Designation: D831/D831M – 12

# Standard Test Method for Gas Content of Cable and Capacitor Oils[1]

This standard is issued under the fixed designation D831/D831M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of the gas content of electrical insulating oils of low and medium viscosities in the general range up to 190 $mm^2/s$ at 104°F [40°C], such as are used in capacitors and paper-insulated electric cables and cable systems of the oil-filled type. The determination of gas content is desirable for any insulating oil having these properties and intended for use in a degassed state.

NOTE 1—For testing insulating oils with viscosities of 19 $mm^2/s$ or below at 40°C, see Test Method D2945.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.3 *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:*

D2945 Test Method for Gas Content of Insulating Oils[2] (Withdrawn 2012)[2]

## 3. Summary of Test Method

3.1 This test method consists essentially of feeding the oil into an evacuated chamber in such a manner that the oil is thoroughly exposed to the vacuum, allowing free escape of any dissolved gas. From the volume of oil admitted to the chamber, the temperature, the pressure produced, and volume occupied by the released gas, the gas volume under standard conditions of pressure and temperature may be calculated as a percentage by volume of oil.

## 4. Significance and Use

4.1 The gas content of cable and capacitor oils is considered to be important, since the evolution of gas in the form of bubbles can have an adverse effect on the insulating properties of these fluids. It is customary to degas these oils prior to use, and this test method provides a means of determining the gas content before and after degassing.

## 5. Apparatus (see Fig. 1)

5.1 *Degassing Chamber*—Degassing chamber, *A*, made of heat-resistant glass[4] (with calibrated oil well at bottom), having a fixed total space volume of about 175 to 300 mL. The oil well shall have a maximum capacity of 50 mL and shall be calibrated in 0.2-mL divisions.

5.2 *Stopcocks*—Glass stopcocks, *B* and *C*, which shall have large-diameter barrels and a mirror finish to ensure against leakage. Use stopcock grease[5] on all stopcocks and ground-glass joints.

5.3 *Atomizer*—Glass pipet, *D*, placed to drop oil on the side of the degassing chamber, or

5.3.1 *Fritted Disk (Alternative)*—Capacity 30 mm, medium-porosity.

NOTE 2—Some experience has shown improvement in the atomization process, particularly for oils of medium viscosity above 95 $mm^2/s$ at 40°C, if a 30-mm medium-porosity fritted disk is substituted for the pipet.

5.4 *Pressure Gage*— Pressure gage, *E*, of modified McLeod type.[6] Include the volume of this gage in the over-all volume of

[1] This test method is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gasesand is the direct responsibility of Subcommittee D27.03 on Analytical Tests.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

[4] Borosilicate glass has been found to be satisfactory for this purpose.

[5] A stopcock grease equivalent to No. 15521. A readily available vacuum sealing compound. Dow Corning grease #1597418, has been found to be satisfactory for this use.

[6] A McLeod gauge Flosdorf modification, available from Sigma-Aldrich Company, has been found satisfactory for this purpose. The sole source of supply of the apparatus known to the committee at this time is Sigma-Aldrich Company. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

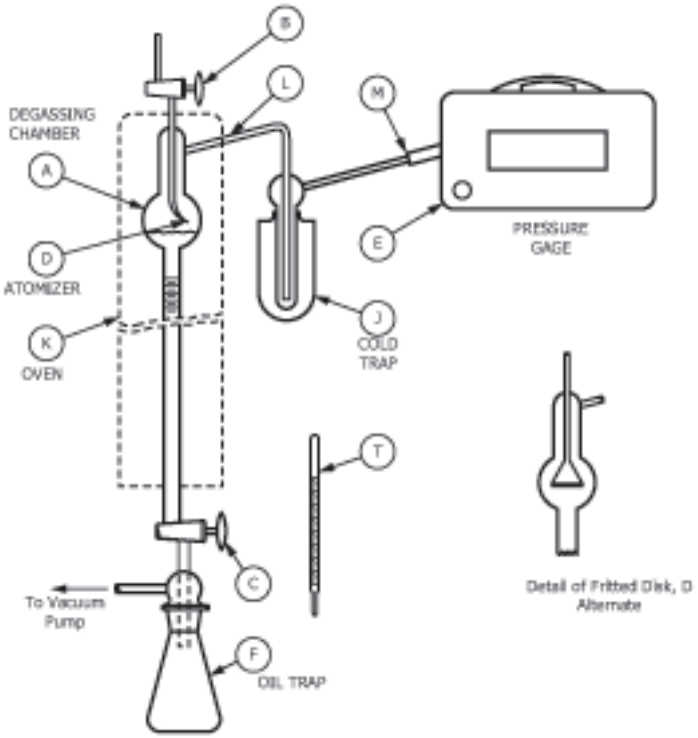


**FIG. 1 Apparatus for Determination of Gas Content of Cable and Capacitor Oils**

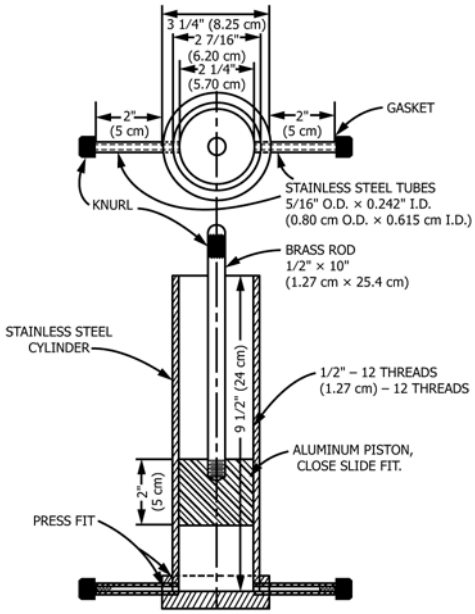


**FIG. 2 Apparatus for Sampling Oils**

the apparatus (Note 3). This is essential and must also include the volume of the gage-connecting tubing.

NOTE 3—The volume of the gage may be obtained from the manufacturer or measured.

5.5 *Oil Trap, F,* having a capacity of 250 mL.

5.6 *Thermometer, T,* room ambient.

5.7 *Cold Trap, J,* employed to eliminate possible error due to presence of condensable vapors.

5.8 *Oven, K,* employed to enhance the atomization process. The oven shall enclose the degassing chamber, *A*, between stopcocks *B* and *C*, and point *L*. Provide suitable means for reading, maintaining, and regulating temperature in a range from 30 to 150°C. Measure temperature by means of a thermocouple fastened to the oil chamber at the 25-mL mark and suitably shielded to eliminate radiation errors.

## 6. Sampling

6.1 When convenient, connect the degassing chamber of the measuring equipment directly to the container from which the oil is to be sampled. This is usually not convenient and is often impossible. The method of sampling described in 6.2 is recommended as an alternative.

6.2 Use the sample container as shown in Fig. 2, which consists of a stainless steel cylinder 2¼ in. [5.7 cm] in inside diameter and 9½ in. [24 cm] in length, closed at the bottom. An aluminum piston, accurately machined for an easy sliding fit, is inserted in the bore of the cylinder. Two nipples, diametrically opposite each other, are inserted at the extreme bottom of the cylinder. Each nipple has a screw plug at the end with a gasket for sealing. Make all connections to the measuring equipment from the sample container using glass or metal tubing. Connect butted joints using short sections of heavy-walled rubber tubing. Coat the tubing thoroughly with suitable sealing compound. Take all samples under slight oil pressure, with the following sequence of operations: Remove plugs from both nipples. Push the piston to the extreme bottom of the cylinder. Hold the cylinder so that the nipples point in a vertical direction. Using a rubber tube connection, force oil in through the nipple in the lowest position and flush a few millilitres out the opposite nipple to remove any trapped air bubbles. Then insert the plug in the outlet nipple and allow oil to push the piston up to fill the cylinder. Hold the piston at the top of the cylinder with one hand and plug the inlet nipple. The aluminum piston "floats" on the oil as the level varies due to temperature changes or removal of test specimens and prevents contamination by absorption and diffusion. Fit the piston accurately so it moves down freely with decreasing oil level to prevent voids forming under the piston which would allow rapid absorption of air by the top oil. Draw the test specimen continuously from the cylinder, weight the piston to ensure maintenance of contact with the oil. Wide variations in the result are possible in two test specimens from the same source unless the greatest care is taken in the sampling procedure. This phase of the test is so involved with the details of what constitutes correct practice that ability to procure consistent representative test specimens depends, to a great extent, on wide experience. The chief precaution to the procurement of representative test specimens involves a complete flushing of all piping and hose between the sample container and sample source, such as pothead, joint, cable, oil reservoir, etc., immediately preceding collection of the sample. Eliminate all long sampling pipe lines. After taking the test specimen, ensure that the piston always remains in contact with the oil prior to and during withdrawal of the test specimen.

## 7. Calibration of Apparatus

7.1 Calibration and conditioning of the apparatus are often done by the manufacturer. If this has not been done, or if a check is desired, the following procedures may be used:

7.1.1 Clean the glass assembly with a cleaning solution, wash with distilled water, and dry. Weigh the empty glass apparatus with a slight film of grease on the stopcocks. Record the weight in grams (or volume in millilitres) as *W*. Fill the glass apparatus between stopcocks *B* and *C* and point *M* with distilled water and weigh. Record the weight in grams (or volume in millilitres) as *R*. The volume represented by the difference between *W* and *R* added to the volume of the pressure gage (Note 4) is the volume, *V*, of the tester in millilitres. The volume, $V_d$, of the degassing chamber enclosed by the oven and the volume, $V_t$, of the cold trap covered with coolant may be determined similarly. Correction for temperature of water may be made, but is an unnecessary refinement.

7.1.2 Empty and thoroughly dry the glass apparatus in an oven. Grease the stopcocks and mount the apparatus on a suitable frame. When properly applied, the grease will coat the whole conical surface of all ground glass, with less than 5 % of said grease extruding beyond the ground surface.

7.1.3 An alternative method of calibration on the completed assembly to determine the volume, *V*, utilizes a gas buret procedure. After evacuating the assembled apparatus, including the vacuum gage, to 0.1 mm Hg or less, a calibrated amount of air at atmospheric pressure is allowed to expand into the evacuated apparatus and the resultant pressure recorded. The total apparatus volume may then be simply calculated.

## 8. Procedure

8.1 Raise the temperature of the oven to the desired level and make certain that it remains constant within ±2°C for at least 30 min. The temperature of the cold trap will depend on the coolant employed. (The temperature of liquid air, or liquid nitrogen, is – 195°C and of carbon dioxide snow is – 78°C.)

8.2 Evacuate the degassing chamber with stopcock *B* closed and stopcock *C* open.

8.3 When the McLeod gage is reasonably constant and less than 0.1 mm Hg (absolute pressure), close stopcock *C* for about 15 min to test for any leaks. In this time, if the pressure rises by more than 5 % of its initial absolute value, search for sources of leakage. (Let the vacuum pump run continuously to enhance the seal of stopcock *C*.)

8.4 Flush oil slowly from the sample through stopcocks *B* and *C* so as to wash thoroughly the walls of the glass tubing and to remove any trapped air bubbles in the system above stopcock *B*. (About 50 mL of oil are commonly used for this flushing.)

8.5 Re-evacuate the degassing chamber, close stopcock *C*, read the McLeod gage and record as $P_1$, then by careful manipulation of stopcock *B* feed oil from the sample container into the degassing chamber at a very slow rate so that the oil falls in single drops from pipet *D*. This rate will generally average 1 drop/s or less. A fritted disk, when employed, requires no regulation as it is self-monitoring. In the case of any significant positive pressure, a suitable means of reducing the pressure should be employed so as to allow the fritted disk to be self-monitoring. The oil should show no appreciable tendency to bubble or foam after falling into the oil well at the bottom of the degassing chamber. When this is seen to occur, the rate of flow should be decreased.

8.6 When 50 mL of oil (Note 4) have been degassed, close stopcock *B* and read the pressure, $P_2$, on the McLeod gage. Record $P_2$, room temperature, $t_a$, oven temperature, $t_d$, and cold trap temperature, $t_t$.

NOTE 4—Use sample aliquots smaller than 50 mL for insulating liquids having high gas content.

8.7 Remove the sample container from the apparatus with stopcock *B* closed. Discharge the sample from the oil well by opening stopcock *C*. Vent the apparatus to atmosphere by careful manipulation of stopcock *B*.

## 9. Calculation

9.1 Calculate the gas content as follows:

$$G = \frac{35.9}{V_1}\left[\frac{(V - V_d - V_t)(P_2 - P_1)}{273 + t_a} + \frac{(V_d - V_1)P_2 - V_d P_1}{273 + t_d} + \frac{V_t(P_2 - P_1)}{273 + t_t}\right]$$

where:

$G$ = gas content (at 760 mm Hg and 0°C) expressed as a percentage by volume of oil,
$V$ = known total fixed volume of apparatus, mL,
$V_1$ = volume of oil, mL,
$V_t$ = volume of cold trap covered by coolant, mL,
$V_d$ = volume of degassing chamber enclosed by oven, mL,
$P_1$ = initial pressure of gas in apparatus, mm Hg,
$P_2$ = final pressure of gas in apparatus, mm Hg,
$t_a$ = ambient temperature,° C,
$t_d$ = temperature of degassing chamber *A*, °C, and
$t_t$ = temperature of cold trap *J*, °C.

NOTE 5—The calculation may appear to be tedious, but when the fixed volumes and temperatures are substituted in the equation, it will be found that it can be simplified considerably. Simplification will also be aided if $P_1$ can be made low enough to be negligible.

## 10. Precision and Bias

10.1 *Precision*—This test method has been found to be reproducible to ±0.02 % gas content for gas contents on the order of 0.10 % (Note 6); for samples of higher gas content the absolute error will be somewhat greater but the percentage error somewhat less.

NOTE 6—Consider the case of an oil with 0.10 % gas. For normal procedure, values of initial pressure and end point would be 0.100 and 0.300 mm Hg, respectively. This pressure increase would be developed by 50 mL of oil tested at 50°C in an apparatus having a total fixed-space volume of approximately 225 mL, the components of which have the following volumes and temperatures: degassing chamber, 125 mL and 50°C; cold trap, 25 mL and – 195°C; pressure gage, 75 mL and 27°C. The Stokes gage (Model 276-AA) can be read to ±0.005 mm below 0.100 mm; ±0.025 mm between 0.100 and 0.700 mm; and ±0.050 mm between 0.700 and 5.000 mm. Oil volume can be read to ±0.05 mL. Assuming errors in pressure and volume readings adding to give maximum deviation, the resultant error would be ±0.02 % gas content.

10.2 *Bias:*

10.2.1 A statement on the bias of this test method cannot be made because there is no material available having an accepted reference value.

10.2.2 The expected value of the gas content determined by this test method is essentially the true gas content provided that there are no leaks in the apparatus, there is no water vapor present, and an adequate water trap is used in series with the vacuum gage, and provided that the techniques for achieving maximum evolution of gas from the sample are carefully followed.

## 11. Keywords

11.1 cable oil; capacitor oil; electrical insulating oil; gas content; oil-filled cable