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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Gas Content (Nonacidic) of Insulating Liquids by Displacement with Carbon Dioxide[1]

This standard is issued under the fixed designation D 1827; 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.

ϵ^1 NOTE—Editorial changes were made throughout in February 1997.

1. Scope

1.1 This test method describes the determination of the gas content of electrical insulating liquids with a viscosity of 216 cSt or less at 100°C. Any gas that is nonreactive with a strong caustic solution may be determined.

NOTE 1—The test method has a bias for samples containing gases other than oxygen and nitrogen in atmospheric ratios due to differential solubility effects. Gases which react with KOH such as carbon dioxide will not be measured. Unsaturated hydrocarbons such as acetylene, if present, will react with KOH to a small degree and will result in an underestimation of the total gas present.

1.2 *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 831 Test Method for Gas Content of Cable and Capacitor Oils[2]
D 923 Test Method for Sampling Electrical Insulating Liquids[2]
D 1193 Specification of Reagent Water[3]
D 3613 Test Methods of Sampling Electrical Insulating Oils for Gas Analysis and Determination of Water Content[2]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *gas content by volume*—of an insulating liquid, the volume of gas contained in a given volume of liquid. It is usually expressed as a percentage at standard atmospheric conditions of 760 mm Hg pressure and 0°C temperature.

3.1.2 *gas content by weight*—the weight of gas contained in a given weight of liquid, usually expressed in parts per million.

4. Summary of Test Method

4.1 This test method consists essentially of purging dissolved gases from a small liquid test specimen with pure carbon dioxide gas. The dissolved gases are then carried into a graduated buret (precision nitrometer) filled with a potassium hydroxide solution for a precise measurement. The carbon dioxide is completely absorbed by the potassium hydroxide and the volume of other gases is measured.

5. Significance and Use

5.1 Electrical insulating liquids, in many applications, require low gas content. This is the case with capacitors and certain types of cable, for example. This test is used as a factory control test and as a control and functional test in installation and maintenance work by utilities. This test requires care in manipulation and trained, careful personnel.

6. Apparatus (see Fig. 1)

6.1 *Precision Nitrometer* (*azotometer*), *A*, with a calibrated capacity of 1.5 mL and calibrated in 0.01-mL divisions. Nitrometers shall have individual calibration correction tables that give the correct volume for each 0.01-mL point on the scale when a 40 % aqueous potassium hydroxide solution is used. The gas inlet shall consist of a 12/2 socket joint. The nitrometer shall be provided with a liquid leveling bulb, *B*, of 125-mL capacity. Rubber stoppers, *K*, with a short piece of glass capillary tubing, should be placed in the top of the nitrometer and the leveling bulb, *B*, to prevent spattering of the KOH solution.

6.2 *Permanent Magnet and Steel Wire*, The small steel wire, suitably consisting of a ⅜-in. (10-mm) length of paper clip, shall be placed inside the nitrometer for manipulation by the small external permanent magnet.

6.3 *Pregl-Type Micro Stopcock*, *C*, to allow delivery of gas to nitrometer or venting the gas to the atmosphere. This stopcock shall be provided with a 12/2 ball joint at the exit side and a 12/2 socket joint at the inlet side.

6.4 *Elbow Adapter, D*, consisting of a 12/2 ball joint and having a drawn-down tip suitable for insertion in a small hole in the rubber stopper, *E*.

6.5 *Stopper, E*, of oil-resistant rubber, cut to fit the sample-purging chamber, *F*, and provided with two small holes to

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[2] *Annual Book of ASTM Standards*, Vol 10.03.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

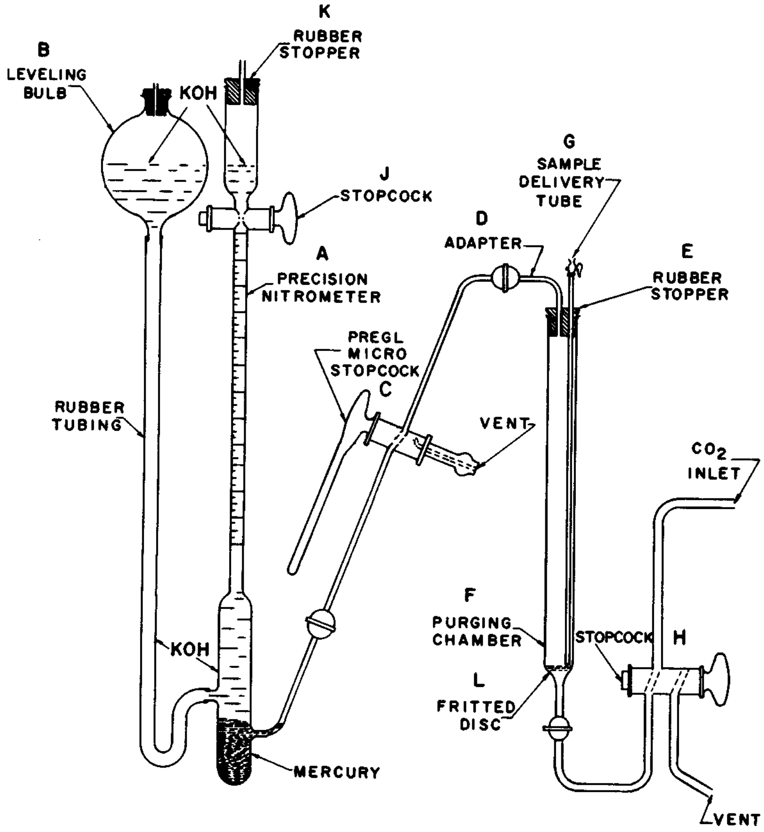

FIG. 1 Semimicro Apparatus for Determination of Gas Content of Insulating Liquids

allow tight fitting of the elbow adapter, *D*, and sample delivery tube, *G*.

6.6 *Sample-Purging Chamber, F*, with a 10-mm diameter fritted-glass disk, *L*, of medium porosity and a capacity of 15 mL above the disk for 10-mL test specimens, or with a 25-mm diameter fritted glass disk of medium porosity and a capacity of 75 mL above the disk for 50-mL test specimens. The gas inlet shall be cut straight to fit the stopper, *E*. A flexible heating tape, wrapped around the purging chamber, is necessary for holding constant temperature when test specimens are purged at temperatures in excess of room temperature.

6.7 *Sample Delivery Tube, G*, made of a length of 1⁄16-in. (1.6-mm) inside diameter stainless steel tubing, tightly inserted into one of the holes in the stopper, *E*, and extending to the fritted-glass filter. A small syringe stopcock shall be soldered to permit the delivery of an accurate volume of liquid and subsequent sealing of the purging chamber.

6.8 *Sampling Device*, consisting of a calibrated glass medical syringe fitted with a syringe stopcock for sealing of the test specimen during transfer or storage. The syringe for 10-mL test specimens shall have a calibrated capacity of 10 mL, and for 50-mL test specimens, a calibrated capacity of 50 mL. It shall be capable of accurately delivering a liquid volume within the accuracy required for the method.

6.9 *Ball-and-Socket Clamps*, size 12, for tightly securing all joints to prevent leakage.

6.10 *Two-Way Stopcock, H*, fitted to the inlet side of the purging chamber and having a 12/2 socket joint. The single-tube end of the stopcock shall be connected to the low-pressure carbon dioxide supply to control the entry of this gas to the apparatus.

6.11 *Low-Pressure Source* of very pure carbon dioxide (CO_2) gas. [4] If gas of sufficient purity is not available, one of the following mechanisms utilizing solid CO_2 shall be used:

6.11.1 A high-pressure cylinder having an opening large enough to insert pieces of solid CO_2 and capable of withstanding 800 to 1000 psi (5.5 to 7 MPa) pressure when the solid evaporates. The cylinder should be provided with a pressure-reduction valve capable of delivering gas at a gage pressure as low as 3 to 5 psi (20 to 34 kPa). A pressure-relief valve set at about 1200 psi (8 MPa) should be included, and care should be exercised to limit the quantity of solid CO_2 placed in the cylinder due to the greater volume occupied by the gas.

6.11.2 A vacuum bottle that can be charged with solid CO_2. The bottle may be closed with a large rubber stopper fitted with a pressure-bleed regulator, a mercury relief valve, a nichrome heating element, and a gas delivery tube to the apparatus.

6.11.3 A 12-qt (12-L) aluminum pressure cooker that can be charged with solid CO_2. It should be fitted with a suitable pressure-relief device and gas delivery tube. A minimum length of heavy-wall rubber or plastic tubing should be used in connecting the source of CO_2 gas to the purging chamber.

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[4] Cylinders of 99.99 % minimum purity liquified carbon dioxide fitted with two stage regulator with stainless steel diaphragms have been found satisfactory. Cylinders of “Instrument Grade” carbon dioxide and regulators (Model No. 18-5, CGA 320) may be obtained from Airco Rare and Specialty Gases, 575 Mountain A, Murray Hill, NJ 07974.

6.12 All glass shall be heat- and chemical-resistant. Capillary tubing should be used wherever possible to minimize the amount of purging necessary to remove air from the apparatus.

6.13 An auxiliary source of vacuum, with trap, to facilitate removal of spent test specimen, is optional.

7. Reagents and Materials

7.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III of Specification D 1193.

7.3 *Carbon Dioxide* (CO_2)—Solid carbon dioxide (dry ice) for producing CO_2 of high purity.

7.4 *Potassium Hydroxide Solution* (40 %)—Dissolve 572 g of potassium hydroxide (KOH) in water and dilute to 1 L.

7.5 *Stopcock Grease* that is resistant to KOH and the oil sample.

8. Sampling

8.1 When convenient, connect the sample delivery tube of the measuring apparatus directly to the container from which the liquid is to be sampled. This is usually not convenient and is often impossible. The methods described in 8.2 to 8.5 are recommended as alternatives.

8.2 For sampling liquid at or below the gas saturation point, a syringe and stopcock may be used in accordance with Test Method D 3613.

8.3 For liquids that are sampled under pressure or that are supersaturated with a gas, follow the same procedure as described in 8.2, except that after sealing off the sample a pressure device is required to maintain a solution of the gas in the liquid. Any spring mechanism that can be built around the syringe is suitable, and the tension required will depend upon the pressure required to maintain solution.

8.4 Use 10 mL test specimens, except where it is desirable to increase the precision of the test. On samples having a gas content of 1 % or less, 25 to 50-mL test specimens are desirable.

8.5 The sample container and method for sampling liquids for the determination of gas content in Test Methods D 831 and D 3613 have been found satisfactory and convenient for obtaining samples in the field. This sample container has been found satisfactory for storing oil samples with a low gas content for a week or more. Transfer the test specimen to the sample delivery tube of the measuring apparatus with a syringe.

9. Preparation of Apparatus

9.1 Fill the bottom portion of the nitrometer, *A*, with mercury to a point about 5 to 7 mm above the gas entry capillary. The mercury acts as a valve to allow the gas to bubble into the buret and also to prevent the KOH solution from flowing into the system due to gravity. Place a ⅜-in. (10-mm) piece of steel wire on the mercury.

9.2 Open stopcock *J* and add KOH solution to the leveling bulb, *B*, until there are 20 or 30 mm of KOH solution above the stopcock, *J*, with the solution in the leveling bulb at the same level. Remove all bubbles from the nitrometer, using the steel wire and magnet.

9.3 The system must be kept completely leak-free, since even small leaks allow diffusion of air into the apparatus and cause indeterminate errors. Leakage can easily be detected in the purging operation. If leakage occurs, check all joints and connections. Carbon dioxide of insufficient purity has the same effect as leaks.

10. Procedure

10.1 Close stopcock *J*, adjust stopcock *C* to allow gas to flow from the purging chamber to the atmosphere, and turn stopcock *H* to the vent position. Connect the syringe to the sample delivery tube, *G*, flush with 1 to 2 mL of the sample, and close the syringe or delivery tube stopcock. Reverse stopcock *H* slowly, and flush the purging chamber with CO_2 for 2 to 5 min. Divert the gas into the nitrometer with stopcock *C*, and allow it to flow for 10 to 15 s. If gas bubbles continue to rise to the top of the nitrometer, again flush the purging chamber. After completion of the purging, gas bubbles should continue to form just above the mercury in the nitrometer and rapidly decrease to micro size near the top of the KOH solution, but there should not be sufficient unabsorbed gas to lower the level of the solution. If this condition does not exist, either repeat purging or replace the spent solution until no change is noted.

10.2 Make certain that the nitrometer is completely filled with KOH solution, that there are 20 to 30 mm of solution above stopcock *J*, and that the stopcock *J* is closed; then lower the leveling bulb to about the center of the calibrated portion of the nitrometer. Close stopcock *C*, vent *H*, open the stopcocks between the syringe and purging chamber *F*, and introduce a measured test specimen of liquid. Ten-millilitre test specimens have been found satisfactory for oils with a gas content of 1 % or more. For liquids with a gas content of less than 1 %, the use of 50-mL samples will increase the precision by a factor of 5. The introduction of test specimens of the more viscous liquids may be accelerated by preheating the sampling device to some known temperature from 40 to 100°C. Record the temperature and volume or weight of the liquid test specimen.

10.3 Close stopcock *G*, then open stopcocks *C* and *H* to allow CO_2 to bubble through the sample into the nitrometer with 1 to 5 bubbles a second in the nitrometer. Initially, the CO_2 may rapidly dissolve in the liquid and no bubbles will appear in the nitrometer, but in time the gas bubbles will begin to appear. Purging the test specimens requires about 10 min, or more for higher viscosity liquids, and the end point is reached when the dissolved gases are completely removed and only

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[5] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

micro bubbles appear at the top of the nitrometer. The purging of the higher viscosity liquids may be accelerated by maintaining the purging chamber at some temperature between 40 and 100°C. This temperature need not be accurately controlled. When the volume of gas in the nitrometer shows no further change, vent stopcocks *C* and *H* to the atmosphere. Make certain that all of the gas is above the KOH solution in the nitrometer, using the magnet and steel wire if necessary.

10.4 After allowing sufficient time for drainage of the nitrometer, hold the liquid in the leveling bulb at the level of the liquid in the nitrometer column, and read and record the volume of gas in the nitrometer to the nearest 0.005 mL.

10.5 Run a blank test immediately after completing the sample analysis, for an equivalent period of time. This blank measurement of gas volume constitutes a correction for CO_2 impurities, which will be subtracted from the primary reading noted in 10.4.

10.6 Record the ambient temperature in degrees Celsius and the barometric pressure in millimetres of mercury.

10.7 With stopcock *C* vented to the atmosphere, remove the test specimen from the purging chamber, *F*, either with a syringe or the auxiliary vacuum trap. Stopcock *H* should be closed during this procedure.

11. Calculation

11.1 Express the gas content of the liquid at standard temperature and pressure. The value can be expressed either on a volume percent or parts per million basis.

11.1.1 Calculate the volume of gas, V_s, in millilitres corrected to 760 mm and 0°C, as follows:

$$V_s = [(V_a - V_c) \times (P_a - 0.65\,P_w) \times T_s]/(P_s \times T_a)$$

where:

V_a = actual volume of gas collected during test specimen run, to the nearest 0.005 mL,
V_c = volume of gas collected during blank run, to the nearest 0.005 mL,
P_a = atmospheric pressure at time of analysis, mm of mercury,
P_w = Vapor pressure of pure water at the temperature at which V_a is measured,
T_s = standard reference temperature 273 K, unless otherwise specified,
P_s = standard reference pressure, mm of mercury (760 mm), and
T_a = temperature, at which V_a was measured, K (273 + °C).

NOTE 2—The vapor pressure of water in a 40 % potassium hydroxide solution is about 65 % of that of pure water. Therefore, when the correction to total pressure for the vapor pressure of the potassium hydroxide solution is made, the vapor pressure of the water is multiplied by 0.65.

11.1.2 Calculate the liquid volume at standard temperature (Kelvin), V_{ls} as follows:

$$V_{ls} = V_l - V_l\,(T_a - T_s)\infty$$

where:

V_l = volume of liquid tested (Note 3),
T_s = standard reference temperature, K, (273 K unless otherwise specified)
α = coefficient of expansion, per degree Kelvin, of liquid under test. A typical value of 0.007/K can be used for mineral oil.

NOTE 3—Tables of vapor pressure can be found in the CRC Handbook.[6]

11.1.3 Calculate the volume percent gas content under standard conditions as follows:

$$\text{Gas content, volume \%} = (V_s/V_{ls}) \times 100$$

11.1.4 Calculate the gas content on a parts per million by weight basis as follows:

$$\text{Gas content, ppm by weight} = [(V_s \times D_g)/W_l] \times 10^6$$

where:

V_s = volume of gas defined in 11.1.1,
D_g = density of the gas at standard conditions, T_s, P_s, (air = 1.293×10^{-3} g/mL at 0 C, 760 mm).
W_l = weight of liquid.

NOTE 4—The density of gases at other temperatures may be found in the CRC Handbook.[6]

12. Report

12.1 Report the following:

12.1.1 The volume or weight of the test specimen tested,

12.1.2 The temperature of the test specimen during purging, and

12.1.3 Gas content of the test specimen, in volume percent, or parts per million by weight.

13. Precision and Bias

13.1 *Precision*:

13.1.1 The repeatability of this test method has not been determined.

13.1.2 The reproducibility of the test method is ±0.05 of the gas content by volume for a sample containing 0.10 to 15.0 % gas by volume, if care is used in delivery of the sample, and if the same sample is compared by different, experienced operators.

13.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in Test Method D 1827, no statement on bias can be made.

14. Keywords

14.1 carbon dioxide; dissolved gases; gas content; insulating liquids

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[6] *CRC Handbook of Chemistry and Physics* is available from CRC Press, Cleveland, Ohio.