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Standard Guide for Sampling, Test Methods, Specifications, and Guide Specifications for Electrical Insulating Oils of Petroleum Origin ¹

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This standard has been approved for use by agencies of the Department of Defense.

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1. Scope

1.1 This guide describes methods of testing and specifications for electrical insulating oils of petroleum origin intended for use in electrical cables, transformers, oil circuit breakers, and other electrical apparatus where the oils are used as insulating, or heat transfer media, or both.

1.2 The purpose of this guide is to outline the applicability of the available test methods. Where more than one is available for measuring a given property, their relative advantages are described, along with an indication of laboratory convenience, precision, (95 % confidence limits), and applicability to specific types of electrical insulating oils.

1.3 This guide is classified into the following categories: Sampling Practices, Physical Tests, Electrical Tests, Chemical Tests, Specifications, and Guide Specifications. Within each test category, the test methods are listed alphabetically by property measured. A list of standards follows:

Category	Section	ASTM Method
<u>Sampling:</u>	3	D 923, D2759, D3305, D3613
<u>Physical Tests:</u>		
Aniline Point	4	D 611
Coefficient of Thermal Expansion	5	D 1903
Color	6	D 1500
Examination: Visual Infrared	7	D 1524, D2144
Flash and Fire Point	8	D 92
Interfacial Tension	9	D 971, D2285
Pour Point of Petroleum Products	10	D 97
Refractive Index	11	D 1218, D1807
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Specific Heat	13	D 2766
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Turbidity	15	D 6181
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<u>Electrical Tests:</u>		
Dielectric Breakdown Voltage	46	D 877, D1816, D3300
<u>Dielectric Breakdown Voltage</u>	17	<u>D 877, D1816, D3300</u>
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<u>Dissipation Factor and Relative Permittivity (Dielectric Constant)</u>	<u>18</u>	<u>D 924</u>
Gas Evolution	48	D 2300
<u>Gassing Tendency</u>	19	<u>D 2300</u>
Resistivity	49	D 1169
<u>Resistivity</u>	<u>20</u>	<u>D 1169</u>

Category	Section	ASTM Method
Stability Under Electrical Discharge	21	D 6180
<i>Chemical Tests:</i>		
Acidity, Approximate	20	D-1534
Acidity, Approximate	22	D 1534
Carbon-Type Composition	24	D-2140
Carbon-Type Composition	23	D 2140
Compatibility with Construction Material	22	D-3456
Compatibility with Construction Material	24	D 3455
Copper Content	23	D-3636
Copper Content	25	D 3635
Furanic Compounds	26	D 5837
Gas Analysis	24	D-3612
Gas Analysis	27	D 3612
Gas Content	25	D-831, D1827, D2945
Gas Content	28	D 831, D1827, D2945
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Inorganic Chlorides and Sulfates	29	D 878
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Neutralization (Acid and Base) Numbers	30	D 664, D974
Oxidation Inhibitor Content	28	D-2668, D4768
Oxidation Inhibitor Content	31	D 2668, D4768
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Oxidation Stability	32	D 1934, D2112, D2440
Polychlorinated Biphenyl Content	30	D-4059
Polychlorinated Biphenyl Content	33	D 4059
Sediment and Soluble Sludge	34	D-1698
Sediment and Soluble Sludge	34	D 1698
Sulfur, Corrosive	32	D-1275
Sulfur, Corrosive	35	D 1275
Water Content	33	D-1533
Water Content	36	D 1533
<i>Specification:</i>		
Mineral Insulating Oil for Electrical Apparatus	34	D-3487 Guide:
Mineral Insulating Oil for Electrical Apparatus	37	D 3487 Guide:
High Firepoint Electrical Insulating Oils	35	D-5222
High Firepoint Electrical Insulating Oils	38	D 5222

1.4 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:

- D 88 Test Method for Saybolt Viscosity ²
- D 92 Test Method for Flash and Fire Points by Cleveland Open Cup ³
- D 97 Test Method for Pour Point of Petroleum Products ³
- D 287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method) ³
- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity) ³
- D 611 Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents ³
- D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration ³
- D 831 Test Method for Gas Content of Cable and Capacitor Oils ⁴
- D 877 Test Method for Dielectric Breakdown Voltage of Insulating Liquids Using Disk Electrodes ⁴
- D 878 Test Method for Inorganic Chlorides and Sulfates in Insulating Oils ⁴
- ~~D-923 Test Method 923 Practices for Sampling Electrical Insulating Liquids ⁴~~

² Annual Book of ASTM Standards, Vol 04.04.

³ Annual Book of ASTM Standards, Vol 05.01.

⁴ Annual Book of ASTM Standards, Vol 10.03.

- D 924 Test Method for Dissipation Factor (or Power Factor) and Relative Permittivity (Dielectric Constant) of Electrical Insulating Liquids⁴
- ~~D 941 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer³~~
- ~~D 971 Test Method for Interfacial Tension of Oil Against Water by the Ring Method⁴~~
- D 974 Test Method for Acid and Base Number by Color-Indicator Titration³
- D 1169 Test Method for Specific Resistance (Resistivity) of Electrical Insulating Liquids⁴
- D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer³
- D 1218 Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids³
- D 1250 Guide for Petroleum Measurement Tables³
- D 1275 Test Method for Corrosive Sulfur in Electrical Insulating Oils⁴
- D 1298 Practice for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method³
- D 1481 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer³
- D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)³
- D 1524 Test Method for Visual Examination of Used Electrical Insulating Oils of Petroleum Origin in the Field⁴
- D 1533 Test Methods for Water in Insulating Liquids—(by Coulometric Karl Fischer Reaction Method) Titration⁴
- D 1534 Test Method for Approximate Acidity in Electrical Insulating Liquids by Color-Indicator Titration⁴
- D 1698 Test Method for Sediments and Soluble Sludge in Service-Aged Insulating Oils⁴
- D 1807 Test Methods for Refractive Index and Specific Optical Dispersion of Electrical Insulating Liquids⁴
- D 1816 Test Method for Dielectric Breakdown Voltage of Insulating Oils of Petroleum Origin Using VDE Electrodes⁴
- D 1827 Test Method for Gas Content (Nonacidic) of Insulating Liquids by Displacement with Carbon Dioxide⁴
- D 1903 Test Method for Coefficient of Thermal Expansion of Electrical Insulating Liquids of Petroleum Origin and Askarels⁴
- D 1934 Test Method for Oxidative Aging of Electrical Insulating Petroleum Oils by Open-Beaker Method⁴
- D 2112 Test Method for Oxidation Stability of Inhibited Mineral Insulating Oil by ~~Rotating Bomb~~ Pressure Vessel⁴
- D 2140 Test Method for Carbon-Type Composition of Insulating Oils of Petroleum Origin⁴
- D 2144 Test Method for Examination of Electrical Insulating Oils by Infrared Absorption⁴
- D 2161 Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity³
- D 2285 Test Method for Interfacial Tension of Electrical Insulating Oils of Petroleum Origin Against Water by the Drop-Weight Method⁴
- D 2300 Test Method for Gassing of Insulating Oils Under Electrical Stress and Ionization (Modified Pirelli Method)⁴
- D 2440 Test Method for Oxidation Stability of Mineral Insulating Oil⁴
- D 2668 Test Method for 2,6-Ditertiary-Butyl Para-Cresol and 2,6-Ditertiary-Butyl Phenol in Electrical Insulating Oil by Infrared Absorption⁴
- D 2717 Test Method for Thermal Conductivity of Liquids⁵
- D 2759 Practice for Sampling Gas from a Transformer Under Positive Pressure⁴
- D 2766 Test Method for Specific Heat of Liquids and Solids⁵
- D 2864 Terminology Relating to Electrical Insulating Liquids and Gases⁴
- D 2945 Test Method for Gas Content of Insulating Oils⁴
- D 3300 Test Method for Dielectric Breakdown Voltage of Insulating Oils of Petroleum Origin Under Impulse Conditions⁴
- D 3305 Practice for Sampling—a Small Gas Volume in a Transformer⁴
- D 3455 Test Methods for Compatibility of Construction Materials with Electrical Insulating Oil of Petroleum Origin⁴
- D 3487 Specification for Mineral Insulating Oil Used in Electrical Apparatus⁴
- D 3612 Test Method for Analysis of Gases Dissolved in Electrical Insulating Oil by Gas Chromatography⁴
- ~~D 3613 Test Methods of 3613 Practice for Sampling Electrical Insulating Oils~~ Liquids for Gas Analysis and Determination of Water Content⁴
- D 3635 Test Method for Dissolved Copper in Electrical Insulating Oil by Atomic Absorption Spectrophotometry⁴
- D 4059 Test Method for Analysis of Polychlorinated Biphenyls in Insulating Liquids by Gas Chromatography⁴
- D 4768 Test Method for Analysis of 2,6-Ditertiary-Butyl Para-Cresol and 2,6-Ditertiary-Butyl Phenol in Insulating Liquids by Gas Chromatography⁴
- ~~D 5222 Guide 5222 Specification for High Fire-Point of Mineral~~ Electrical Insulating Oils⁴
- D 5837 Test Method for Furanic Compounds in Electrical Insulating Liquids by High-Performance Liquid Chromatography (HPLC)⁴
- D 6180 Test Method for Stability of Insulating Oils of Petroleum Origin Under Electrical Discharge⁴
- D 6181 Test Method for Measurement of Turbidity in Mineral Insulating Oil of Petroleum Origin⁴

⁵ Annual Book of ASTM Standards, Vol 05.02.

SAMPLING**3. Sampling**

3.1 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, careless sampling procedure or contamination in the sampling equipment will result in a sample that is not truly representative. This generally leads to erroneous conclusions concerning quality and incurs loss of the time, effort, and expense involved in securing, transporting, and testing the sample.

3.2 Sample the insulating oil in accordance with ~~Test Methods~~ Practices D 923, D 2759, D 3305, and D 3613, as appropriate.

PHYSICAL PROPERTIES**4. Aniline Point**

4.1 *Scope*—This test method covers the determination of the aniline point of petroleum products, provided that the aniline point is below the bubble point and above the solidification point of the aniline-sample mixture.

4.2 *Summary of Test Method:*

4.2.1 *Test Methods D 611*—Equal volumes of aniline and test specimen or aniline and test specimen plus *n*-heptane are placed in a tube and mixed mechanically. The mixture is heated at a controlled rate until the two phases become miscible. The mixture is then cooled at a controlled rate, and the temperature at which the two phases separate is recorded as the aniline point.

4.3 *Significance and Use*—The aniline point of an insulating oil indicates the solvency of the oil for some materials that are in contact with the oil. A higher aniline point implies a lower aromaticity and a lower degree of solvency for some materials.

5. Coefficient of Thermal Expansion

5.1 *Scope*—This test method covers the determination of the coefficient of thermal expansion of electrical insulating liquids of petroleum origin.

5.2 *Definition:*

5.2.1 *coefficient of thermal expansion*—the change in volume per unit volume per degree change in temperature. It is commonly stated as the average coefficient over a given temperature range.

5.3 *Summary of Test Method*—The specific gravity of insulating oils is determined at two temperatures below 90°C and separated by not less than 5°C nor more than 14°C. Test methods used may be D287, D 1217, D1298, or D1481. The calculation of average coefficient of thermal expansion over this temperature range is given in Test Method D 1903.

5.4 *Significance and Use*—A knowledge of the coefficient of expansion of a liquid is essential to compute the required size of a container to accommodate a volume of liquid over the full temperature range to which it will be subjected. It is also used to compute the volume of void space that would exist in an inelastic device filled with the liquid after the liquid has cooled to a lower temperature.

6. Color

6.1 *Scope*—This test method covers the visual determination of color of a wide variety of liquid petroleum products, including mineral insulating oils.

6.2 *Summary of Test Method:*

6.2.1 *Test Method D 1500*—The test specimen is placed in a glass sample jar (an ordinary 125-mL test specimen bottle is satisfactory for routine tests). The color of the sample by transmitted light is compared with a series of tinted glass standards. The glass standard matching the sample is selected, or if an exact match is not possible, the next darker glass is selected. The results are reported numerically on a scale of 0.5 to 8.0.

6.3 *Significance*—A low color number is an essential requirement for inspection of assembled apparatus in a tank. An increase in the color number during service is an indicator of deterioration or contamination of the insulating oil.

7. Examination

7.1 *Scope:*

7.1.1 Both visual examination and qualitative infrared absorption are described in this section. The test methods are:

7.1.2 *Test Method D 1524*—This is a visual examination of mineral insulating oils that have been used in transformers, oil circuit breakers, or other electrical apparatus as insulating or cooling media, or both. This test is intended for use in the field.

7.1.3 *Test Method D 2144*—The infrared absorption from 2.5 to 25 μm (4000 to 667 cm^{-1}) is recorded as a means of (a) establishing continuity by comparison with the spectra of previous shipments by the same supplier, (b) for the detection of some types of contaminants, (c) for the identification of oils in storage or service. This test method is not intended for the determination of the various constituents of an oil.

7.2 *Summary of Test Methods:*

7.2.1 *Test Method D 1524*—Estimate the color of the oil by use of an oil comparator, matching the oil test specimen with tinted glass color standards. Note the presence of cloudiness, particles of insulation, metal corrosion products, or other undesirable suspended materials in the oil.

7.2.2 *Test Methods D 2144*—The infrared spectrum is recorded from 2.5 to 25 μm (4000 to 667 cm^{-1}) either as the absorption spectrum itself, or as the differential between the test specimen and reference oil. The spectra are compared with reference spectra to establish the identity of the oil.

7.3 *Significance and Use:*

7.3.1 *Test Method D 1524*—The observation of the color and condition of the oil in a field inspection permits a determination of whether the sample should be sent to a central laboratory for full evaluation.

7.3.2 *Test Methods D 2144*—The infrared spectrum of an electrical insulating oil indicates the general chemical composition of the sample. Because of the complex mixture of compounds present in insulating oils, the spectrum is not sharply defined and may not be suitable for quantitative estimation of components. The identity of the oil can be quickly established as being the same or different from previous samples by comparison with the reference spectra.

8. Flash and Fire Point

8.1 *Scope:*

8.1.1 This test method covers the determination of flash and fire points of all petroleum products except fuel oils and those having an open cup flash below 79°C (175°F).

8.1.2 This test method should be used solely to measure and describe the properties of materials in response to heat and flame under controlled laboratory conditions and should not be used for the description, appraisal, or regulation of the fire hazard of materials under actual fire conditions.

8.2 *Definitions:*

8.2.1 *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.

8.2.2 *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.

8.3 *Summary of Test Method*—Fill the test cup to the specified level with the test specimen. Heat the sample initially at 14 to 17°C/min (25 to 30°F/min) until the temperature is 56°C (100°F) below the expected flash point. Reduce the rate of temperature change to 5 to 6°C/min (9 to 11°F/min) and apply the test flame every 2°C (or 5°F) until a flash occurs. Continue heating and testing every 2°C (or 5°F) until the oil continues to burn for at least 5 s. The procedure is described in Test Method D 92.

8.4 *Significance and Use*—The flash point and fire point tests give an indication of the flammability of an oil. They may also be used to provide a qualitative indication of contamination with more flammable materials. In the latter context, the flash point test is more sensitive.

9. Interfacial Tension

9.1 *Scope*—These test methods cover the measurement, under nonequilibrium conditions, of the interfacial tension of insulating oils against water. These test methods have been shown by experience to give a reliable indication of the presence of hydrophilic compounds.

9.2 *Definition:*

9.2.1 *interfacial tension*—the molecular attractive force between unlike molecules at an interface. It is usually expressed in dynes per centimetre or millinewtons per metre.

9.3 *Summary of Test Methods:*

9.3.1 *Test Method D 971*—Interfacial tension is determined by measuring the force necessary to detach a platinum wire upward from the oil-water interface. To calculate the interfacial tension, the force so measured is corrected by an empirically determined factor which depends upon the force applied, the densities of both oil and water, and the dimensions of the ring. The measurement is completed within 1 min of the formation of the interface.

9.3.2 *Test Method D 2285*—Interfacial tension is determined by measuring the volume of a drop of water that the oil will support. The larger the drop of water, the higher the interfacial tension of the oil. The instrument used to measure the volume of the drops of water is calibrated to read approximately in dynes per centimetre interfacial tension. For better accuracy, the reading can be corrected by a factor that depends on the density of the oil. The drop is allowed to age for 30 s and to fall between 45-s and 60 s after formation.

9.4 *Significance and Use*—Interfacial tension measurements on electrical insulating oils provide a sensitive means of detecting small amounts of soluble polar contaminants and products of oxidation. A high value for new mineral insulating oil indicates the absence of undesirable polar contaminants. The test is frequently applied to service-aged oils as an indication of the degree of deterioration.

10. Pour Point

10.1 *Scope*—The pour point is applicable to any petroleum oil.

10.2 *Definition:*

10.2.1 *pour point*—the lowest temperature, expressed as a multiple of 3°C at which the oil is observed to flow when cooled and examined under prescribed conditions.

10.3 *Summary of Test Method*—After preliminary heating, the test specimen is cooled at a specified rate and examined at

intervals of 3°C for flow characteristics. The lowest temperature at which movement of the oil is observed within 5 s is reported as the pour point. The procedure is described in Test Method D 97.

10.4 Significance and Use:

10.4.1 The pour point of an insulating oil gives an indication of the temperature below which it may not be possible to pour or remove the oil from its container.

10.4.2 In connection with oil for use in cable systems, the pour point may be useful to indicate the point at which no free movement will take place in the cable or to indicate the temperature at which partial separation of wax may occur.

10.4.3 The pour point of a transformer oil 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 oil. Some materials are sensitive to temperature cycling or prolonged storage at low temperatures, and their pour points may not adequately predict their low temperature flow properties.

11. Refractive Index and Specific Optical Dispersion

11.1 Scope:

11.1.1 *Test Method D 1218*—Describes a precision method for determining refractive index accurate to 0.00006 and refractive dispersion accurate to 0.00012. The liquid must be transparent, no darker than ASTM 4.0 color (see Test Method D 1500) and have a refractive index between 1.33 and 1.50. The specific optical dispersion is calculated by dividing the refractive dispersion value by the specific gravity of the liquid.

11.1.2 *Test Method D 1807*—Describes a routine method for measuring refractive index accurate to three units in the fourth decimal place, measuring refractive dispersion, and calculating specific optical dispersion accurate to three units in the fourth decimal place. The oils must be transparent and light colored.

11.2 Definitions:

11.2.1 *refractive index*—the ratio of the velocity of light in air to its velocity in the substance under test.

11.2.2 *specific optical dispersion*—the difference between the refractive indexes of light of two different wave lengths, both indexes measured at the same temperature, the difference being divided by the specific gravity also measured at the test temperature. For convenience, the specific dispersion value is multiplied by 10^4 .

11.3 Summary of Test Method:

11.3.1 The two methods differ in the accuracy of the refractometer used. After adjusting the instrument temperature to 25°C, apply the test specimen to the refracting prism, read the refractive index, and read the compensator dial reading. From the correlation tables supplied with the instrument obtain the refractive dispersion. Calculate the specific optical dispersion by dividing refractive dispersion by the specific gravity of the oil.

11.4 Significance and Use:

11.4.1 *Refractive Index* of an insulating liquid varies with its composition and with the nature and amount of contaminants held in solution. Where the refractive index of an insulating liquid when new is known, determinations made on the same liquid after periods of service may form a basis for estimating any change in composition or the degree of contamination acquired through solution.

11.4.2 *Specific Optical Dispersion* serves as a quick index to the amount of unsaturated compounds present in an oil. As the dispersion values for paraffinic and naphthenic compounds are nearly the same and are essentially independent of molecular weight and structural differences, values above a minimum of about 97 bear a direct relationship to the amount of aromatic compounds present in insulating oil.

12. Relative Density (Specific Gravity)

12.1 Scope:

12.1.1 The methods used to measure relative density (specific gravity) may use a hydrometer, pycnometer, or ~~a pycnometer~~ an oscillating tube.

12.1.1.1 *Test Method D 287*—Uses an API hydrometer and is limited to liquids having a Reid vapor pressure of 180 kPa (26 psi) or less.

12.1.1.2 *Test Method D 1217*—Covers the use of a pycnometer to measure the relative density (specific gravity) of petroleum fractions.

12.1.1.3 *Test Method D 1298*—Covers the use of a hydrometer to measure relative density (specific gravity) directly or the measurement of API gravity followed by conversion to relative density (specific gravity). This test method is limited to liquids having a Reid vapor pressure of 179 kPa (26 psi) or less. This test method is most suitable for use with mobile transparent liquids, although it can also be used with viscous oils if sufficient care is taken in the measurement.

12.1.1.3.4 *Test Method D 1481*—Covers the determination of the densities of oils more viscous than 15 cSt at 20°C. The liquid should not have a vapor pressure greater than 13 kPa (100 mm Hg) at the test temperature. To measure the density of less viscous liquids more accurately than permitted by the hydrometer method, ~~Test Methods D 941 and Method D 1217~~ are is available.

12.1.1.5 *Test Method D 4052*—Covers the measurement of relative density (specific gravity) by the measurement of change in oscillation frequency of a vibrating glass tube filled with test liquid.

12.2 Definition:

12.2.1 *relative density (specific gravity)*—the ratio of the mass (weighed in vacuum) of a given volume of liquid at 15.6°C

(60°F) to the mass of an equal volume of pure water at the same temperature. When reporting results, explicitly state the reference temperature, for example, specific gravity 15.6/15.6°C.

12.3 Summary of Test Method:

12.3.1 API gravity may be measured at the oil temperature using a hydrometer (Test Methods D 287 or D 1298) and converting to 15.6°C using Guide D 1250.

12.3.2 Relative density (specific gravity) may be measured at the oil temperature using a hydrometer (Test Method D 1298) and converted to 15.6°C using Guide D 1250.

12.3.3 *Test Method D 1481*—The liquid is drawn into the bicapillary pycnometer through the removable siphon arm and adjusted to volume at the temperature of test. After equilibration at the test temperature, liquid levels are read; and the pycnometer is removed from the thermostated bath, cooled to room temperature, and weighed. Density or relative density (specific gravity), as desired, is then calculated from the volume at the test temperature, and the weight of the sample. The effect of air buoyancy is included in the calculation.

12.4 Significance and Use:

12.4.1 Electrical insulating oils are usually sold on the basis of volume delivered at 15.6°C (60°F). Delivery is often made on the basis of net weight of product in drums, and the specific gravities often are measured at temperatures other than 15.6°C. The values of relative density (specific gravity) at 15.6°C must be known to calculate the volume at 15.6°C of the oil delivered.

12.4.2 The relative density (specific gravity) of a mineral insulating oil influences the heat transfer rates and may be pertinent in determining suitability for use in specific applications. In certain cold climates, ice may form in de-energized transformers exposed to temperatures below 0°C, and the maximum specific gravity of the oil used in such equipment should be at a value that will ensure that ice will not float in the oil at any temperature the oil might attain.

12.4.3 When making additions of insulating liquid to apparatus in service, a difference in relative density (specific gravity) may indicate a tendency of the two bodies of liquid to remain in separate layers rather than mixing into a homogeneous single body of liquid. Such conditions have caused serious overheating of self-cooled apparatus. Suitable precautions should be taken to ensure mixing.

13. Specific Heat

13.1 *Scope*—This test method covers determination of the specific heat of electrical insulating liquids of petroleum origin.

13.2 Definition:

13.2.1 *specific heat (or heat capacity) of a substance*—a thermodynamic property that is a measure of the amount of energy required to produce a given temperature change within a unit quantity of that substance. The standard unit of heat capacity is calories/gram°C Joules/Kg°C at some defined temperature; specific heat is dimensionless as it is the ratio of the substance's heat capacity relative to that of water.

13.3 *Summary of Test Method*—The specific heat is determined by Test Method D 2766. The measurement is made by heating a test specimen at a known and fixed rate. Once dynamic heating equilibrium is obtained, the heat flow is recorded as a function of temperature. The heat flow normalized to specimen mass and heating rate is directly proportional to the specimen's specific heat capacity.

13.4 *Significance and Use*—A knowledge of the specific heat is helpful in designing adequate heat transfer properties for electrical apparatus. A higher specific heat value indicates a more efficient heat transfer medium.

14. Thermal Conductivity

14.1 *Scope*—This test method covers the determination of the thermal conductivity of electrical insulating liquids of petroleum origin.

14.2 Definition:

14.2.1 *thermal conductivity*—the ability of a substance to transfer energy as heat in the absence of mass transport phenomena. The standard unit of thermal conductivity is as follows:

$$W/(mK) \text{ (Cal/cm s } ^\circ\text{C)}$$

14.3 *Summary of Test Method*—The thermal conductivity is determined by Test Method D 2717. This test method measures the temperature gradient produced across the liquid by a known amount of energy introduced into the test cell by an electrically heated platinum element.

14.4 *Significance and Use*—A knowledge of thermal conductivity is helpful in designing adequate heat transfer properties for electrical apparatus. A high value indicates a good heat transfer efficiency property for the liquid.

15. Viscosity-Turbidity

15.1 *Scope*—This test method determines the amount of suspended particulate matter in electrical insulating oil of petroleum origin.

15.2 Definition:

15.2.1 *turbidity, n*—the reduction of transparency due to presence of particulate matter. The standard unit of turbidity is the nephelometric turbidity unit (NTU), which is defined as the intensity of light scattered by a known aqueous suspension of formazine.

15.3 Summary of Test Method—The turbidity is determined by Test Method D 6181. This test method measures the scattered light at 0.5π rad (90°) or 0.5 and 1.5π rad (90° and 270°) angles to the incident beam using a nephelometer that has been calibrated with a standard aqueous suspension of formazine.

15.4 Significance and Use—Turbidity measures particulate contamination in electrical insulating oil that may not be apparent to the unaided human eye and could affect the performance of the dielectric fluid.

16. Viscosity

16.1 Scope:

16.1.1 Test Method D 88—Covers the empirical measurement of Saybolt viscosity of petroleum products using the Saybolt viscometer at temperatures between 24 ± 3.1 and 98.9°C (70 and 210°F).

156.1.2 Test Method D 445—Covers the determination of the kinematic viscosity of liquid petroleum products by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer.

156.1.3 Practice D 2161—Provides tables or equations for the conversion of centistokes into Saybolt Universal Seconds or Saybolt Furol Seconds at the same temperatures.

156.2 Summary of Test Methods:

156.2.1 Test Method D 88—The efflux time in seconds for 60 mL of test specimen to flow through a calibrated orifice in the Saybolt viscometer is measured under carefully controlled conditions, particularly temperature and liquid head. The time is converted by an orifice factor and reported as the viscosity of the sample at that temperature.

156.2.2 Test Method D 445—The time is measured in seconds for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

156.2.3 Practice D 2161—The Saybolt Universal viscosity equivalent to a given kinematic viscosity varies with the temperature at which the determination is made. The basic conversion values are given in Table 1 of this practice for 37.8°C (100°F). Factors are given for converting units at other temperatures. The Saybolt Furol viscosity equivalents are given in Table 3 of this practice for 50.0 and 98.9°C (122 and 210°F) only.

156.3 Significance and Use:

156.3.1 The fundamental and preferred method for measuring kinematic viscosity is by use of Test Method D 445. The Saybolt instrument in Test Method D 88, being of all-metal construction, may be more rugged for field use, but values obtained are significantly less accurate than those obtained by the use of the capillary viscometers in Test Method D 445.

156.3.2 Viscosity of electrical insulating oils influences their heat transfer properties, and consequently the temperature rise of energized electrical apparatus containing the liquid. At low temperatures, the resulting higher viscosity influences the speed of moving parts, such as those in power circuit breakers, switchgear, load tapchanger mechanisms, pumps, and regulators. Viscosity controls insulating oil processing conditions, such as dehydration, degassification and filtration, and oil impregnation rates. High viscosity may adversely affect the starting up of apparatus in cold climates (for example, spare transformers and replacements). Viscosity affects pressure drop, oil flow, and cooling rates in circulating oil systems, such as in pipe-type cables and transformers.

ELECTRICAL PROPERTIES

167. Dielectric Breakdown Voltage

167.1 Scope:

167.1.1 There are two standard test methods for determining the dielectric breakdown voltage of electrical insulating fluids at commercial power frequencies, D 877 and D 1816, and one standard test method for determining the dielectric breakdown voltage of insulating oils under impulse conditions, D 3300.

167.1.2 Test Method D 877—~~Is a~~Applicable to liquid petroleum oils, hydrocarbons, and askarels commonly used as insulating and cooling media in cables, transformers, oil circuit breakers, and similar apparatus. The suitability of Test Method D 877 for testing liquids having viscosities exceeding 900 cSt (5000 SUS) at 40°C (104°F) has not been determined.

167.1.3 Test Method D 1816—~~Is a~~Applicable to liquid petroleum oils commonly used as an insulating and cooling medium in cables, transformers, oil circuit breakers, and similar apparatus. The suitability of Test Method D 1816 for testing oils having viscosities of more than 19 cSt (100 SUS) at 40°C (104°F) has not been determined.

167.1.4 Test Method D 3300—~~Is a~~Applicable to any liquid commonly used as an insulating and cooling medium in high-voltage apparatus subjected to impulse conditions, such as transient voltage stresses arising from such causes as nearby lightning strikes and high-voltage switching operations.

167.2 Definition:

167.2.1 dielectric breakdown voltage—the potential difference at which electrical failure occurs in an electrical insulating material or insulation structure, under prescribed test conditions.

167.3 Summary of Test Methods:

167.3.1 Test Method D 877—The insulating liquid is tested in a test cup between two 25.4-mm (1-in.) diameter disk electrodes spaced 2.54 mm (0.100 in.) apart. A 60-Hz voltage is applied between the electrodes and raised from zero at a uniform rate of 3 kV/s. The dielectric breakdown voltage at which is recorded, prior to the current produced by breakdown occurrence of disruptive

discharge, when the liquid reaches voltage across the range of 2 specimen has dropped to 20 mA, tripping a circuit breaker, is considered to be the dielectric breakdown voltage, less than 100 V. In the referee procedure, one breakdown test is made on each of five fillings of the test cup, and the average and individual values of breakdown voltage are reported.

167.3.2 *Test Method D 1816*—The oil is tested in a test cell between spherically capped (VDE) electrodes spaced either 1 mm (0.040 in.) or 2 mm (0.080 in.) apart. The oil is stirred before and during application of voltage by means of a motor-driven stirrer. A 60-Hz voltage is applied between the electrodes and raised from zero at a uniform rate of ½ kV/s. The voltage at which the current produced by breakdown of the oil reaches the range of 2 to 20 mA, tripping a circuit breaker, is considered to be the dielectric breakdown voltage. In the procedure, five breakdown tests are made on one filling of the test cell. If the five breakdowns fall within the statistical requirements, the average value is reported. If not, five additional breakdowns are required with the average of the ten values reported.

167.3.3 *Test Method D 3300*—The electrode system consists of either: (1) two 12.7-mm (0.5-in.) diameter spheres spaced 3.8 mm (0.15 in.) apart or (2) a 12.7-mm (0.5-in.) diameter sphere and a steel phonograph needle of 0.06-mm radius of curvature of point, spaced 25.4 mm (1.0 in.) apart. The polarity of the needle with respect to the sphere can be either positive or negative. The electrodes are immersed in the oil in a test cell. An impulse wave of 1.2 by 50 µs wave shape (times to reach crest value and to decay to half of crest value, respectively) is applied at progressively higher voltages until breakdown occurs.

167.4 *Significance and Use:*

167.4.1 *Power Frequencies (Test Methods D 877 and D 1816)*—The dielectric breakdown voltage of an insulating liquid at commercial power frequencies is of importance as a measure of the liquid's ability to withstand electric stress. It is the voltage at which breakdown occurs between two electrodes under prescribed test conditions. It also serves to indicate the presence of contaminating agents, such as water, dirt, moist cellulosic fibers, or conducting particles in the liquid, one or more of which may be present when low dielectric breakdown values are found by test. However, a high dielectric breakdown voltage does not indicate the absence of all contaminants. See Appendix X1 of either test method for other influences that affect the dielectric breakdown voltage of a liquid.

167.4.1.1 The ability of a fluid to resist breakdown under the test conditions is an indication of the ability of the fluid to perform its insulating function in electrical apparatus. The average breakdown voltage is commonly used in specifications for the qualification and acceptance of insulating fluids. It is also used as a control test for the refining of new or reclaiming of used insulating fluids. Because of the complex interactions of the factors affecting dielectric breakdown voltage the values obtained cannot be used for design purposes.

167.4.1.2 The square-edged disk electrodes of Test Method D 877 are relatively insensitive to dissolved water in concentrations below 60 % of the saturation level. This method is recommended for acceptance tests on unprocessed insulating liquids received from vendors in tank cars, tank trucks, and drums. It may also be used for the routine testing of liquids from selected power systems apparatus.

167.4.1.3 The more uniform electric field associated with VDE electrodes employed in Test Method D 1816 is more sensitive to the deleterious effects of moisture in solution, especially when cellulosic fibers are present in the oil, than is the field in Test Method D 877. Test Method D 1816 is recommended can be used for testing filtered, degassed, and dehydrated oil prior to, during, and after the filling of power systems apparatus. It is also finding increased usage for testing oils from transformers in service: processed or as received oils. Filtering and dehydrating the oil may increase Test Method D 1816 dielectric breakdown voltages substantially.

167.4.2 *Impulse Conditions (Test Method D 3300):*

167.4.2.1 This test method is most commonly performed using a negative polarity point opposing a grounded sphere (NPS). The NPS breakdown voltage of fresh unused oils measured in the highly divergent field in this configuration depends on oil composition; decreasing with increasing concentration of aromatic, particularly polyaromatic, hydrocarbon molecules.

167.4.2.2 This test method may be used to evaluate the continuity of composition of an oil from shipment to shipment. The NPS impulse breakdown voltage of an oil can also be substantially lowered by contact with materials of construction, by service aging, and by other impurities. Test results lower than those expected for a given fresh oil may also indicate use or contamination of that oil.

167.4.2.3 Although polarity of the voltage wave has little or no effect on the breakdown strength of an oil in uniform fields, polarity does have a marked effect on the breakdown voltage of an oil in nonuniform electric fields.

178. Dissipation Factor and Relative Permittivity (Dielectric Constant)

178.1 *Scope:*

178.1.1 This test method covers new electrical insulating liquids as well as liquids in service or subsequent to service in cables, transformers, oil circuit breakers, and other electrical apparatus.

178.1.2 This test method provides a procedure for making referee and routine tests at a commercial frequency of approximately 60 Hz.

178.2 *Summary of Test Method:*

178.2.1 The loss characteristic is commonly measured in terms of dissipation factor (tangent of the loss angle) or of power factor (sine of the loss angle). For values up to 0.05, dissipation factor and power factor values are equal to each other within about one part in one thousand and the two terms may be considered interchangeable.

178.2.2 Test Method D 924—The oil test specimens are tested in a three-terminal or guarded electrode test cell maintained at the desired test temperature. Using a bridge circuit, measure the loss characteristics and capacitance following the instructions appropriate to the bridge being used. For routine tests, a two-electrode cell may be used.

178.3 Significance and Use:

178.3.1 Dissipation Factor (or Power Factor)—This property is a measure of the dielectric losses in an oil, and hence, of the amount of energy dissipated as heat. A low value of dissipation factor (or power factor) indicates low dielectric losses and a low level of soluble polar ionic or colloidal contaminants. This characteristic may be useful as a means of quality control and as an indication of oil changes in service resulting from contamination and oil deterioration.

178.3.2 Relative Permittivity (Dielectric Constant)—Insulating liquids are used in general either to insulate components of an electrical network from each other and from ground, alone or in combination with solid insulating materials, or to function as the dielectric of a capacitor. For the first use, a low value of relative permittivity is often desirable in order to have the capacitance be as small as possible, consistent with acceptable chemical and heat transfer properties. However, an intermediate value of relative permittivity may sometimes be advantageous in achieving a better voltage distribution between the liquid and solid insulating materials with which the liquid may be in series. When used as the dielectric in a capacitor, it is desirable to have a higher value of relative permittivity so the physical size of the capacitor may be as small as possible.

189. Gas Evolution

189.1 Scope—~~This test method~~—Test Method D 2300 describes ~~two procedures each using different apparatus~~ a procedure to measure the rate at which gas is evolved or absorbed by insulating oils when subjected to electrical stress of sufficient intensity to cause ionization. The oil test specimen is initially saturated with a selected gas (usually hydrogen) at atmospheric pressure.

189.2 Summary of Test Method:

189.2.1 Test Method D 2300—After being saturated with a gas (usually hydrogen) the oil is subjected to a radial electrical stress at a controlled temperature. The gas space above the oil is ionized due to the electrical stresses; and therefore, the oil surface at the oil-gas interface is subjected to ion bombardment. The evolution or absorption of gas is measured with a gas burette and reported in $\mu\text{L}/\text{min}$. ~~Two different apparatus are used with this test method (see Procedures A and B). The two procedures give different numerical values of gassing tendency, but rate oils is generally the same order in gas-evolving or absorbing tendencies.~~

~~18.3~~ $\mu\text{L}/\text{min}$.

19.3 Significance and Use—This test method indicates whether insulating oils are gas absorbing or gas evolving under the test conditions. Numerical results obtained in different laboratories ~~or by using two different procedures~~ may differ significantly in magnitude, and the results of this test method should be considered as qualitative in nature.

189.3.1 For certain applications when insulating oil is stressed at high voltage gradients, it is desirable to be able to determine the rate of gas evolution or gas absorption under specified test conditions. At the present time, correlation of such test results with equipment performance is limited.

~~19.~~

20. Resistivity

~~19.1~~

20.1 Scope:

~~19.2.1~~ **20.1.1** This test method covers the determination of specific resistance (resistivity) applied to new electrical insulating liquids, as well as to liquids in service, or subsequent to service, in cables, transformers, circuit breakers, and other electrical apparatus.

~~19.2.2~~ **20.1.2** This test method covers a procedure for making referee and routine tests with ~~the~~ dc potential.

~~19.2~~

20.2 Definition:

~~19.2.1~~ **20.2.1** *specific resistance (resistivity)*—of a liquid, the ratio of the ~~the~~ dc potential gradient in volts per centimetre paralleling the current flow within the test specimen, to the current density in amperes per square centimetre at a given instant of time and under prescribed conditions. This is numerically equal to the resistance between opposite faces of a centimetre cube of a liquid. It is measured in ohm centimetres.

~~19.3~~

20.3 Summary of Test Method:

~~19.3.1~~ **20.3.1 Test Method D 1169**—The oil test specimen is tested in three-terminal, or guarded-electrode test cell maintained at the desired test temperature. A ~~the~~ dc voltage is applied of such magnitude that the electric stress in the liquid is between 200 and 1200 V/mm (5 to 30 V/mL). The current flowing between the high-voltage and guarded measuring electrode is measured at the end of 1 min of electrification and the resistivity calculated using specified equations appropriate to the method of measurement used. A two-electrode cell may be used for routine tests.

~~19.4~~

20.4 Significance and Use—The resistivity of a liquid is a measure of its electrical insulating properties under conditions comparable to those of the test. High resistivity reflects low content of free ions and ion-forming particles and normally indicates a low concentration of conductive contaminants.

21. Stability Under Electric Discharge

21.1 Scope—Test Method D 6180 measures the relative stability of new, used, or reclaimed insulating oils of petroleum origin in the presence of a controlled electric discharge by monitoring the pressure increase in the evacuated discharge chamber.

21.2 Summary of Test Method—A test specimen is introduced into a discharge cell and degassed under vacuum at room temperature. An ac potential of 10 KV is applied for 300 min. The gradual rise of pressure inside the discharge cell is measured as a function of time. The dissipation factor of the oil at 100°C is determined before and after the stability test using Test Method D 924.

21.3 Significance and Use—The changes observed in the generation of gases as noted by pressure change and the composition modification as reflected in dissipation factor increases may provide a relative assessment of the stability of the oil for high voltage application.

CHEMICAL PROPERTIES

202. Acidity, Approximate

202.1 Scope—~~This test method~~—Test Method D 1534 covers the determination of the approximate total acid value of used electrical insulating liquids, in general, those having viscosities less than 24 cSt at 40°C. It is a simple procedure that can be applied in the field.

202.2 Summary of Test Method:

202.2.1 Test Method D 1534—To determine whether the acidity is greater or less than a fixed arbitrary value, a fixed volume of liquid to be tested is added to the test bottle or graduated cylinder, together with a small amount of indicator (phenolphthalein) and the appropriate quantity of standard potassium hydroxide solution. The mixture is shaken and allowed to separate. The color of the aqueous layer at the bottom of the container when testing mineral oils, or at the top when testing askarels, determines whether the acidity is less than or greater than the arbitrary value chosen.

202.3 Significance and Use:

202.3.1 The approximate acidity of used electrical insulating oils is an estimate of the total acid value of the oil. As acid values increase, usually due to oxidation of the oil in service, the impairment of those oil qualities, important to proper functioning of specific apparatus, increases. In general, acidic by-products produce increased dielectric loss, increased corrosivity, and may cause thermal difficulties attributable to insoluble components called “sludge.” This test method is adapted to a specific volume of oil; total acid values of 0.05 to 0.5 mg of potassium hydroxide per gram of oil is a range which is functionally significant.

213. Carbon-Type Composition

213.1 Scope—This test method covers the determination of carbon-type composition of insulating oils by correlation with basic physical properties. Carbon-type composition is expressed as percentage of aromatic carbons, percentage of naphthenic carbons, and percentage of paraffinic carbons. Viscosity, relative density (or specific gravity), and refractive index are the only measurements required for use of this test method.

213.2 Summary of Test Method:

213.2.1 Test Method D 2140—The viscosity, density and specific gravity, and refractive index of the oil are measured. From these values, the viscosity-gravity constant and refractivity intercept are calculated. Using these two computed values, percentage of aromatic carbons, naphthenic carbons, and paraffinic carbons are estimated from a correlation chart.

213.3 Significance and Use—The primary purpose of this test method is to characterize the carbon-type composition of an oil. It is also applicable in observing the effect on oil constitution of various refining processes, such as solvent extraction, acid treatment, etc. and so forth. It has secondary application in relating the chemical nature of an oil to other phenomena that have been demonstrated to be related to oil composition.

224. Compatibility with Construction Material

224.1 Scope—This test method covers screening for the compatibility of materials of construction with electrical insulating oil for use in electrical equipment. Solid materials that can be tested for compatibility include varnishes, dip coatings, core steel, core steel coatings, gaskets, and wire enamels.

224.2 Summary of Test Method:

224.2.1 Test Methods D 3455—The electrical insulating oil and the material whose compatibility is being tested are aged for 164 h at 100°C. Changes in the oil and compatibility sample are observed and appropriate tests conducted.

224.3 Significance and Use:

224.3.1 The magnitude of the change in the electrical properties of the insulating oil is of importance in determining the contamination of the oil by the test specimen.

224.3.2 Physical and chemical changes in the oil such as color, interfacial tension, and acidity also indicate solubility or other adverse effects of the test specimen on the oil.

224.3.3 The physical changes of the test specimen, such as hardness, swelling, and discoloration, show the effect of the oil on the test specimen and are used to determine the suitability of the material for use in insulating oil.

224.3.4 A material meeting the criteria recommended does not necessarily indicate suitability for use in electrical equipment. Other properties must also be considered. Additionally, certain materials containing additives may meet the requirements of this

procedure, yet be unsatisfactory when subjected to longer term evaluations.

235. Copper Content

235.1 Scope:

235.1.1 Test Method D 3635—Covers the determination of copper in new or used electrical insulating oil. For flame atomization, the lower limit of detectability is of the order of 0.1 ppm. For nonflame atomization, the lower limit of detectability is less than 0.01 ppm.

235.2 Summary of Test Method:

235.2.1 Test Method D 3635—The test specimen of oil is diluted with an appropriate organic solvent and analyzed in an atomic absorption spectrophotometer. Alternative procedures are provided for instruments employing flame and nonflame atomization. Concentration is determined by means of calibration curves prepared from standard samples.

235.3 Significance and Use—Electrical insulating oil may contain small amounts of dissolved metals derived either directly from the base oil or from contact with metals during refining or service. When copper is present, it ~~has acts as a tendency to accelerate the deterioration~~ catalyst in promoting oxidation of the oil. This test method is useful for research and to assess the condition of service-aged oils.

246. Furanic Compounds

26.1 Scope—Test Method D 5837 covers the determination, in electrical insulating liquids, of the products of the degradation of cellulosic materials such as paper, pressboard, and cotton material typically found as insulating materials in electrical equipment. These degradation products are substituted furan derivatives, commonly referred to as furanic compounds or furans.

26.1.1 The commonly identified furans that may be identified by this method include: 5-hydroxymethyl-2-furaldehyde, furfuryl alcohol, 2-furaldehyde, 2-acetylfuran, and 5-methyl-2-furaldehyde.

26.2 Summary of Test Method—Furanic compounds in electrical insulating liquids are extracted from a known volume of test specimen by means of a liquid/liquid extraction or solid-phase extraction. A method for direct introduction of oil into the chromatograph is also described. An aliquot of the extract is introduced into a High Performance Liquid Chromatography (HPLC) system equipped with a suitable analytical column and UV detector. Furanic compounds in the test specimen are identified and quantified by comparison to standards of known concentration.

26.3 Significance and Use—Furanic compounds are generated by the degradation of cellulosic materials used in solid insulation systems of electrical equipment. Furanic compounds which are oil soluble to an appreciable degree will migrate into the insulating liquid. High concentrations or unusual increases in the concentration of furanic compounds in oil may indicate cellulose degradation from aging or incipient fault conditions.

27. Gas Analysis

247.1 Scope:

247.1.1 This test method covers ~~two~~ three procedures for the extraction and measurement of gases dissolved in electrical insulating oil having a viscosity of 20 cSt (100 Saybolt Universal seconds) or less at 40°C (104°F), and the identification and determination of the individual component gases extracted.

247.1.2 The individual component gases that may be identified and determined include: hydrogen, oxygen, nitrogen, carbon monoxide, carbon dioxide, methane, ethane, ethylene, acetylene, propane, and propylene.

247.2 Summary of Test Methods:

247.2.1 Method A (Test Method D 3612)—Dissolved gases are extracted from a sample of oil by introduction of the oil sample into a pre-evacuated known volume. The evolved gases are compressed to atmospheric pressure and the total volume measured.

247.2.2 Method B (Test Method D 3612)—Dissolved gases are extracted from a sample of oil by sparging the oil with the carrier gas on a stripper column containing a high surface area bead.

247.2.3 Method C (Test Method D 3612)—The sample is brought in contact with a gas phase in a sealed vial and the dissolved gases are allowed to equilibrate with the gas phase. The headspace above the oil is sampled and analyzed. The amount of dissolved gasses in the oil is calculated from predetermined partition coefficients for each gas.

27.2.4 There may be some differences in limits of detection and precision and bias between Methods A, B, and ~~B~~ C for the various gases.

247.2.45 A portion of the extracted gases (Methods A and C) or all of the gases extracted (Method B) are introduced into a gas chromatograph equipped with suitable adsorption column(s). The composition of the sample is calculated from its chromatogram by comparing the area of the peak of each component with the area of the peak of the same component on a reference chromatogram made on a standard mixture of known composition.

247.3 Significance and Use:

247.3.1 Oil and oil-immersed electrical insulating materials may decompose under the influence of thermal and electrical stresses and in doing so generate gaseous decomposition products of varying composition, which dissolve in the oil. The nature and amount of the individual component gases that may be recovered and analyzed may be indicative of the type and degree of the abnormality responsible for the gas generation. The rate of gas generation and changes in concentration of specific gases over time are also used to evaluate the condition of the electric apparatus.

258. Gas Content

258.1 Scope:

258.1.1 *Test Method D 831*—Electrical insulating oils of low and medium viscosities up to 190 cSt at 40°C (corresponding to 1000 SUS at 100°F), including oils used in capacitors and paper-insulated electric cables and cable systems of the oil-filled type.

258.1.2 *Test Method D 1827*—Electrical insulating liquids with a viscosity of 216 cSt (1000 SUS) or less at 100°C. Acidic gases absorbed by a strong caustic solution are not detectable. Carbon dioxide and hydrogen chloride will not be included in gas content determined by this test method.

258.1.3 *Test Method D 2945*—Electrical insulating oils of up to 19 cSt at 40°C (corresponding to 100 SUS at 100°F). This test method is suitable for either field or laboratory use. It was designed to use a self-contained apparatus in the analysis of oils of low gas content. Unlike Test Methods D 831 and D 1827, gas content is not corrected for ambient temperature or pressure.

258.2 Definition:

258.2.1 *gas content of an oil by volume*—The total volume of gases, corrected to 101 kPa (760 mm Hg) and 0°C, contained in a given volume of oil, expressed as a percentage.

258.3 Summary of Test Methods:

258.3.1 *Test Method D 831*—The oil is fed slowly into a degassing chamber, located in an oven and initially evacuated to a pressure below 13 Pa (0.1 Torr) with a vacuum pump, so that the oil is thoroughly exposed to the vacuum. Condensable gases are removed from the system by a cold trap. The gas volume is calculated from the increase in pressure in the degassing chamber, measured by a McLeod gage.

258.3.2 *Test Method D 1827*—A small liquid sample is purged of dissolved gases with pure carbon dioxide gas. The gas stream is then led into a gas burette containing a potassium hydroxide solution. The carbon dioxide and any other acidic gases are completely absorbed, and the volume of the remaining gas is measured.

258.3.3 *Test Method D 2945*—The oil sample is allowed to flow as a thin film into a chamber evacuated by the lowering of a connecting mercury reservoir. By raising the mercury reservoir, the pressure is returned to atmospheric, and the volume of the evolved gases is measured. No correction is made for atmospheric pressure or ambient temperature.

258.4 Significance and Use:

258.4.1 Some types of electrical equipment require use of electrical insulating liquids of low gas content. Capacitors and certain types of electrical cable, particularly where used at high voltages, may suffer from the formation of gas bubbles with consequent gaseous ionization if gas content is not sufficiently reduced. In filling electrical apparatus, a low gas content reduces foaming and also reduces available oxygen in sealed equipment, increasing the service life of the insulating oil.

258.4.2 These tests are not intended for use in purchase specifications because the oil is customarily degassed immediately before use. These test methods can be used, however, as a factory control test and a control and functional test in installation and maintenance work by utilities. These tests require care in manipulation and the availability of trained, careful personnel.

258.5 Precision:

258.5.1 The precisions of two of the test methods are given in the table below. Refer to the original test methods for the conditions under which these precision values are applicable.

Test Method	Unit	Repeatability	Reproducibility
D 831	Gas Content % (if 0.1 %)	...	±0.02
D 1827	Gas Content % (0.1 to 15 %)	...	±0.05

269. Inorganic Chlorides and Sulfates

269.1 *Scope*—This test method covers the qualitative determination of inorganic chlorides and sulfates in electrical insulating oils.

269.2 Summary of Test Method:

269.2.1 *Test Method D 878*—The electrical insulating oil is extracted with water. The water layer is tested with silver nitrate solution for the presence of chlorides and with barium chloride for the presence of sulfates. If a precipitate is obtained with either reagent, report the corresponding ion as present.

269.3 *Significance and Use*—The presence of inorganic chlorides or sulfates may be due either to improper refining or to contamination from outside sources. The presence of these contaminants may affect the corrosivity or dielectric properties of the oil and may adversely affect its ability to function properly under service conditions.

~~27.~~

30. Neutralization Number

~~27.1~~

30.1 Scope:

2730.1.1 The two procedures available determine the acidic or basic constituents in petroleum products. Because the titration end points of these methods differ, results may differ between the test methods.

2730.1.2 *Test Method D 664*—Resolves the constituents into weak-acid and strong-acid components, provided the dissociation constants of the more highly ionized compounds are at least 1000 times that of the next weaker group. Because the end point is

determined potentiometrically, this test method is suitable for use with very dark samples.

~~2730.1.3~~ *Test Method D 974*—Applicable for the determination of acids or bases whose dissociation constants in water are larger than 10^{-9} . Constituents are classified as strong acid, weak acid, or strong base. Excessively dark-colored oils cannot be tested by this test method due to obscuration of the color indicator end point.

~~27.2~~

30.2 Definitions:

~~2730.2.1~~ *total acid number*—the number of milligrams of KOH required to neutralize all acidic constituents present in 1 g of test specimen. When neutralization number is specified without further qualification, total acid number is implied.

~~2730.2.2~~ *strong acid number*—the number of milligrams of KOH required to neutralize the strong acid constituents present in 1 g of test specimen.

~~27.3~~

30.3 Summary of Test Methods:

~~2730.3.1~~ *Test Method D 664*—The test specimen is dissolved in a mixture of toluene and isopropyl alcohol containing a small amount of water and titrated potentiometrically with alcoholic potassium hydroxide or hydrochloric acid solution, using a glass-indicating electrode and a calomel reference electrode. The meter readings are plotted against the respective volumes of titrating solution, and the end points are taken at the inflections in the resulting curve. When no definite inflections are obtained, end points are taken at meter readings corresponding to those found for standard nonaqueous acidic and basic buffer solutions.

~~2730.3.2~~ *Test Method D 974*—To determine the total acid or strong base number, the test specimen is dissolved in a mixture of toluene and isopropyl alcohol containing a small amount of water, and the resulting single-phase solution is titrated at room temperature with standard alcoholic base or alcoholic acid solution, respectively, to the end point indicated by the color change of the added *p*-naphtholbenzein solution. To determine the strong acid number, a separate portion of the sample is extracted with hot water, and the aqueous extract is titrated with potassium hydroxide solution, using methyl orange as an indicator.

~~2730.3.3~~ *Modified Test Method D 974*—For acid numbers less than 0.04, use of a closed microburet (5 mL, 0.02-mL subdivisions) with preneutralized titration solvent, shielding the titration flask with a rubber cap through which the buret tip extends, is recommended.

~~2730.3.3.1~~ For service-aged test specimens from electrical apparatus in which insulation deterioration could result in the solution of carbon dioxide in electrical insulating oil, the test specimen may be freed of carbon dioxide by blowing for 2 min at room temperature with nitrogen prior to testing.

~~2730.3.3.2~~ When using the modification of Test Method D 974 as noted in 258.3.3, the following precision applies:

~~2730.3.3.3~~ *Repeatability*—Duplicate determinations by the same operator should not differ by more than 0.008. For two operators in the same laboratory, each making duplicate determinations and comparing average values, results should not differ by more than 0.005 (95 % probability).

~~2730.3.3.4~~ *Reproducibility*—Results from two different laboratories, comparing average values from duplicate determinations, should not differ by more than 0.015 (95 % probability).

~~27.4~~

30.4 Significance and Use:

~~2730.4.1~~ A low total acid content of an insulating oil is necessary to minimize electrical conduction and metal corrosion and to maximize the life of the insulation system.

~~2730.4.2~~ In used insulating oils, an increase in total acid number from the value of the unused product indicates contamination by substances with which the oil has been in contact or a chemical change in the oil from processes such as oxidation. An increase in total acid number may indicate the desirability of replacing used with fresh oil, provided suitable rejection limits have been established and other tests confirm the need for the change.

~~28.~~

31. Oxidation Inhibitor Content

~~28.1~~

31.1 Scope:

~~2831.1.1~~ New electrical insulating oil may contain inhibitors to inhibit oxidation. Two test methods are available for the determination of the commonly used inhibitors.

~~2831.1.2~~ *Test Method D 2668*—Determines the concentration of either inhibitor, or their mixtures, in concentrations up to 0.5 mass %, by measuring the infrared absorbance of the oil at selected frequencies.

~~2831.1.3~~ *Test Method D 4768*—This test measures the concentration of either inhibitors or their mixtures, in concentrations up to 0.5 % mass, by gas chromatographic separation and quantitation to a suitable standard.

~~28.2~~

31.2 Summary of Test Methods:

~~2831.2.1~~ *Test Method D 2668*—The infrared absorbance of the test specimen is measured at the frequencies appropriate to 2,6-ditertiary-butyl para-cresol and 2,6-ditertiary-butyl phenol and the concentrations calculated from a calibration curve.

~~2831.2.2~~ *Test Method D 4768*—A column clean-up is employed to remove interfering substances, followed by a gas

chromatographic separation and concentration measured by comparison to suitable standards.

28.3—

31.3 Significance and Use—The quantitative determination of 2,6-ditertiary-butyl para-cresol or 2,6-ditertiary-butyl phenol measures the amount of this material that has been added to new electrical insulating oil as protection against oxidation or the amount remaining in a used oil. These test methods are also suitable for manufacturing control and for use as specification acceptance tests.

329. Oxidation Stability

329.1 Scope:

329.1.1 Three oxidation test methods are applied to insulating oil:

329.1.2 Test Method D 1934—Covers two procedures for subjecting electrical insulating oils to oxidative aging: Procedure A, without a metal catalyst, and Procedure B, with a metal catalyst.

329.1.2.1 This test method is applicable to oils used as impregnating or pressure media in electrical power transmission cables as long as less than 10 % of the oil evaporates during the aging procedures. It applies and is generally useful primarily in the evaluation and quality control of unused oils, either inhibited or uninhibited.

329.1.2.2 The precision statement for Test Method D 1934 should be the standard deviation of the logarithm of the dissipation factor ratios rather than the coefficient of the variation of the ratios.

329.1.3 Test Method D 2112—Is intended as a rapid method for the evaluation of the oxidation stability of new mineral insulating oils containing oxidation inhibitor. This test is considered of value in checking the oxidation stability of new mineral insulating oils containing synthetic oxidation inhibitor in order to control the continuity of this property from shipment to shipment. The applicability of this procedure for use with inhibited insulating oils of more than 12 cSt at 40°C has not been established.

329.1.4 Test Method D 2440—Covers the evaluation of the acid- and sludge-forming tendency of new mineral transformer oils. It is considered of value in studying the acid- and sludge-forming propensity of a new grade of mineral transformer oil before commercial application.

329.2 Summary of Test Methods:

329.2.1 Test Method D 1934—This test method consists of exposing for 96 h 300 mL of oil in a 400-mL beaker to moving air in an oven controlled at 115°C, with or without 15 cm² of metal catalyst. Changes in such properties as color, total acid number, power factor, and resistivity of the aged oil can be used to determine the oxidative deterioration of the oil.

329.2.2 Test Method D 2112—The test specimen is agitated by rotating axially at 100 rpm at an angle of 30° from the horizontal under an initial oxygen pressure of 620 kPa (90 psi) in a ~~bomb~~ pressure vessel with a glass sample container and copper catalyst coil, in the presence of water, at a bath temperature of 140°C. The time for an oil to react with a given volume of oxygen is measured; completion of the test is indicated by a 172 kPa (25 psi) drop in pressure.

329.2.3 Test Method D 2440—The test oil is charged to a glass oxidation tube containing copper wire catalyst. The tube is placed in an oil bath at 110°C, and oxygen is bubbled through separate oil samples for 72 and 164 h. The *n*-heptane insoluble sludge and total acid number of the aged oil is measured to determine the extent of oxidation.

329.3 Significance and Use:

329.3.1 The development of oil sludge and acidity resulting from oxidation during storage, processing, and long service life should be held to a minimum. This minimizes electrical conduction and metal corrosion, maximizes insulation system life and electrical breakdown strength, and ensures satisfactory heat transfer.

329.3.2 The oxidation stability tests described in Section ~~29~~ 32 may be used to evaluate the tendency to form sludge or acids under oxidizing conditions, to ensure the continuity of quality of mineral insulating oil shipments, and for specification purposes. A low tendency to form sludge and acid in laboratory tests is desirable, although the oil showing the least deterioration in the laboratory is not necessarily the best in service.

329.3.3 The oxidation stability tests are used in the following specifications for insulating oils:

D 3487	Mineral Insulating Oil Used in —Electrical Apparatus	D 2112, D2440
D 3487, D 5222	Mineral Insulating Oil Used in —Electrical Apparatus	D 2112, D2440

303. Polychlorinated Biphenyl Content

303.1 Scope:

303.1.1 Test Method D 4059—Describes a quantitative technique for determining the concentration of polychlorinated biphenyls (PCB²s) in electrical insulating liquids by gas chromatography.

303.2 Definition:

303.2.1 PCB concentration—is normally expressed in units of parts per million (PPM) on a weight by weight basis. Standard chromatograms of Aroclors ⁶ 1242, 1254, and 1260 are used to determine the concentration of PCB in the sample.

303.3 Summary of Test Method—Following dilution of the test specimen in a suitable solvent, the solution is treated to remove interfering substances. A small portion is then injected into a gas chromatographic column where the components are separated

⁶ Registered trademark of Monsanto Co.

and their presence measured by an electron capture or halogen-specific electrolytic conductivity detection. The test 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.

303.4 *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.

314. Sediment and Soluble Sludge

314.1 *Scope*—This test method covers the determination of sediment and soluble sludge in service-aged insulating oils of petroleum origin. Also, provision is made for determining organic and inorganic content of the sediment. The test method is intended primarily for oils of comparatively low viscosity, for example, 7 to 15 cSt at 40°C. Suitability for high-viscosity oils has not been determined.

314.2 *Summary of Test Method:*

314.2.1 *Test Method D 1698*—A test specimen portion is centrifuged to separate sediment from the oil. The upper, sediment-free portion is decanted and retained for determination of soluble sludge. The sediment is dislodged and filtered through a specially prepared Gooch crucible. After drying and weighing to obtain total sediment, the crucible is ignited at 500°C and reweighed. Loss in weight is organic and remainder is inorganic content of sediment. Soluble sludge is determined on sediment-free portion by dilution with *n*-pentane to precipitate *n*-pentane insolubles, and filtration through a Gooch crucible.

314.3 *Significance and Use:*

314.3.1 Sediment in insulating oil may deposit on transformer parts and interfere with heat transfer, and may choke oil ducts, and so hinder oil circulation and heat dissipation. Inorganic sediment usually indicates contamination of some type, and organic sediment indicates either deterioration of the oil or contamination.

314.3.2 Soluble sludge indicates deterioration of the oil, presence of contaminants, or both. It serves as a warning that formation of sediment may be imminent.

314.3.3 The determination of sediment and soluble sludge in a used insulating oil assists in deciding whether the oil may continue to be used in its existing condition or should be replaced, reclaimed, or reconditioned.

325. Sulfur, Corrosive

325.1 *Scope*—This test method covers the detection of corrosive sulfur compounds in electrical insulating oils of petroleum origin. Compounds capable of severely discoloring a copper surface under prescribed test conditions are reported as corrosive.

325.2 *Summary of Test Method:*

325.2.1 *Test Method D 1275*—A polished copper specimen is exposed to the test oil for 19 h at 140°C. The appearance of the copper surface determines whether the oil is classified as corrosive or noncorrosive.

325.3 *Significance and Use*—In most of their uses, insulating oils are continually in contact with metals that are subject to corrosion, such as copper or silver. Since the presence of detrimental corrosive sulfur compounds will result in deterioration of these metals to an extent dependent upon the quantity and type of corrosive agent and the time and temperature factors, the detection of these undesirable impurities, even though not in terms of quantitative values, is a means for recognizing the hazard involved.

336. Water Content

336.1 *Scope*—~~These test methods cover~~—Test Method D 1533 covers the determination of water present in insulating liquids, in concentrations most commonly below 200 ppm.

336.2 *Summary of Test Methods:*

336.2.1 ~~These test methods are~~ method is based on the reduction of iodine in accordance with the traditional Karl Fischer reaction.

~~33.2.2 Methods A and C utilize iodine present in a titration solution, while~~

~~36.2.2 Test Method B D 1533 electrochemically generates the iodine in the equipment. Method A uses the traditional required for Karl Fischer reagent, while Method C uses a two-part buffer titrant solution.~~

~~33.2.3 Method A—Using an titration.~~

~~36.2.3 This automatic buret, the end point is determined by measuring the current flow between two platinum electrodes maintained at a 20 to 50 mV potential. The chloroform-methanol titration solvent is first dehydrated with Karl Fischer reagent, the oil test specimen added, and the titration continued until the microgalvanometer or recorder chart reading indicates the end point (depolarization of the platinum cathode).~~

~~33.2.4 Method B—The automatic coulometric titration procedure requires the use of an instrument that is designed and calibrated to deliver a known electrical current which generates sufficient iodine to neutralize a known weight of water per minute. The two-part titration solution is first brought to near a zero dryness by iodine produced by the generator when the controls are placed in the “standby” setting. The test specimen is added; and the titration begun, allowing the test specimen to be automatically titrated by producing iodine at the generator anode until the equivalent point is reached and the titration is complete. Water content is read directly on the meter in micrograms (or parts per million).~~

~~33.2.5 Method C~~—This method utilizes the same apparatus as Method A but is improved by the addition of a buffer solution which increases the reaction rate, thus making a sharper, more stable endpoint. In addition, the use of pyridine may be eliminated, and no yellow SO_2I^- complex is formed to obscure the visual endpoint.

~~33.3~~—

36.3 Significance and Use—A low water content of insulating oil is necessary to achieve adequate electrical strength and low dielectric loss characteristics, to maximize the insulation system life, and to minimize metal corrosion. Water in solution cannot be detected visually and must be determined by other means. This test shows the presence of water that may not be evident from electrical tests.

SPECIFICATIONS

347. Mineral Insulating Oil for Electrical Apparatus

347.1 *Scope*:

347.1.1 *Specification D 3487*—The physical, chemical, and electrical properties of two types of new mineral insulating oil of petroleum origin for use as an insulating and cooling medium in new and existing power and distribution electrical apparatus, such as transformers, regulators, reactors, circuit breakers, switchgear, and attendant equipment are given in this specification.

347.1.2 Type I oil has a maximum oxidation inhibitor content of 0.08 mass % and Type II oil a maximum of 0.3 mass %. Except for the inhibitor content and oxidation stability requirements, the two oils have similar performance properties.

347.1.3 Specification D 3487 is intended to define a mineral insulating oil that is functionally interchangeable with existing oils, is compatible with existing apparatus and with appropriate field maintenance, and will satisfactorily maintain its functional characteristics in its application in electrical equipment. This specification applies only to new insulating oil prior to introduction into apparatus.

GUIDE

~~35~~—

38. High Fire-Point Electrical Insulating Oils

358.1 *Scope*:

35.1.1 *This guide*

38.1.1 *Specification D 5222* lists properties of ~~an example of a~~ fresh unused high fire-point insulating oil of petroleum origin for use as a dielectric and cooling medium in new power and distribution electrical apparatus, such as transformers and switchgear. The users should contact the manufacturers of the equipment or oil if questions of recommended characteristics or maintenance procedures arise.

358.1.2 High fire-point insulating oil differs from conventional mineral insulating oil by possessing a fire-point of at least 300°C. This property is necessary in order to comply with certain application requirements of the National Electric Code (Article 450-23) or other agencies.

369. Keywords

369.1 chemical properties; electrical insulating oils; electrical properties; measured; physical properties; properties; sampling; specification

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