods are under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gases and are the direct responsibility of Subcommittee D27.02 on Gases and Non-Mineral Oil Liquids.

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[2] A modified cell cleaning procedure is given for Test Methods D924 and D1169.

[3] A modified cell cleaning procedure is recommended for Test Method D877.

[4] 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.

[5] The last approved version of this historical standard is referenced on www.astm.org.

D1816 Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using VDE Electrodes
D2129 Test Method for Color of Clear Electrical Insulating Liquids (Platinum-Cobalt Scale)
D2864 Terminology Relating to Electrical Insulating Liquids and Gases
D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
D4059 Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography
D4559 Test Method for Volatile Matter in Silicone Fluid
D4652 Specification for Silicone Liquid Used for Electrical Insulation
D5282 Test Methods for Compatibility of Construction Material with Silicone Fluid Used for Electrical Insulation

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 percent 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 D2864.

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 evaluation 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 Practices D923.

PHYSICAL METHODS

6. Color

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

6.2 *Procedure*—Determine the color in accordance with Test Method D2129.

7. Flash and Fire Points

7.1 *Significance*—The flash point of a silicone insulating liquid 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. The fire point of a silicone insulating liquid indicates the limit to which the material may be heated, under specified conditions, in which ignition is sustained for at least 5 s. 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 D92.

8. Polychlorinated Biphenyl Content

8.1 *Scope:*

8.1.1 *Test Method D4059*—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 mg/kg 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 detector. 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*—National, state, and local regulations require that electrical apparatus and electrical insulating liquids containing PCB be handled and disposed of through the use of specific procedures as determined by the PCB content of the liquid. 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 liquid.

9.2 *Procedure*—Determine the pour point in accordance with Test Method D97.

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 liquids.

10.2 *Procedure*—Determine the refractive index in accordance with Test Methods D1807.

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 D1298, D1481 or D4052.

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 D4559.

13. Viscosity

13.1 *Significance*— The viscosity of a silicone liquid is important during the process of impregnation.

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

13.2 *Procedure*—Determine the kinematic viscosity in accordance with Test Method D445. The same test method can be used to convert the kinematic viscosity to dynamic viscosity.

CHEMICAL METHODS

14. Acid Number

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

14.1.1 Since final oxidation products of silicone liquids 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 acid number in accordance with Test Method D974.

15. Water Content

15.1 *Significance*—Under high humidity conditions, polydimethylsiloxane liquids can absorb water up to about 204 mg/kg by weight at 25°C (Note 1). High levels of water content will significantly lower the resistivity and dielectric breakdown voltage of the liquid.

NOTE 1—The solubility of water in silicone insulating liquid can be calculated at any temperature based on Eq 1:[6]

$$10^{[(-1187/K)+6.2906]} \quad (1)$$

where:

K = 273.1 ± °C

15.2 *Procedure*—Determine water content in accordance with Test Method D1533.

ELECTRICAL METHODS

16. Relative Permittivity (Dielectric Constant)

16.1 *Significance*—Silicone insulating liquids are used to insulate components of an electrical network from each other and from ground. For this use, a low value of relative permittivity, generally desirable in order 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 D924. 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 D877, 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. If the discharge energy into the sample is less than 20 mJ (milli joule) then semisolid breakdown products will not be produced and testing can continue uninterrupted without any cleaning of the electrodes.

[6] Griffin, P. J., Bruce, C. M., and Christie, J. D., "Comparison of Water Equilibrium in Silicone and Mineral Oil Transformers," Minutes of the Fifty-Fifth Annual International Conference of Doble Clients, 1988, Sec. 10-9.1.