



# Standard Test Method for Stability of Insulating Oils of Petroleum Origin Under Electrical Discharge<sup>1</sup>

This standard is issued under the fixed designation D 6180; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers a laboratory technique that measures the stability of new, used, or reclaimed insulating oils, similar to those described in Specification D 3487 in the presence of a controlled electric discharge. When subjected to this type of discharge, insulating oils absorb energy and produce gases as well as ionized molecules (charge carriers). The quantity of these decay products can be measured and can provide an indication of the stability of oils under the conditions of this test.

1.2 The gases are retained in the discharge cell and their pressure measured. The charge carriers remain in the test specimen. The change in the dissipation factor before and after the discharge is determined.

1.3 The values stated in SI units are to be regarded as the standard. The values stated in parentheses are for information only.

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.* Specific cautionary statements are given in 5.3 and 7.1.

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 923 Practice for Sampling Electrical Insulating Liquids<sup>2</sup>

D 924 Test Method for Dissipation Factor (or Power Factor) and Relative Permittivity (Dielectric Constant) of Electrical Insulating Liquids<sup>2</sup>

D 3487 Specification for Mineral Insulating Oil Used in Electrical Apparatus<sup>2</sup>

### 2.2 IEEE Standard:

4–1995 IEEE Standard Techniques for High-Voltage Testing<sup>3</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.05 on Electrical Test.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 10.03.

<sup>3</sup> Available from the Institute of Electrical and Electronic Engineers, Inc., P.O. Box 1331, Piscataway, NJ 08855.

## 3. Summary of Test Method

3.1 A test specimen is introduced into a discharge cell and degassed under vacuum at room temperature. An ac potential of 10 kV is applied between a high voltage electrode and a grounded salt water electrode for 300 min. The gradual rise of the pressure inside the discharge cell is measured on an electronic vacuum meter as a function of time. The dissipation factor of the oil at 100°C is determined before and after the stability test.

## 4. Significance and Use

4.1 During this test, insulating oil in an evacuated cell is subjected to a high voltage discharge between two electrodes. The discharge generates free electrons. These electrons collide with the oil molecules causing many of them to become electronically excited. Some of these molecules lose this energy as a quanta of light emitting fluorescent radiation. Some of the other excited molecules decompose into gases, ionized molecules and free radicals. These changes can provide an indication of the stability of oils under the conditions of this test method. The measures of these changes are the increase of the pressure in the test cell and the increase in the dissipation factor of the test specimen.

4.2 During the test, the gas content increases in the cell and the concentration of charge carriers increases in the oil.

## 5. Apparatus

5.1 *Discharge Cell*,<sup>4</sup> shown in Fig. 1, includes an electronic vacuum meter. The cell must be made of quartz, be of spherical shape, and have a 500 mL capacity. The electrode is sealed in the cell as shown in Fig. 1. The free electrons are generated by a cylindrical copper electrode 15 mm (0.6 in.) in diameter and 10 mm (0.4-in.) long, which is placed in the center of the discharge cell.

5.2 *Glass Dish*, approximately 150 mm (6 in.) in diameter and 100 mm (4 in.) deep for holding a salt-water ground electrode.

5.3 *Test Chamber*, with safety interlocked door, which deenergizes the test transformer when opened and large enough

<sup>4</sup> A suitable discharge cell is obtainable from Insoil Canada Ltd., 231 Hampshire Place, N.W., Calgary, AB Canada T3A 4Y7.

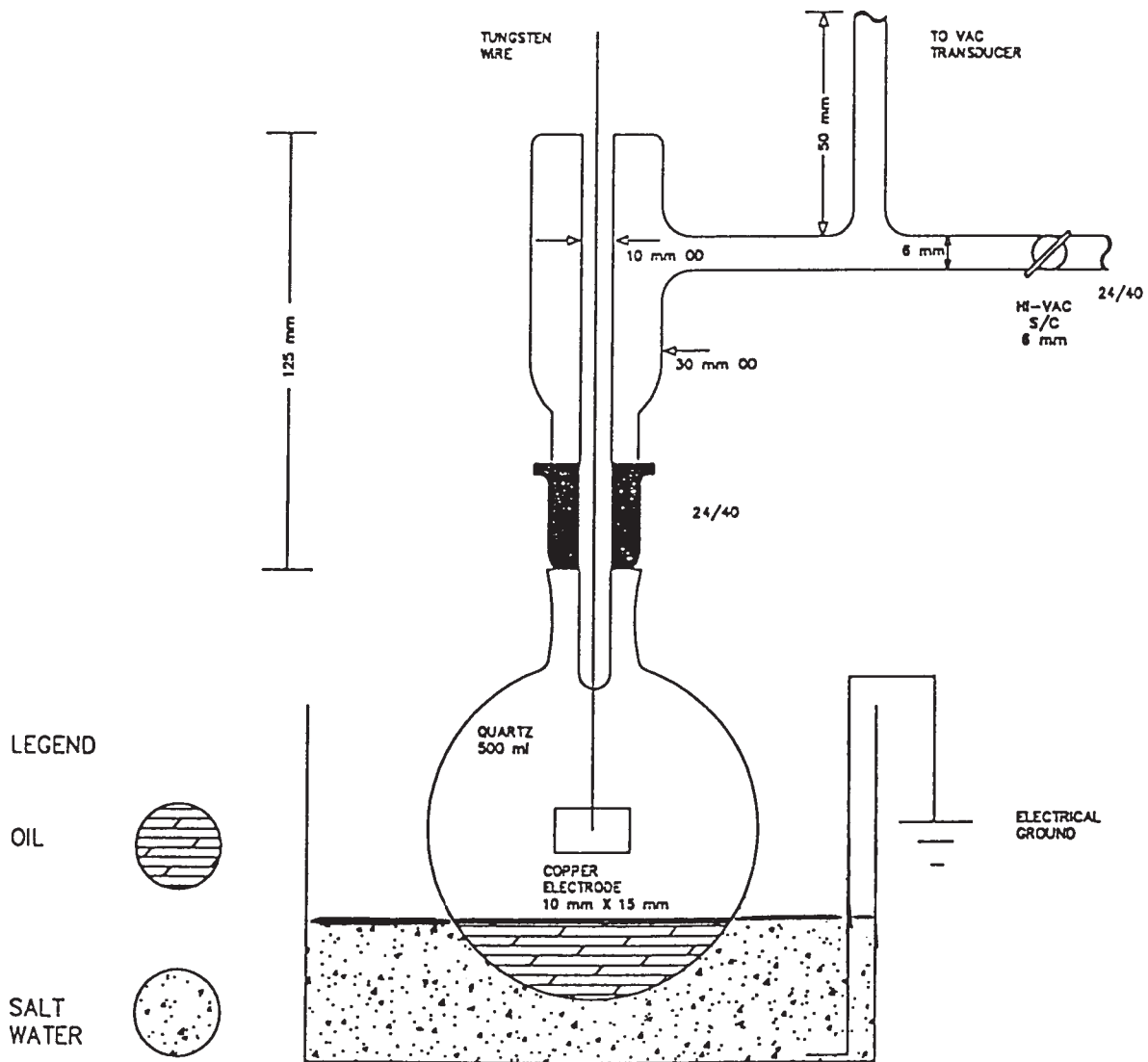


FIG. 1 Discharge Cell

to contain the desired number of cells with at least 100 mm (4 in.) clearance between cells, sides, and top.

5.4 *Power Source Step-Up Transformer*, 60 Hz, 200 VA, 115 or 230 V to at least 10 kV. Design the transformer of such a size that, with the test specimen in the circuit, the voltage waveshape is approximately a sinusoid with both half cycles alike, and it should have a ratio of peak-to-rms value equal to the  $\sqrt{2} \pm 5\%$ .

5.5 *Variable-Tap Autotransformer or Equivalent*, 200 VA min, 115 or 230 V for applying voltage to transformer.

5.6 *Relay*, 115 or 230 V, double-pole, for energizing transformer.

5.7 *Switch*, double-pole, 115 or 230 V, on-off switch for applying voltage to circuit.

5.8 *Voltmeter*—Measure the voltage by a method that fulfills the requirements of IEEE Standard No. 4<sup>3</sup>, giving rms values, preferably by means of:

5.8.1 A voltmeter connected to the secondary of a separate potential transformer, or

5.8.2 A voltmeter connected to a well-designed tertiary coil in the test transformer, or

5.8.3 A voltmeter connected to the low voltage side of the testing transformer if the measurement error can be maintained within the limit specified in 5.9.

5.9 *Accuracy*—The combined accuracy of the voltmeter and voltage measuring circuit should be such that the measurement error does not exceed 5%, calibrated by means of a recommended method in IEEE Standard No. 4.<sup>3</sup>

5.10 *Vacuum Pump*, laboratory-type capable of attaining a vacuum of less than 100 Pa.

5.11 *Clamp Stand*, for supporting cell in chamber.

5.12 *Vacuum Gage*, Electronic vacuum meter.

## 6. Sampling

6.1 Insulating oils for this test should be sampled in accordance with Test Method D 923.

## 7. Cleaning Test Cell

7.1 Before using the cell, it should be thoroughly flushed out with suitable solvent such as petroleum ether or heptane.

NOTE 1—**Caution:** Solvents should be used under a ventilated hood.

Deposits that are not removed by solvent cleaning may accumulate on the side of the flask. If this occurs, the deposit should be removed by mechanical means, using a strong detergent and brush to ensure that the cell is free of all impurities.

7.2 The electrode also should be free of liquid dielectric or solid deposits which may be accumulated on its surface. This can be done using very fine sand paper followed by wiping the surface with filter paper to remove any residual sand paper grit. Finally, rinse the cell with distilled water followed by a volatile solvent such as alcohol. Remove as much of the water-solvent from the cell as possible. The last traces of water-solvent will evaporate from the cell and electrode during the 30 min degassing operation.

**8. Procedure**

8.1 Test up to four cells at the same time, preparing and connecting each in the same manner to the high voltage and vacuum system.

8.2 Assemble each clean discharge cell using an Apiezon M<sup>5</sup> or equivalent stopcock grease on all joints. Evacuate each cell to an absolute pressure of 100 Pa or less, then close the valve and shut off the pump. After 15 min, there should be no increase in the pressure readings; if there is, locate and stop the leak.

8.3 Determine the dissipation factor at 100°C of the liquid dielectric to be tested. See Test Method D 924.

8.4 Use a pipet to introduce a test specimen of 50 ± 0.5 mL. Degas the reassembled cell for 30 min. After isolating the discharge cell from the vacuum source, ensure pressure is maintained at 100 Pa or less for 15 min to prove no leaks have been introduced.

8.5 Place a glass dish containing 500 mL of tap water at room temperature with 10 g of dissolved sodium chloride, technical grade, under each discharge cell. The level of the salt solution must be the same as that of the test specimens for each test station (see Fig. 1). For each dish, use a stainless steel wire of approximate length 500 mm (19 in.) and diameter 0.5 mm (0.02 in.) to form a ring around the inside circumference of the dish containing the salt solution. This wire must be below the surface of the salt solution. Connect this wire to the ground.

8.6 Ground one terminal of the high-voltage transformer and connect the other terminal to the high-voltage electrode.

8.7 Perform this test method at room temperature maintained at 25 ± 5°C. With the door of the test chamber closed, raise the voltage no faster than 0.5 kV/s to approximately 3 to 4 kV, at which voltage a sudden burst of foam will appear on the surface of the test specimen. After the foam subsides, increase the voltage no faster than 1 kV/s to 10 kV. Maintain discharge voltage for 5 h.

NOTE 2—An emission of bluish fluorescent light provides evidence that, under the impact of accelerated electrons, a significant number of hydrocarbon molecules become electronically excited. Some of them decompose evolving gases.

<sup>5</sup> Hydrocarbon grease for use at normal room temperature with a vapor pressure of 2.6 × 10<sup>-7</sup> Pa at 20°C. Available in the U. S. A. from AVO Biddle Instruments, 510 Township Line Road, Blue Bell, PA 19422.

8.8 Measure the pressure inside the discharge cell in Pa by reading the electronic vacuum meter to obtain a measure of the quantity of gases evolved.

8.9 Remove the test specimen from the cell and determine its dissipation factor at 100°C.

**9. Report**

9.1 Report the following information:

9.1.1 Initial pressure of the Discharge Cell at beginning of test.

9.1.2 The dissipation factor of the dielectric liquid in percent at 100°C before the stability test.

9.1.3 Voltage impressed across Discharge Cell during test.

9.1.4 Length of time voltage was impressed across Discharge Cell.

9.1.5 Pressure of the Discharge Cell at end of test.

9.1.6 The dissipation factor of the dielectric liquid in percent at 100°C after the stability test.

**10. Precision and Bias**

10.1 *Precision*—The precision of this test method has not been investigated through an interlaboratory test program.

10.2 *Repeatability*— An estimate of the repeatability has been developed based on data supplied by a single laboratory. This data is listed in Table X1.1, Table X1.2 and Table X1.3 of the Appendix. These results suggest that the 95 % repeatability limit for the pressure measurement is 199 Pa. Also, these results suggest that the 95 % repeatability limit for the dissipation factor measurement is 0.50 %.

10.3 *Reproducibility*— No data are available on which to base an estimate of the reproducibility or this test method. An interlaboratory test program will be conducted to develop this data.

NOTE 3—Fig. 2 and Table 1 show test results produced by this test method. Fig. 2 shows the absolute pressure change for three mineral oils. Table 1 shows the dissipation factor change for three mineral insulating oils. Sample No. 1 is an oil sample, which has been reclaimed by Fuller’s Earth. Upon reclaiming, the color of Sample No. 1 was reduced from 3.5 to 1.5. Sample No. 2 is an oil sample, which has never been in a transformer. Sample No. 3 is an oil sample, which was taken from an operating transformer, which was experiencing an unexplained gassing problem.

10.4 *Bias*—No information can be presented on the bias of the procedure in this test method for measuring the pressure change or the dissipation factor because no material having an accepted reference value is available.

**11. Keywords**

11.1 dissipation factor; electrical discharge; gas formation; gassing tendency; insulating oil; oxidation stability; stability

**TABLE 1 Dissipation Factor Increase of Oils Subjected to an Electrical Discharge**

Oil Type	Dissipation Factor at 100°C, %	
	Before Test	After Test
Reclaimed	0.38	0.72
New	0.32	0.94
Aged	0.83	3.36

STABILITY TEST

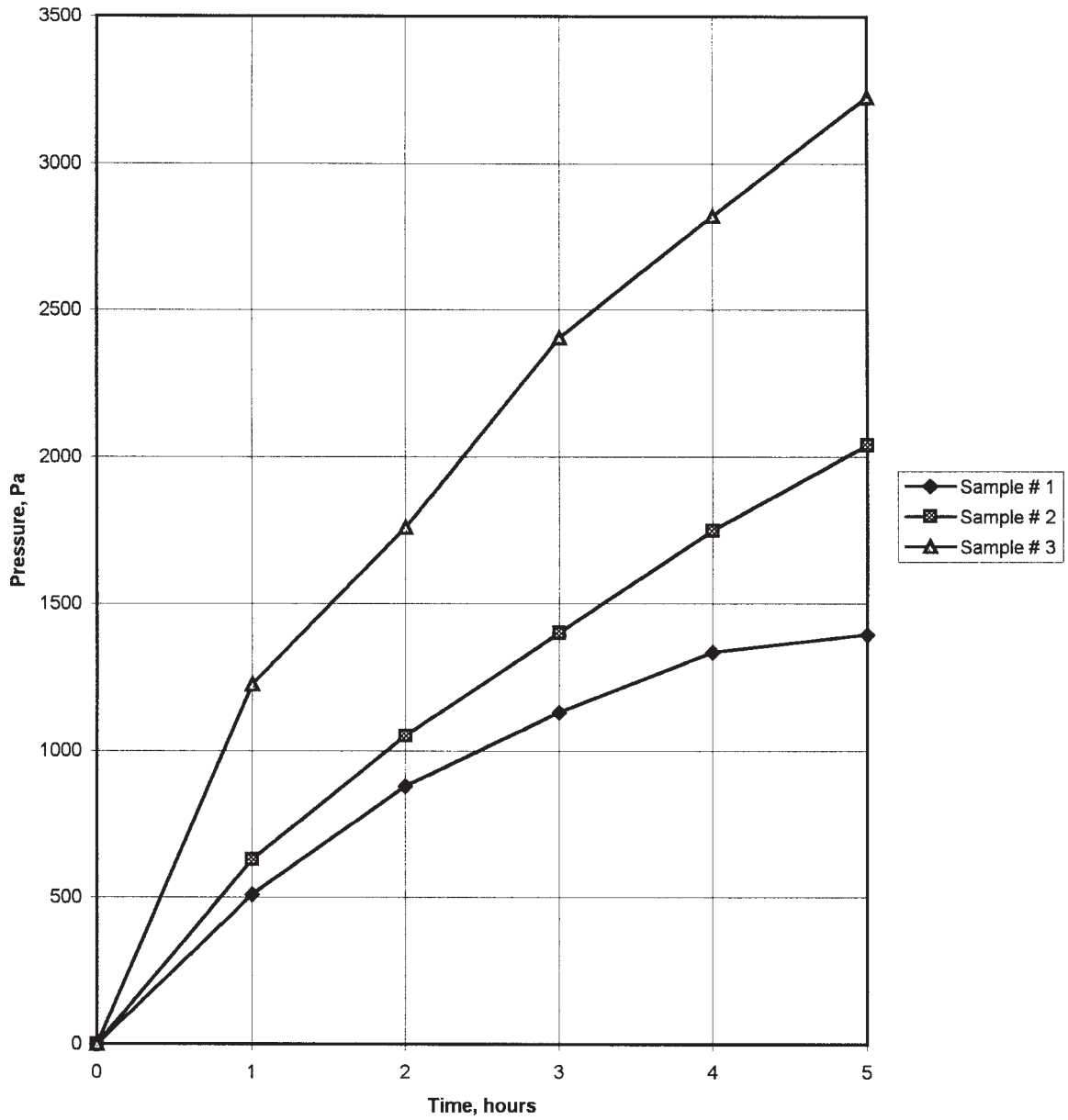


FIG. 2 Pressure Increase With Time for Insulating Oils Subjected to Electrical Discharge

**APPENDIX**
**(Nonmandatory Information)**
**X1. DATA USED TO DEVELOP PRECISION AND REPEATABILITY ESTIMATES**

X1.1 Data are given from a single laboratory for the

**TABLE X1.1 Results of Stability Measurement<sup>A</sup>**

Date Tested	Pressure, Pa	Dissipation Factor at 100°C
January 20, 1997	2033	0.97
January 29, 1997	2117	n/a
January 31, 1997	2099	n/a
February 21, 1997	2133	1.23
February 24, 1997	2150	1.25
March 26, 1997	2199	1.00
March 27, 1997	2259	0.97

<sup>A</sup> The above results were obtained by testing a new mineral transformer oil.

measurement of stability for a single new oil sample. The stability tests are the results of testing on several different days and are presented in Table X1.1.

**TABLE X1.2 Results of Stability Measurement<sup>A</sup>**

	First New Oil		Second New Oil	
	Pressure, Pa	Dissipation Factor @ 100°C, %	Pressure, Pa	Dissipation Factor at 100°C, %
Test 1	2266	1.25	2319	1.10
Test 2	2199	0.94	2173	0.86

<sup>A</sup>The above results were obtained by testing two new transformer mineral oils.

**TABLE X1.3 Results of Stability Measurement<sup>A</sup>**

	First New Oil		Second New Oil	
	Pressure, Pa	Dissipation Factor @ 100°C %	Pressure, Pa	Dissipation Factor at 100°C %
Test 1	1733	1.95	1733	1.33
Test 2	1800	1.67	1733	1.66
Test 3	1733	2.11	1866	1.54

<sup>A</sup>The above results were obtained by treating two new transformer mineral oils.

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