



Standard Test Method for High Temperature Universal Oxidation Test for Turbine Oils¹

This standard is issued under the fixed designation D 6514; 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 a procedure for evaluating the oxidation of inhibited lubricants in the presence of air, copper, and iron metals.

1.2 This test method was developed and is used to evaluate the high temperature oxidation stability and deposit forming tendency of oils for steam and gas turbines. It has been used for testing other lubricants made with mineral oil and synthetic basestocks for compressors, hydraulic pumps, and other applications, but these have not been used in cooperative testing.

1.3 The values stated in SI units are to be regarded as the standard.

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.* Identified hazardous chemicals are listed in Section 7. Before using this test method, refer to suppliers' safety labels, Material Safety Data Sheets and other technical literature.

2. Referenced Documents

2.1 ASTM Standards:

- A 510 Specification for General Requirements for Wire Rods and Coarse Round Wire, Carbon Steel²
- B 1 Specification for Hard-Drawn Copper Wire³
- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids and the Calculation of Dynamic Viscosity⁴
- D 664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration⁴
- D 943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils⁴
- D 974 Test Method for Acid and Base Number by Color-Indicator Titration⁴

D 3339 Test Method for Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration⁵

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁵

D 4871 Guide for Universal Oxidation/Thermal Stability Test Apparatus⁵

D 5846 Test Method for Universal Oxidation Test for Hydraulic and Turbine Oils Using the Universal Oxidation Test Apparatus⁶

2.2 British Standards:⁷

BS 1829 Specification for Carbon Steel, alternate to Specification A 510

2.3 Institute of Petroleum Standard:⁸

IP 2546 Practice for Sampling of Petroleum Products; Alternate to Practice D 4057

2.4 ASTM Adjunct⁹

ADJD6514 Oxidation Cell Varnish Standard⁹

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *Universal Oxidation Test*—the apparatus and procedures described in Guide D 4871.

4. Summary of Test Method

4.1 After determining the viscosity at 40°C and acid number of a sample, a test specimen is stressed at 155°C for 96 h. After cooling, the test specimen is vacuum filtered for the determination of the total insolubles formed during the test. Total insolubles are reported as low, medium, or high.

4.2 The viscosity and the acid number of the filtrate are determined and compared with the initial values to ascertain any increase in those values. Both the change in acid number and the increase in viscosity at 40°C are reported.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of D02.09 on Oxidation.

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² *Annual Book of ASTM Standards*, Vol 01.03.

³ *Annual Book of ASTM Standards*, Vol 02.03.

⁴ *Annual Book of ASTM Standards*, Vol 05.01.

⁵ *Annual Book of ASTM Standards*, Vol 05.02.

⁶ *Annual Book of ASTM Standards*, Vol 05.03.

⁷ Available from British Standards Institute (BSI), 389 Chiswick High Rd., London W4 4AL, U.K.

⁸ Available from Institute of Petroleum (IP), 61 New Cavendish St., London, WIG 7AR, U.K.

⁹ Available from ASTM International Headquarters. Order Adjunct No. ADJD6514. Names of suppliers in the United Kingdom can be obtained from the Institute of Petroleum. Two master standards are held by the IP for reference.

4.3 The glass cell in which the test specimen was stressed is rinsed with heptane and dried. Residual deposits are compared with ASTM Adjunct ADJD6514, and the results are reported.

5. Significance and Use

5.1 Degradation of fluid lubricants because of oxidation or thermal breakdown can result in fluid thickening or in the formation of acids or insoluble solids and render the fluid unfit for further use as a lubricant.

5.2 This test method can be used for estimating the oxidation stability of oils. It can function as a formulation screening tool, specification requirement, quality control measurement, or as a means of estimating remaining service life. It shall be recognized, however, that correlation between results of this test method and the oxidation stability of an oil in field service can vary markedly with field service conditions and with various oils.

5.3 This test method is designed to compliment Test Method D 5846 and is intended for evaluation of fluids which do not degrade significantly within a reasonable period of time at 135°C.

6. Apparatus

6.1 *Heating Block*, as shown in Fig. 1, and as further described in Guide D 4871, to provide a controlled constant temperature for conducting the test.

6.1.1 Test cells are maintained at constant elevated temperature by means of a heated aluminum block which surrounds each test cell. Alternate apparatus designs for sample heating and for temperature and flow control shall be acceptable, provided they are shown to maintain temperature and gas flow within the standard's specified limits.

6.1.2 Holes in the aluminum block to accommodate the test cells shall provide 1.0 mm maximum clearance for 38-mm



FIG. 1 Heating Block

outside diameter glass tubes. The test cells shall fit into the block to a depth of 225 ± 5 mm.

6.2 *Temperature Control System*, as shown at lower left in Fig. 1, and as further described in Guide D 4871, to maintain the test oils in the heating block at $155 \pm 0.5^\circ\text{C}$ for the duration of the test.

6.3 *Gas Flow Control System*, as shown at the upper left in Fig. 1, and as further described in Guide D 4871, to provide dry air at a flow rate of 3.0 ± 0.5 L/h to each test cell.

6.3.1 A gas flow controller is required for each test cell.

6.3.2 Flowmeters shall have a scale length sufficiently long to permit accurate reading and control to within 5 % of full scale.

6.3.3 The total system accuracy shall meet or exceed the following tolerances:

6.3.3.1 Inlet pressure regulator within 0.34 kPa (0.05 psig) of setpoint; total flow control system reproducibility within 7 % of full scale; repeatability of measurement within 0.5 % of full scale.

6.4 *Oxidation Cell*, borosilicate glass, as shown in Fig. 2, and as further described in Guide D 4871. This consists of a test cell of borosilicate glass, standard wall, 38 mm outside diameter, 300 ± 5 -mm length, with open end fitted with a 34/45 standard-taper, ground-glass outer joint.

6.5 *Gas Inlet Tube*, as shown in Fig. 2, and as further described in Guide D 4871. This consists of an 8-mm outside diameter glass tube, 455 ± 5 mm long, lower end with fused capillary 1.5-mm inside diameter. The capillary bore shall be 15 ± 1 mm long. The lower tip is cut at a 45° angle.

6.6 *Basic Head*, as shown in Fig. 3, and as further described in Guide D 4871. This is an air condenser, with 34/45 standard-taper, ground-glass inner joint, opening for gas inlet tube, septum port for sample withdrawal, and exit tube to conduct off-gases and entrained vapors. Overall length shall be 125 ± 5 mm.

6.7 Test precision was developed using the universal oxidation/thermal stability test apparatus described in Guide

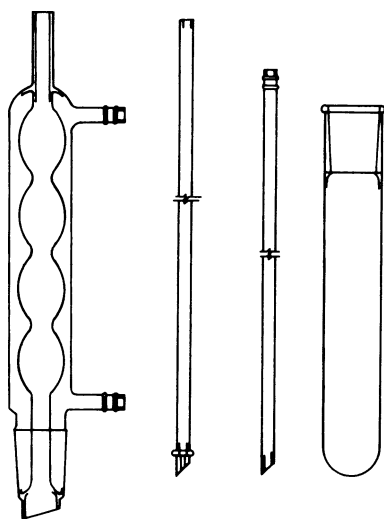


FIG. 2 Oxidation Cell

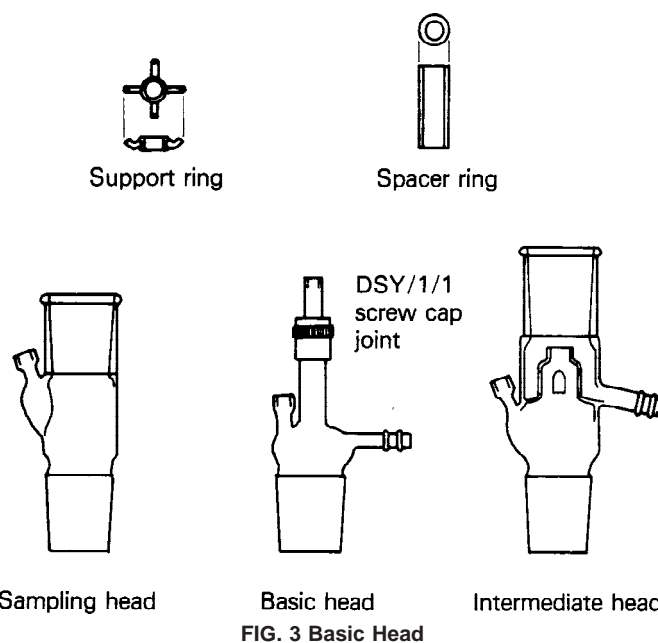


FIG. 3 Basic Head

D 4871.^{10,11} Alternate apparatus designs for sample heating, and for temperature and flow control shall be acceptable provided they are shown to maintain temperature and gas flow within the specified limits.

6.8 *Drying Oven*, explosion proof model recommended.

7. Reagents and Materials

7.1 Reagent grade chemicals shall be used 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.¹² 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 *Catalyst Coil (comprised of the following)*:^{11,13}

7.2.1 *Low-Metalloid Steel Wire*, 1.59 mm in diameter (No. 16 Washburn and Moen Gage). Carbon steel wire, soft bright

¹⁰ The sole source of supply of the standard commercial apparatus, including heating block, temperature control system, flow control system, and glassware, known to the committee at this time is Falex Corp., 1020 Airpark Drive, Sugar Grove, IL 60554-9585. Glassware for the Universal Oxidation test apparatus is also available from W.A. Sales, Ltd., 419 Harvester Ct., Wheeling, IL 60090.

¹¹ 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 committee¹, which you may attend.

¹² *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

¹³ The sole source of supply of prepared catalyst coils for use with this test method known to the committee at this time is C & P Catalyst, P.O. Box 520984, Tulsa, OK 74152.

annealed and free from rust, of grade 1008 as described in Specification A 510, is preferred. Similar wire conforming to British Standard 1829 is also satisfactory.

7.2.2 *Electrolytic Copper Wire*, 1.63 mm in diameter (No. 14 American Wire Gage or No. 16 Imperial Standard Wire Gage), 99.9 % purity, conforming to Specification B 1, is preferred.

7.3 *Acetone*, reagent grade. (**Warning**—Flammable. Health hazard.)

7.4 *Heptane*, reagent grade. (**Warning**—Flammable. Health hazard.)

7.5 *Propan-2-ol (iso-Propyl Alcohol)*, reagent grade. (**Warning**—Flammable. Health hazard.)

7.6 *Isooctane*, reagent grade. (**Warning**—Flammable. Health hazard.)

7.7 *Abrasive Cloth*, silicon carbide, 100-grit with cloth backing.

7.8 *Whatman Filter Paper*, No. 41, 47-mm diameter.

7.9 *Membrane Filters*, white, plain, 47 mm in diameter pore size 8 μm . Millipore SC membrane filters (MF-type, cellulose ester),^{11,14} or equivalent have been found satisfactory.

7.10 *Air*, dry.

7.11 *Cleaning Reagent*, either Nochromix^{11,15} (**Warning**—Health hazard. Corrosive. Harmful if inhaled) or Micro^{11,16} solution.

NOTE 1—Because of extreme hazards, chromic acid cleaning solution is not recommended.

8. Corrosion Standards

8.1 ASTM Oxidation Cell Varnish Standards (ADJD6514) consist of reproductions in color of typical oxidation cell internal surfaces representing increasing degrees of staining, the reproductions being encased in plastic in the form of a plaque.

8.1.1 Keep the plastic-encased printed ASTM Oxidation Cell Varnish Standards (ADJD6514) protected from light to avoid the possibility of fading. Inspect for fading by comparing two different plaques, one of which has been carefully protected from light (new). Observe both sets in diffuse daylight (or equivalent) first from a point directly above and then from an angle of 45°. When any evidence of fading is observed, particularly at the left-hand of the plaque, it is suggested that the one that is more faded with respect to the other be discarded.

8.1.1.1 Alternatively, place a 20 mm ($\frac{3}{4}$ in.) opaque strip (masking tape) across the top of the colored portion of the plaque when initially purchased. At intervals, remove the opaque strip and observe. When there is any evidence of fading of the exposed portion, it is suggested that the standards be replaced.

8.1.2 If the surface of the plastic cover shows excessive scratching, it is suggested that the plaque be replaced.

¹⁴ The sole source of supply of the apparatus known to the committee at this time is Millipore Filter Corp., Bedford, MA.

¹⁵ The sole source of supply of Nochromix Reagent known to the committee at this time is Godax Laboratories, 720-B Erie Ave., Takoma Park, MD 20192.

¹⁶ The sole source of supply of Micro known to the committee at this time is International Products Corp., P.O. Box 70, Burlington, NJ 08016.

9. Sampling

9.1 Samples for this test can come from laboratory blends, tanks, drums, small containers, or operating equipment. Therefore, use the applicable apparatus and techniques described in Practice D 4057 or IP 2546 to obtain suitable samples.

9.2 Special precautions to preserve the integrity of a sample will not normally be required. It is good practice to avoid undue exposure of samples to sunlight or strong direct light. Fluid samples which are not homogeneous on visual inspection shall be rejected and fresh samples obtained.

10. Preparation of Apparatus

10.1 *Cleaning Glassware:*

10.1.1 Clean new glassware by washing with a hot detergent solution (using a bristle brush) and rinse thoroughly with tap water. If any visible deposits remain, soaking with a hot detergent solution has proven helpful. After final cleaning, by 24-h soak at room temperature with cleaning reagent,^{11,15} rinse thoroughly with tap water, then distilled water and allow to dry at room temperature or in an oven. Following the final distilled water rinse, an *iso*-propyl alcohol or acetone rinse will hasten drying at room temperature.

10.1.2 Used glassware should be cleaned immediately following the end of a test. Drain the used oil completely. Rinse all glassware with heptane to remove traces of oil. Then clean the glassware by the procedure described in 9.1.1 before later use.

10.2 *Cleaning Catalyst:*

10.2.1 Clean equal lengths (0.50 ± 0.01 m) of iron and copper wire with wads of absorbed cotton wet with heptane or *isooctane*. Follow by abrasion with silicon carbide cloth until fresh metal surfaces are exposed. Then wipe with dry absorbent cotton until all loose particles of metal and abrasive have been removed. In following operations, handle the catalyst with clean gloves (cotton, rubber, or plastic) to prevent contact with the skin.

NOTE 2—One procedure for preparing clean catalyst wire is to cut 0.50 ± 0.01 m lengths of wire. Hold one end of the wire tightly with a pair of clean pliers or in a vise while cleaning with the abrasive cloth. Reverse ends of the wire and repeat. Alternatively, clean a longer length of wire (3 to 5 m) and then cut 0.50 ± 0.01 m lengths from the clean wire.

10.3 *Preparation of Catalyst Coil:*

10.3.1 Twist the iron and copper wires tightly together at one end for three twists. With the two wires parallel, wind the wires around a cylindrical mandrel to produce a single coil 16 mm in inside diameter. The mandrel described in Test Method D 943 is satisfactory, but other cylindrical metal or wood stock may be used. Remove the coil from the mandrel and secure the free ends with three twists. Bend the twisted ends to conform to the shape of the spiral coil. Stretch the coil to produce a finished coil with an overall length of 80 ± 8 mm.^{11,13}

10.3.2 Store the catalyst coil in a dry, inert atmosphere until use. For storage up to 24 h, the coil can be stored in heptane. Before use, inspect stored coils to ensure that no corrosion products or contaminating materials are present.

10.4 Use a fresh catalyst coil for each test. Do not reuse coils.

11. Procedure

11.1 Measure the initial acid number of the oils to be tested by Test Method D 664, D 3339, or D 974 and the initial viscosity at 40°C by Test Method D 445.

11.2 Adjust the heating block to a temperature high enough to maintain the oil in the oxidation test cell at $155 \pm 0.5^\circ\text{C}$. The $155 \pm 0.5^\circ$ setting is predetermined with a calibrated thermometer or thermocouple sitting on the bottom of a test cell containing 100 g test fluid with 3.0 ± 0.5 L/h dry air flowing through the fluid.

11.3 Place the catalyst coil and gas inlet tube in the test cell. Obtain the total weight, W_1 , of these three components to the nearest 0.1 mg. The components may be weighed either separately or together.

11.4 Remove the gas inlet tube and weigh 100 ± 1 g of test fluid into the oxidation test cell

11.5 Fit the gas inlet tube and basic head into the test cell so that the tip of the gas inlet tube rests on the bottom of the test cell inside the catalyst coil. A PTFE sleeve may be used when inserting the basic condenser head into the cell. No grease is to be used in the ground glass joint.

11.6 Place the test cell into the preheated constant temperature block.

11.7 Connect a gas delivery tube from the flow control system to the gas inlet tube and adjust the flowmeter control to deliver dry air at 3 ± 0.5 l/h. Record this time as the start of the test.

11.8 Recheck the air flow and temperature after the test is underway and at least once daily during the test. Adjust to maintain the air flow and temperature as needed.

NOTE 3—Exhaust gases need not be collected. Vent exhaust gases properly.

11.9 After 96 h, shut off the temperature and air flow. Remove the gas inlet tube supporting the catalyst coil and remove the test cell from the heating block. Wash gas inlet tube with sufficient heptane to remove all test fluid and set it aside in a clean location.

11.10 Allow sample to cool for a minimum of 16 but no longer than 72 h

11.11 Dry a No. 41 Whatman filter and an 8 μm filter membrane in an oven at 70°C for 1 h and cool in a desiccator. Using weighing vessel, obtain the weight of the No. 41 Whatman filter, W_2 , and the 8 μm filter membrane, W_3 , each to the nearest 0.1 mg.

11.12 Filter the oil first through the preweighed No. 41 Whatman filter paper and then in a separate filtration apparatus through the 8 μm membrane filter. Transfer the filtrate into a clean 250 mL beaker and set aside for acid number and viscosity determinations.

11.13 Reassemble the Whatman filtration apparatus. Rinse the oxidation cell plus catalyst coil with sufficient 100 mL portions of heptane to ensure complete transfer of loose insolubles to the Whatman filter and removal of all test fluid from the test cell. Do not attempt to dislodge insolubles from the coil, gas inlet tube, or oxidation tube.

11.14 Rinse both filters with sufficient heptane till the fluid passing through the filters is completely colorless. With the vacuum applied, remove the clamp and funnel from the filter

membrane and funnel base. Rinse the surface of the membrane with a gentle stream of heptane, directing the stream from the edge towards the center so as to remove final traces of oil from the membrane. Maintain the vacuum for a short time to remove final traces of heptane. Transfer the membranes to the identical weighing vessels used in the initial weighing and dry for at least 1 h in the oven at 105°C . Allow to cool for at least 1 h and determine the weights of the No. 41 Whatman filter, W_{2a} , and the 8 μm filter membrane, W_{3a} , to the nearest 0.1 mg.

11.15 Dry the test cell plus gas delivery tube plus catalyst coil in an oven at 70°C for at least 1 h. Allow to cool for at least 1 h and determine the combined weight of these three components, W_{1a} .

11.16 Determine the acid number of the oxidized oil filtrate according to the same procedure used in 11.1.

11.17 Determine the viscosity of the oxidized oil filtrate at 40°C according to Test Method D 445.

11.18 Compare test cell appearance to the ASTM Oxidation Cell Varnish Standards (ADJD6514). Hold both the cell and the standard in such a manner that light reflected from them at an angle of approximately 45° will be observed.

12. Calculations

12.1 Total weight of insoluble material:

$$\text{Total Insolubles, mg: } (W_{1a} - W_1) + (W_{2a} - W_2) + (W_{3a} - W_3) \quad (1)$$

Total Weight, mg	Report
30 or lower	low
greater than 30 but less than 100	medium
100 or more	high

12.2 Change in Acid Number (AN):

$$\text{Change in AN} = AN_f - AN_i \quad (2)$$

where:

AN_f = final AN.

AN_i = initial AN.

12.3 Percent Increase in Viscosity at 40°C :

$$\% \text{ Increase in Viscosity @ } 40^\circ\text{C} = (V_f - V_i)/V_i \times 100 \quad (3)$$

where:

V_f = final viscosity.

V_i = initial viscosity.

13. Interpretation

13.1 Interpret the oxidation stabilization of the sample accordingly as the appearance of the oxidation cell agrees with one of the varnishes of the ASTM Oxidation Cell Varnish Standard (ADJD6514).

13.1.1 When the appearance of the oxidation cell is in the obvious transition state between that indicated by any two adjacent standard varnishes, judge the sample by the more varnished standard color.

14. Report

14.1 Total weight of insoluble materials in milligrams.

14.2 Change in acid number (AN).

14.3 Percent increase in viscosity at 40°C .

14.4 Report the varnishing in accordance with one of the classifications listed in Table 1.

TABLE 1 Oxidation Cell Varnish Classification

Classification	Designation	Description ^A
0	no varnish	clear
1	slight varnish	clear to light gray-brown
2	medium varnish	amber to light brown
3	severe varnish	dark brown to dark red and black with black deposits

^A The ASTM Oxidation Cell Varnish Standard (ADJD6514) is a colored reproduction of oxidation cells characteristic of these descriptions.

15. Precision and Bias

15.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

15.2 *Repeatability*—The difference between two single and independent 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 in one case in twenty:

Sludge	Repeatability 3.4X ^{0.8}
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Viscosity Change	0.54(X + 1)
AN Change	0.75(X + 0.1)
where: X denotes mean value	

15.3 *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, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Sludge	Repeatability 7.4X ^{0.8}
Viscosity Change	1.59(X + 1)
AN Change	1.57(X + 0.1)
where: X denotes mean value	

15.4 *Bias*—The procedure in this test method for measuring oxidation life of an oil has no bias because the value of the oxidation life is defined only in terms of this test method.

15.5 In the case of pass/fail data, no generally accepted method for determining precision or bias is currently available.

16. Keywords

16.1 high temperature fluid stability test; oils—lubricating; oxidation stability; turbine oil; universal oxidation test

APPENDIX

(Nonmandatory Information)

X1. PROCEDURE FOR PACKAGING CATALYST COILS

X1.1 Materials

X1.1.1 *Test Tubes*, borosilicate glass, 250 mm length, 25 mm outside diameter, approximately 22 mm inside diameter.

X1.1.2 *Caps*, for test tubes, polyethylene, cylindrical shape, designed to closely grip outside surface of the test tube.^{11,17}

X1.1.3 *Desiccant Bags*, 3-g silica gel granules in paper package, approximately 76 mm (3 in.) long, 51 mm (2 in.) wide, 3 mm (1/8 in.) thick.^{11,18}

¹⁷ The sole source of supply of the apparatus known to the committee at this time is Caplugs, No. 1-SC, available from Unisco, Inc., 600 Palisade Ave., Englewood Cliffs, NJ.

¹⁸ The sole source of supply of the apparatus known to the committee at this time is Dri-Pax Air Dryers, available from Davison Chemical Div., W.R. Grace and Co., Baltimore, MD.

X1.1.4 *Flushing Tube*, stainless steel or glass, approximately 5 mm (3/16 in.) outside diameter, 305 mm (12 in.) long, to deliver nitrogen to bottom of test tube.

X1.1.5 *Nitrogen Gas*, 99.7 % minimum purity.

X1.2 *Procedure*—Flush a new test tube with nitrogen gas, using the flushing tube, to blow out any loose particles. The tube must be visibly clean and dry. Hold the tube on an angle and gently slide the catalyst coil into the tube. Add a desiccant bag which has been folded lengthwise to fit in the tube. Insert the nitrogen flushing tube down the middle of the test tube, to the bottom, and blow nitrogen through the tube for several seconds. Immediately after withdrawing the flushing tube, seal the test tube with a polyethylene cap.

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