



# Standard Test Method for Oxidation Stability of Mineral Insulating Oil <sup>1</sup>

This standard is issued under the fixed designation D 2440; 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.

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

## 1. Scope

1.1 This test method determines the resistance of mineral transformer oils to oxidation under prescribed accelerated aging conditions. Oxidation stability is measured by the propensity of oils to form sludge and acid products during oxidation. This test method is applicable to new oils, both uninhibited and inhibited, but is not well defined for used or reclaimed oils.

NOTE 1—A shorter duration oxidation test for evaluation of inhibited oils is available in Test Method D 2112.

NOTE 2—For those interested in the measurement of volatile acidity, reference is made to the work of IEC Subcommittee 10A. <sup>2</sup>

1.2 The values stated in metric units are to be regarded as 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:

- B 1 Specification for Hard-Drawn Copper Wire<sup>3</sup>
- D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration<sup>4</sup>
- D 974 Test Method for Acid and Base Number by ColorIndicator Titration<sup>4</sup>
- D 2112 Test Method for Oxidation Stability of Inhibited Mineral Insulating Oil by Rotating Bomb<sup>5</sup>
- D 2272 Test Method for Oxidation Stability of Steam Turbine Oils by Rotating Pressure Vessel<sup>4</sup>
- D 3487 Specification for Mineral Insulating Oil Used in Electrical Apparatus<sup>5</sup>

### 2.2 IEC Publication:

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.06 on Chemical Test.

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<sup>2</sup> ASTM Research Report No. RR: D-27-1001, available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 02.03.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 05.01.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 10.03.

CEI/IFC 61125: 1992 Unused Hydrocarbon-Based Insulating Liquids—Test Methods for Evaluating the Oxidation Stability<sup>6</sup>

## 3. Summary of Test Method

3.1 A test specimen of mineral transformer oil is oxidized at a bath temperature of 110°C, in the presence of a copper catalyst coil, by bubbling oxygen through duplicate test specimens for 72 and 164 h, respectively. The oil is evaluated at the end of each aging period by measuring the amount of sludge and acid formed. The test specimen is diluted with *n*-heptane and the solution filtered to remove the sludge. The sludge is dried and weighed. The sludge-free solution is titrated at room temperature with standard alcoholic base to the end point indicated by the color change (green-brown) of the added *p*-naphthol-benzene solution.

## 4. Significance and Use

4.1 The oxidation stability test of mineral transformer oils is a method for assessing the amount of sludge and acid products formed in a transformer oil when the oil is tested under prescribed conditions. Good oxidation stability is necessary in order to maximize the service life of the oil by minimizing the formation of sludge and acid. Oils that meet the requirements specified for this test in Specification D 3487 tend to minimize electrical conduction, ensure acceptable heat transfer, and preserve system life. There is no proven correlation between performance in this test and performance in service, since the test does not model the whole insulation system (oil, paper, enamel, wire). However, the test can be used as a control test for evaluating oxidation inhibitors and to check the consistency of oxidation stability of production oils.

## 5. Apparatus

5.1 *Aging (Oxidation) Bath*—An oil bath, wax bath, or aluminum block heater (see IEC Standard 61125) of a suitable type capable of controlling the temperature at  $110 \pm 0.5^\circ\text{C}$  with a temperature gradient of less than 1°C in the body of the liquid. Use any nontoxic liquid having low volatility at 110°C and containing no volatile additives and having a flash point above the test temperature. Mineral oils, waxes and silicone

<sup>6</sup> International Electrotechnical Commission. Available from American National Standards Institute, 1430 Broadway, New York, NY 10018.

oils have been used satisfactorily. Circulation of the oil or wax heating medium by means of a pump or stirrer is recommended. See also 5.4, relating to the depth of fluid in aging bath and position of oil receptacles.

5.2 *Drying Tower*—A drying tower 25 to 30 cm in height, fitted with a ground-glass stopper and side tubes for conditioning of the oxygen supply.

5.3 *Oil Receptacle and Head*—An oil receptacle consisting of a heat-resistant glass test tube 25 mm in outside diameter, 1.25 mm in wall thickness, 210 mm in overall length including a standard taper 24/40 outer joint, with a Dreschsel-type head consisting of a standard taper 24/40 inner joint with side outlet tube 5.0 mm in outside diameter and an oxygen delivery tube 5.0 mm in outside diameter and 3 mm, minimum, in inside diameter which extends to within  $2.5 \pm 0.5$  mm of the bottom of the oil receptacle and has its end ground at an angle of  $30^\circ$  to the axis of the tube. The design is shown in Fig. 1.

5.4 Position the oil receptacle in the aging bath in accordance with the drawing shown in Fig. 2.

NOTE 3—The oil receptacle and position in the aging bath are essentially the same as specified in IEC Standard 61125, "Test Method for Oxidation Stability of Inhibited Mineral Insulating Oils," Glassware dimensions have been altered slightly to conform to sizes readily available in the United States.

5.5 *Filtering Crucibles*—A glass filter crucible having a maximum diameter of the pores between 5 and 15  $\mu\text{m}$  as determined in accordance with the method described in the Appendix. Cellulose ester type membrane filters of 5- $\mu\text{m}$  porosity have been used successfully also.

## 6. Reagents and Materials

6.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, all reagents are to conform to the specifications of the Committee on Analytical Reagents of

the American Chemical Society, where such specifications are available.<sup>7</sup>

6.2 *Chloroform*, cp, or *acetone*, cp.

6.3 *n-Heptane*,<sup>8</sup> for dilution of the oxidized oil, precipitation, and washing of the sludge shall conform to the following requirements:

Relative Density at 20°C	0.6836 to 0.6839
Refractive index at 20°C	1.3876 to 1.3879
Solidification temperature, min, °C	-90.72
Distillation	50 % shall distill between 98.38 and 98.48°C. Temperature rise between 20 and 80 % recovery shall be 0.20°C maximum

6.4 *p-Naphtholbenzein Indicator Solution*—The specifications for *p*-naphtholbenzein are prescribed in Test Method D 974.<sup>9</sup> Prepare a solution containing 10 g *p*-naphtholbenzein per litre of titration solvent (see 6.7).

6.5 *Oxygen*—Obtained from liquid oxygen, minimum purity 99.4 %.

6.6 *Potassium Hydroxide Solution, Standard Alcoholic (0.1 N)*—Prepare alcoholic potassium hydroxide solution and standardize as described in Test Method D 974. Commercially prepared Alcoholic Potassium Hydroxide Solution is available.

6.7 *Titration Solvent*—Mix 3 parts by volume of toluene with 2 parts by volume of isopropyl alcohol.

6.8 *Silicon Carbide Abrasive Cloth*, 100-grit with cloth backing.

6.9 *Wire Catalyst*—AWG No. 18 (0.0403-in. (1.01-mm) diameter) 99.9 % purity conforming to Specification B 1. Soft-drawn copper wire of equivalent grade may also be used.

6.10 *Hydrochloric Acid*, 10 Volume %.

## 7. Hazards

7.1 Consult Material Safety Data Sheets for all materials used in this test method.

## 8. Preparation of Oxygen Supply

8.1 Oxidize the oil by contact with oxygen of 99.4 % minimum purity conditioned as follows:

8.1.1 Use metal or glass tubing to deliver the oxygen to the oxygen delivery tube. Eliminate rubber connections if possible. If used, the rubber should be at room temperature. Limit the length of rubber tubing exposed to the oxygen flow to 13 mm. Do not use rubber connections in contact with the liquid of the heating bath or under conditions where the temperature of the rubber will be above room temperature. Where it is necessary to use rubber connections, only sulfur-free rubber is permissible. FOA/USP Nalgene tubing has been found satisfactory in this application.

8.1.2 Dry the oxygen by forcing it through a solid desiccant

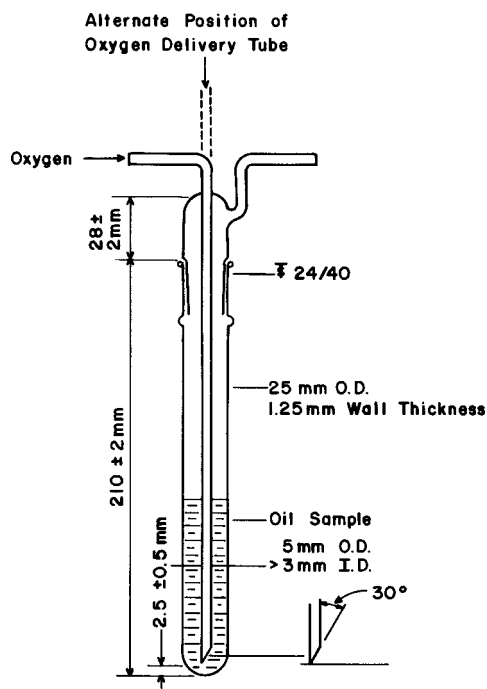


FIG. 1 Oil Receptacle and Head

<sup>7</sup> "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents 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."

<sup>8</sup> A suitable material is ASTM *n*-Heptane Knock Test Reference Fuel, marketed by the Phillips Petroleum Co., Bartlesville, OK.

<sup>9</sup> Solid *p*-naphtholbenzein conforming to the specifications indicated is available from Distillation Products Industries, Eastman Organic Chemicals Dept., Rochester 3, NY.

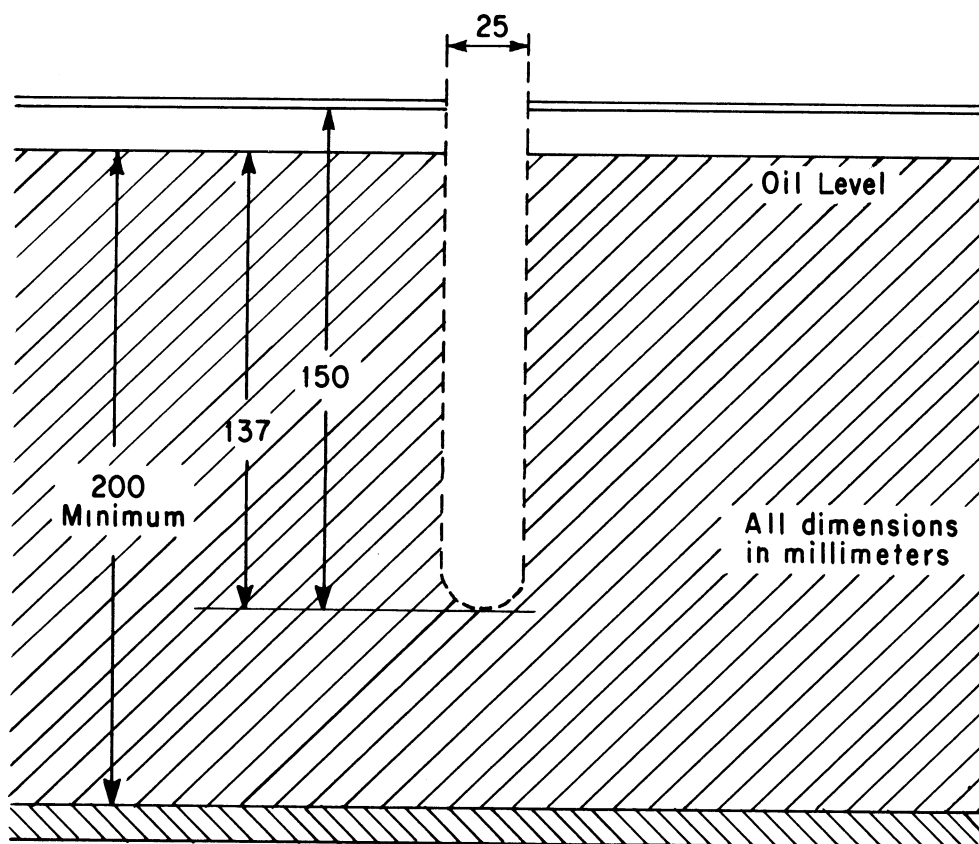


FIG. 2 Position of Oil Receptacle in Aging Bath

of high moisture-absorbing capacity.<sup>10</sup> Arrange the desiccant in the drying tower to a depth of 205 to 254 mm. Change the desiccant when the indicator begins to change colors from moisture absorption. If an indicator is not used, change the desiccant at least weekly.

8.1.3 After passing oxygen through the drying tower, admit it directly to the receptacle containing the oil to be tested. Do not preheat the oxygen.

8.1.4 Determine the rate of oxygen supply with an electronic flow meter, calibrated rotameter, or soap bubble buret, and adjust the flow rate to deliver the conditioned oxygen at a rate of  $1.0 \pm 0.1$  L/h to each tube of oil being tested.

## 9. Preparation of Oil Receptacle

9.1 Wash each oil receptacle thoroughly, first with acetone and then with soap and water, and rinse in acid solution. The following reagents have been found suitable: chromic acid, aqua regia, and ammonium persulfate.<sup>11</sup> Wash each receptacle free of acid, using tap water, and finally rinse with distilled water. Dry in an oven at 105 to 110°C for at least 3 h, cool to room temperature in a desiccator, and keep the receptacle in the desiccator until ready for use.

## 10. Preparation of Copper Catalyst

10.1 Immediately before use, polish the copper wire with

silicon carbide abrasive cloth and wipe free of abrasive with a clean dry cloth.

10.2 Wind a 300-mm length of the polished wire into a helical coil approximately 16 mm in outside diameter and 50 mm in height. Clean the coil thoroughly with chloroform or acetone, air dry, and insert immediately into the oil receptacle. Handle the clean copper coil only with clean tongs to avoid contamination.

10.3 Commercially available, prepackaged, preformed coils that meet the requirements described in this test method may be used as an alternative method of catalyst preparation. Clean the coil in a 10% HCl solution for 30 s, rinse three times with tap water and with distilled water, chloroform or acetone and air dry immediately before use. Preformed coils were used in a 1997 Round Robin Study and found suitable for this application.<sup>12</sup>

## 11. Preparation of Glass Filtering Crucible

11.1 Clean the glass filtering crucible and dry in an air oven at 105 to 110°C until it has reached constant mass. Cool and store in a desiccator and, when needed, weigh to the nearest 0.0001 g.

## 12. Conditioning of Test Specimen

12.1 Filter the oil test specimen by gravity at ambient pressure or at reduced pressure, through acid-free filter paper to

<sup>10</sup> Anhydrous magnesium perchlorate (Anhydron or Dehydrite) is a suitable desiccant for this purpose.

<sup>11</sup> Ammonium Persulfate is available as Nochromix from Interex Corporation, 3 Strathmore Road, Natick, MA 01760.

<sup>12</sup> A research report is available from ASTM headquarters. Request RR: D-27-1011.

remove traces of sediment, fiber, and excess water. Alternatively, filter the test specimen under vacuum through a 0.45 µm mixed ester or cellulose filter. Discard the first 25 mL of the oil filtrate. Carefully protect the filtrate during the filtration against dust and other contaminations.

### 13. Procedure

13.1 Prepare two copper catalysts as described in Section 10. Insert one in each of two clean, dry oil receptacles prepared as described in Section 9.

13.2 Transfer  $25 \pm 0.01$  g of the conditioned oil test specimen into each of the two prepared oil receptacles by means of a clean dry pipet. Immediately place the head on the oil receptacle to protect the oil from contamination during the interval between placing the oil in the receptacle and the actual start of the oxidation.

13.3 Adjust the heating bath to maintain a temperature of  $110 \pm 0.5^\circ\text{C}$  during the oxidation of the sample.

### 14. Oxidation

14.1 Immerse the oil receptacle in the heating bath, which has been adjusted to the test temperature. Immerse the oil receptacle in the bath to a depth such that the surface of the liquid in the heating bath will be 3 to 5 cm higher than the surface of the test oil in the receptacle, after the test oil reaches test temperature. Attach the oxygen inlet tubes to the oxygen supply line. When more than one oil receptacle is fed from a single oxygen supply line, adjust the oxygen flow to each oil receptacle to deliver  $1 \pm 0.1$  L/h.

14.2 Oxidize the two oil test specimens for 72 and 164 h respectively, under carefully controlled conditions.

### 15. Treatment of Oxidized Oil

15.1 *Sludge Formation*—After the required period of oxidation, shut off the oxygen supply and remove the oil receptacle and test specimen from the oxidizing bath. Cool for 1 h to room temperature in a clean dark box or dark room, protecting the test specimen from dust, moisture, or other contamination and light. At the end of the cooling period, remove the oxygen delivery tube and the copper catalyst coil from the oil test receptacle and transfer the oxidized oil into a 500-mL Erlenmeyer flask fitted with a ground glass stopper. Separate 300 mL of *n*-Heptane into three equal aliquots to be used for the sequential washing of the oxygen delivery tube copper catalyst coil and the test receptacle to recover the adhering oil. Into the same flask, thoroughly rinse the catalyst coil, delivery tube, and test receptacle to recover adhering oil, using a total of 300 mL of *n*-Heptane.

15.2 *Weighing Sludge*—Allow the mixture to stand in the dark for 24 h, at a temperature of  $20 \pm 2^\circ\text{C}$ , before filtering through a glass filter or polymer membrane, previously dried to constant weight. To prevent sludge from passing through the filter, use a small pressure drop at the start of filtering. Pass cloudy filtrates through a second time. Remove all traces of oil from the sludge by repeated washing with *n*-Heptane. Wash the 500-mL Erlenmeyer flask with *n*-heptane to remove adherent oil, and empty each rinse into the filter. Use a total volume of 150 mL of *n*-heptane, divided into three aliquots for sequential washes, for washing the sludge. Dry the filter containing the

sludge at  $110^\circ\text{C}$  to constant mass and weigh to the nearest 0.0001 g. Transfer sludge adhering to the catalyst, test receptacle, oxygen delivery tube, and the 500-mL Erlenmeyer flask by dissolving the sludge in small quantities of chloroform or acetone (a total of 30 mL) into a tared porcelain vessel. Dry at  $110^\circ\text{C}$  after the evaporation of the chloroform or acetone to constant mass. Add the weight of the residue to that of the sludge obtained by precipitation with *n*-heptane. Express the total sludge as a percentage of the initial weight of the oil as follows:

$$\text{Sludge, \%} = (\text{weight of sludge/weight of oil sample}) \times 100$$

15.3 *Neutralization Value*—Collect the *n*-heptane solution in the 500-mL volumetric flask after filtering the sludge. Rinse the vacuum flask three times with *n*-heptane, emptying the contents into the volumetric flask, and dilute to the mark. Make three determinations of the neutralization value on 100-mL aliquots of the *n*-heptane solution containing test specimens. Add 0.5 mL of *p*-naphtholbenzein indicator solution to 100 mL of titration solvent. Add this indicator solution (100 mL) while stirring to 100 mL of the heptane solution. Titrate this mixture with 0.1 *N* alcoholic potassium hydroxide solution at a temperature not exceeding  $25^\circ\text{C}$ . The orange color of the indicator changes to a green or green-brown as the end point is approached. Consider the end point definite if the color change persists for 15 s or if it reverses with 2 drops of 0.1 *N* HCl. A buret having a capacity of 10 mL and with calibrations of 0.01 mL is recommended for the purpose of accurately controlling the flow of titration solvent. Hypodermic needle tips have been used to provide finer control of the flow rate.

15.3.1 On dark colored oils, it is permissible to determine the neutralization value with Test Method D 664. The precision and bias statements for this test method do not apply, however, when Test Method D 664 is used. Moreover, the relationship between the values obtained with the colorimetric and potentiometric methods has not been proven. Values obtained by the potentiometric titration method cannot be used to meet the Test Method D 2440 limits of Specification D 3487.

15.4 *Calculation*—Calculate the neutralization value, in milligrams of KOH per gram of oil, as follows:

$$\text{Neutralization value} = (N_2 - N_1) 5.61/5$$

where:

$N_2$  = millilitres of 0.1 *N* alcoholic potassium hydroxide solution necessary to neutralize the *n*-heptane solution,

$N_1$  = millilitres of 0.1 *N* alcoholic potassium hydroxide solution necessary to neutralize 100 mL of *n*-heptane to which have been added 100 mL of the titrating solution,

5.61 = milligrams of KOH per millilitre of 0.1 *N* solution, and

5 = grams of oil in 100 mL of heptane.

### 16. Report

16.1 Report the neutralization value as the arithmetic average of the three determinations of soluble activity.

16.2 Report the sludge as a percentage of the initial weight of the oil.

**17. Precision and Bias**

17.1 Use the following criteria for judging precision of test results on new, used, and reclaimed oils at the 95 % probability level.

NOTE 4—The precision statements for this test method were based on two test specimens of each new, used, and reclaimed oils. Six laboratories participated. No research report is available.

17.1.1 *Repeatability*—The difference between results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only one case in twenty.

Sludge, wt %	0.12 $\sqrt{X}$
Neutralization value, mgKOH/g	0.22 $\sqrt{X}$

where X is the average of two determinations on the same test sample.

17.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only one case in twenty.

Sludge, wt %	0.23 $\sqrt{X}$
Neutralization value, mgKOH/g	0.36 $\sqrt{X}$

where X is the average of the results submitted by each of two laboratories on the average of two determinations on the same test sample.

17.1.3 Note that the neutralization value for a single test specimen is itself the average of three concurrent titrations on a single oxidized test specimen. Do not consider this average suspect unless the range of the three titrations (largest value minus smallest value) exceeds 0.08 mgKOH/g.

17.2 *Bias*—No justifiable statement can be made on the bias of the procedure in this test method, since there is no accepted material suitable for reference in determining oxidation stability.

**18. Keywords**

18.1 acid; aging; electrical oil; insulating mineral oil; oxidation; sludge; stability; transformer

**APPENDIX**

(Nonmandatory Information)

**X1. DETERMINATION OF MAXIMUM DIAMETER OF THE PORES OF A GLASS FILTER**

**X1.1 Summary of Method**

X1.1.1 The maximum diameter of the pores is determined by measuring the air pressure necessary to cause the first air bubble to pass through the filter moistened by the liquid to be tested.

**X1.2 Significance and Use**

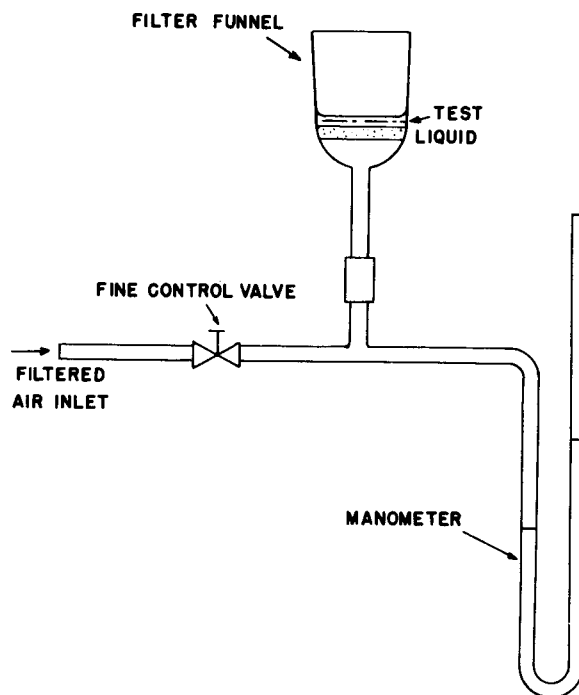
X1.2.1 It is not easy to determine the mean diameter of the pores of a glass filter. The determination of the maximum diameter of the pores provides a valuable indication of the retaining capacity of the filter, that is, the maximum diameter of a spherical particle that can pass through it.

**X1.3 Procedure**

X1.3.1 Use water as a test liquid in order to check a filter having a maximum pore diameter of 5 to 15  $\mu\text{m}$ .

X1.3.2 Completely immerse the washed filter in the test liquid, allowing a film of several millimetres thickness to remain on the upper surface of the filter attached to the apparatus (Fig. X1.1).

X1.3.3 Apply a uniformly increasing pressure to the filter until 1 or 2 bubbles of air appear in the liquid above the filter. The manometer reading enables the maximum diameter of the pores to be calculated. This manometer can be directly calibrated in pore diameters.



**FIG. X1.1 Apparatus for Determination of Maximum Diameter of the Pores of a Glass Filter**

**X1.4 Calculation**

X1.4.1 Calculate the maximum pore diameter as follows:

$$D = 30 Y/P$$

where:

$D$  = diameter of the pore,  $\mu\text{m}$ ,

$Y$  = surface tension of the test liquid, dynes/cm, and

$P$  = observed pressure, mm Hg.

X1.4.2 For a filter whose maximum pore diameter is between 5 and 15  $\mu\text{m}$ , the manometer reading in millimetres of mercury will be 435 to 145 for water.

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