



## Standard Test Method for Oxidation Characteristics of Inhibited Mineral Oils<sup>1</sup>

This standard is issued under the fixed designation D 943; 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 is used to evaluate the oxidation stability of inhibited steam-turbine oils in the presence of oxygen, water, and copper and iron metals at an elevated temperature. The test method is also used for testing other oils such as hydraulic oils and circulating oils having a specific gravity less than that of water and containing rust and oxidation inhibitors.

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.* For specific precautionary statements, see Section 6.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- A 510 Specification for General Requirements for Wire Rods and Coarse Round Wire, Carbon Steel<sup>2</sup>
- B 1 Specification for Hard-Drawn Copper Wire<sup>3</sup>
- D 329 Specification for Acetone<sup>4</sup>
- D 770 Specification for Isopropyl Alcohol<sup>4</sup>
- D 1193 Specification for Reagent Water<sup>5</sup>
- D 3244 Practice for Utilization of Test Data to Determine Conformance with Specifications<sup>6</sup>
- D 3339 Test Method for Acid Number by Semi-Micro Color Indicator Titration<sup>6</sup>
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>6</sup>
- D 4310 Test Method for Determination of the Sludging and Corrosion Tendencies of Inhibited Mineral Oils<sup>6</sup>
- E 1 Specification for ASTM Thermometers<sup>7</sup>

#### 2.2 IP Standards:<sup>8</sup>

#### Specifications for IP Standard Thermometers

- 2.3 *British Standard:*<sup>9</sup>
- BS 1829

### 3. Summary of Test Method

3.1 The oil sample is contacted with oxygen in the presence of water and an iron-copper catalyst at 95°C. The test continues until the measured acid number of the oil is 2.0 mg KOH/g or above. The number of test hours required for the oil to reach 2.0 mg KOH/g is the “oxidation lifetime.”

### 4. Significance and Use

4.1 This test method is widely used for specification purposes and is considered of value in estimating the oxidation stability of lubricants, especially those that are prone to water contamination. It should be recognized, however, that correlation between results of this method and the oxidation stability of a lubricant in field service may vary markedly with field service conditions and with various lubricants. The precision statement for this method was determined on steam turbine oils.

NOTE 1—Furthermore, in the course of testing a lubricant by this method, other signs of deterioration, such as sludge formation or catalyst coil corrosion, may appear that are not reflected in the calculated oxidation lifetime. The subcommittee responsible for this method is investigating the application of alternative criteria for evaluation of lubricants using this test apparatus. Test Method D 4310 is now available for sludge measurement.

### 5. Apparatus

5.1 *Oxidation Cell*, of borosilicate glass, as shown in Fig. 1, consisting of a test tube, condenser, and oxygen delivery tube. The test tube has a calibration line at 300 mL (maximum error 1 mL). This calibration applies to the test tube alone at 20°C.

5.2 *Heating Bath*, thermostatically controlled, capable of maintaining the oil sample in the oxidation cell at a temperature of  $95 \pm 0.2^\circ\text{C}$ , fitted with a suitable stirring device to provide a uniform temperature throughout the bath, and large enough to hold the desired number of oxidation cells immersed in the heating bath to a depth of  $390 \pm 10$  mm and in the heating liquid itself to a depth of  $355 \pm 10$  mm.

5.2.1 Studies have suggested that direct sunlight or artificial

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.09 on Oxidation.

Current edition approved June 10, 1999. Published August 1999. Originally published as D 943 – 47. Last previous edition D 943 – 98.

In 1976, the method ceased to be a joint ASTM-IP standard.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 01.03.

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

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 06.04.

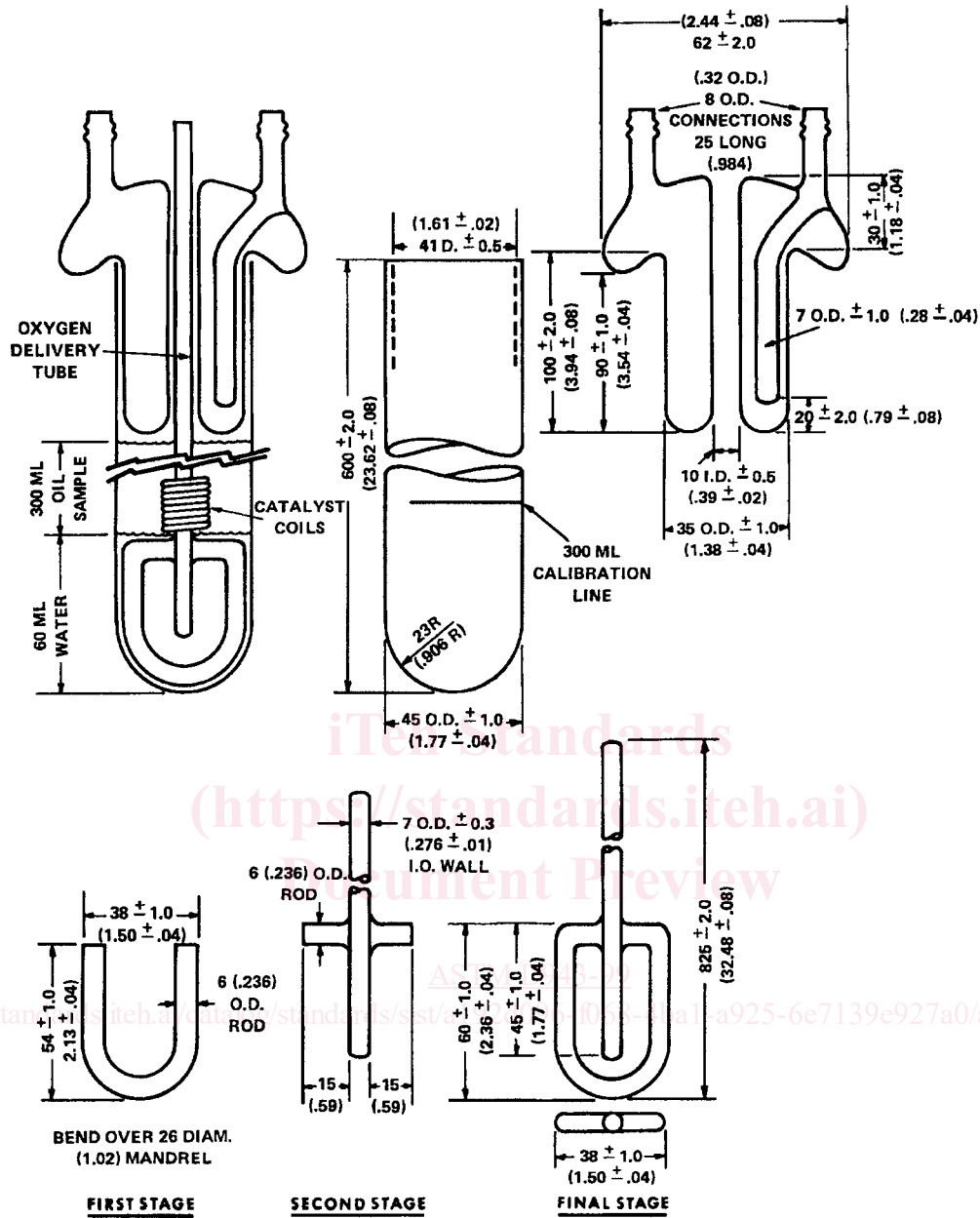
<sup>5</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>6</sup> *Annual Book of ASTM Standards*, Vol 05.02.

<sup>7</sup> *Annual Book of ASTM Standards*, Vol 14.03.

<sup>8</sup> Available from the Institute of Petroleum, 61 New Cavendish St., London W1M, 8AR, England.

<sup>9</sup> Available from British Standards Institute, 2 Park St., London, England W1A2135.



All dimensions are in millimetres (inches)

- NOTES:
1. The oxidation test tube has a calibration line at 300 ml. This calibration applies to the test tube alone at 20°C.
  2. Open tube ends to be ground and fire-polished.

FIG. 1 Oxidation Cell

light may adversely influence the results of this test.<sup>10</sup> To minimize effects of light exposure on the lubricant being tested, light shall be excluded from the lubricant by one or more of the following ways:

5.2.1.1 Use of heated liquid baths that are designed and constructed of metal, or combinations of metals and other suitable opaque materials, that prevent light from entering the

test cell from the sides is preferred. If a *viewing window* is included in the design, this *viewing window* shall be fitted with a suitable opaque cover and be kept closed when no observation is being made.

5.2.1.2 If glass heating baths are used, the bath shall be wrapped with aluminum foil or other opaque material.

5.2.1.3 Bright light entering the test cell from directly overhead can be eliminated by use of an opaque shield.

5.3 *Flowmeter*, with a capacity of at least 3 L of oxygen/h, and an accuracy of ± 0.1 L/h.

<sup>10</sup> A summary of the results of these studies is available from ASTM Headquarters. Request RR:D02-1365.

5.4 *Heating Bath Thermometer*—ASTM Solvents Distillation Thermometer having a range from 72 to 126°C, and conforming to the requirements for Thermometer 40C as prescribed in Specification E 1, or for Thermometer 70C as prescribed in Specifications for IP Standard Thermometers. Alternatively, temperature-measuring devices of equal or better accuracy may be used.

5.5 *Oxidation Cell Thermometer*,<sup>8</sup> having a range from 80 to 100°C, graduated in 0.1°C, total length—250 mm, stem diameter—6.0 to 7.0 mm, calibrated for 76-mm immersion.<sup>11</sup> Alternatively, temperature-measuring devices of equal or better accuracy may be used.

<sup>11</sup> Brooklyn thermometer No. 21276-RM, available from Brooklyn Thermometer Co., Farmingdale, N. Y., or equivalent has been found satisfactory for this purpose.

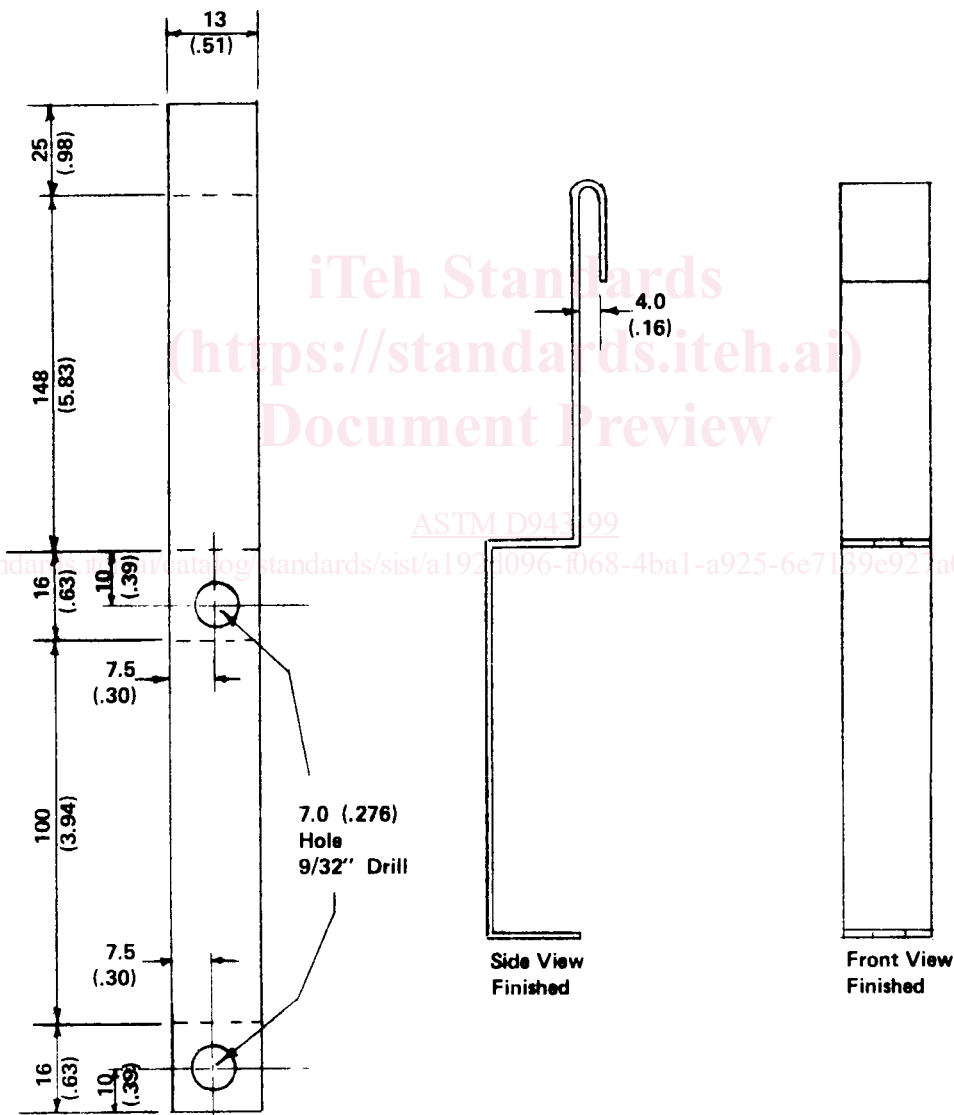
5.6 *Thermometer Bracket*, for holding the oxidation cell thermometer, of 18-8 stainless steel, having the dimensions shown in Fig. 2. The thermometer is held in the bracket by two fluoroelastomer O-rings of approximately 5 mm inside diameter. Alternatively, thin stainless steel wire may be used.

5.7 *Wire Coiling Mandrel*, as shown in Fig. 3.

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

5.9 *Syringes*, glass, with Luer-Lok locking connectors, 10 and 50-mL capacities for sampling, and water additions, respectively.

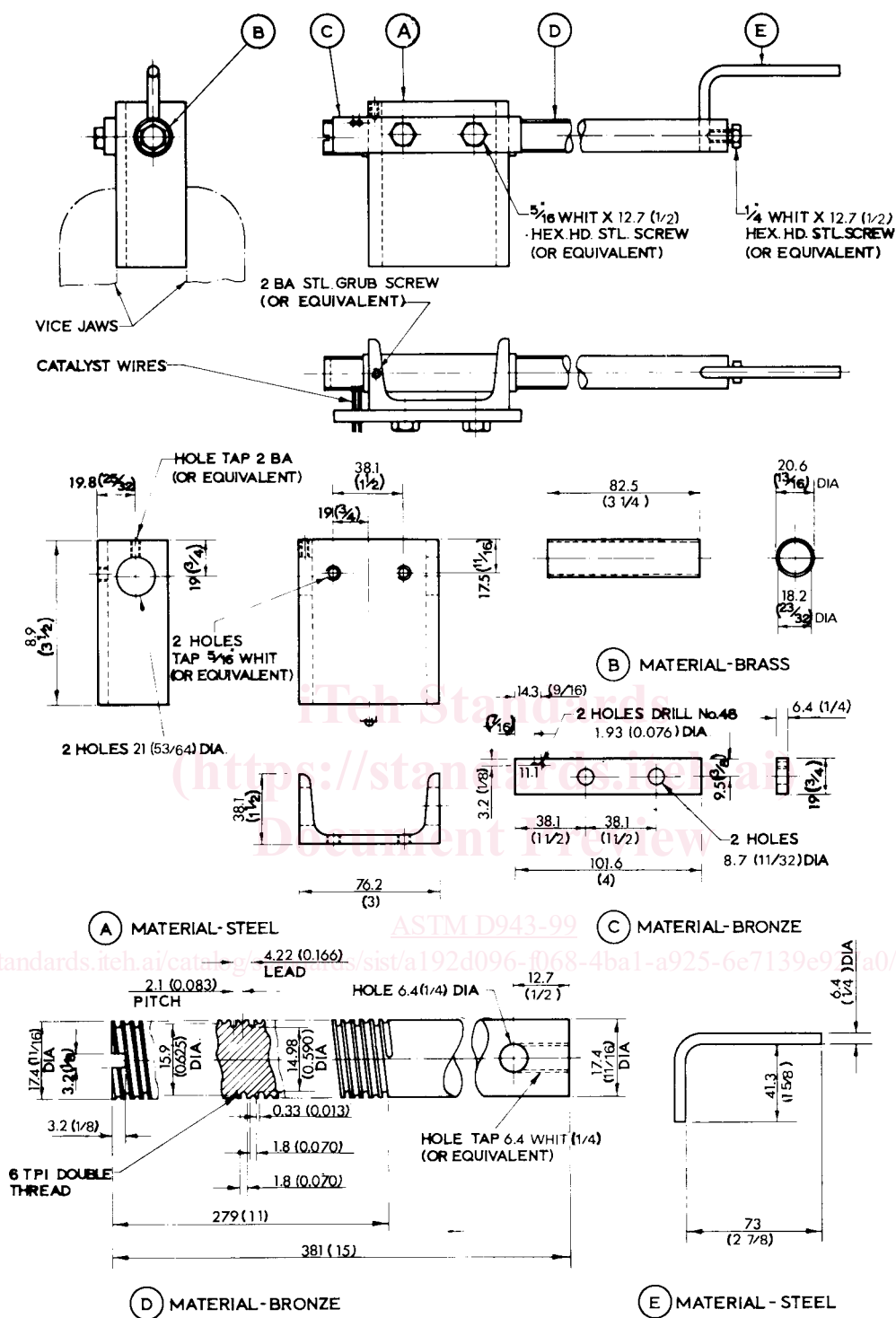
5.10 *Syringe Sampling Tube*, Grade 304 stainless steel tubing, 2.11 mm (0.083 in.) in outside diameter, 1.60 mm (0.063 in.) in inside diameter, 559 ± 2 mm (24.0 ± 0.08 in.) long, with one end finished at 90° and the other end fitted with



All dimensions are in millimetres (inches).  
Material: 18-8 Stainless Steel, 22 Gage (0.792 mm).

FIG. 2 Thermometer Bracket

ASTM D 943



Dimensions are in millimetres (inches).  
**FIG. 3 Mandrel for Winding Catalyst Coils**

a Luer-Lok female connector. The Luer-Lok connector is preferably of elastomeric material, such as polyfluorovinylchloride to provide a good seal with the syringe.<sup>12</sup>

5.11 *Stopper*, for Luer fitting of syringe sampling tube,

made of polytetrafluoroethylene or polyfluorovinylchloride.<sup>13</sup>  
 5.12 *Sampling Tube Holder*, for supporting the syringe sampling tube, made of methyl methacrylate resin, having the dimensions shown in Fig. 4.

<sup>12</sup> Hamilton Co. catalog number KF-714 syringe needles with polychlorotrifluoroethylene hub, or equivalent, have been found satisfactory for this purpose.

<sup>13</sup> Suitable stoppers are available from suppliers of infrared spectrometer sample cells.

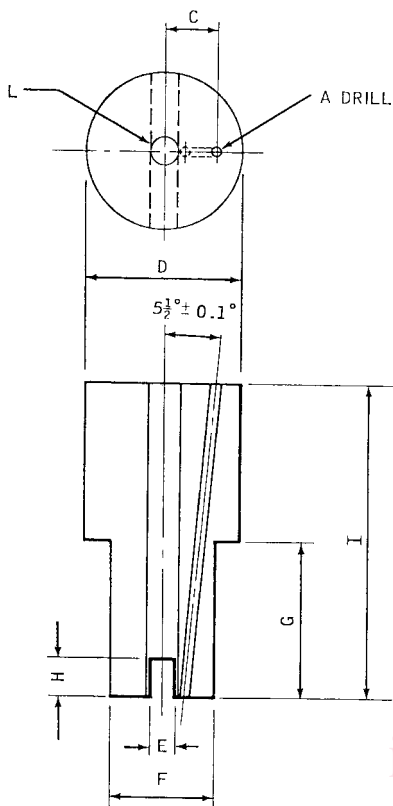


Table of Dimensions

Dimension	mm		in.	
	Dimension	Tolerance	Dimension	Tolerance
A	2.4	0.1	0.09375	0.005
B	7.5	0.1	0.296875	0.005
C	12.7	0.25	0.50	0.01
D	38.1	0.50	1.50	0.02
E	6.4	0.50	0.25	0.02
F	25.4	0.25	1.00	0.01
G	38.1	0.50	1.50	0.02
H	9.5	0.50	0.375	0.02
I	76.2	0.50	3.00	0.02

FIG. 4 Sampling Tube Holder

5.13 *Sampling Tube Spacer*, for positioning the end of the sampling tube above the sampling tube holder, made of a length of plastic tubing polyvinyl chloride, polyethylene, polypropylene, or polytetrafluoroethylene having an inside diameter of approximately 3 mm (1/8 in.) and 51 ± 1 mm (2.0 ± 0.04 in.) length.

5.14 *Flexible Tubing*, polyvinyl chloride approximately 6.4 mm (1/4 in.) in inside diameter with a (3/32-in.) wall for delivery of oxygen to the oxidation cell.

## 6. Reagents and Materials

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

6.2 *Acetone* (**Warning**—Health hazard flammable), 99.5 % grade, conforming to Specification D 329.

6.3 *Catalyst Wires*:

6.3.1 *Low-Metalloid Steel Wire*, <sup>14</sup> 1.59 mm (0.0625 in.) in diameter (No. 16 Washburn and Moen Gage).

6.3.2 *Electrolytic Copper Wire*, 1.63 mm (0.064 in.) in diameter (No. 16 Imperial Standard Wire Gage or No. 14 American Wire Gage), 99.9 % purity, conforming to Specification B 1. Soft copper wire of an equivalent grade may also be used.

NOTE 2—Alternatively, suitably prepared catalyst coils may be purchased from a supplier.

6.4 *Detergent*, water-soluble.

6.5 *n-Heptane*, reagent grade. (**Warning**—Flammable. Harmful if inhaled.)

6.6 *Hydrochloric Acid* (**Warning**—Toxic and Corrosive.), concentrated [36 mass % (relative density 1.19)].

6.7 *Isopropyl Alcohol* (**Warning**—Flammable. See Annex A1.4), conforming to Specification D 770.

6.8 *Oxygen* (**Warning**—Vigorously accelerates combustion.), 99.5 % minimum purity, with pressure regulation adequate to maintain a constant flow of gas through the apparatus. The use of a two-stage pressure regulator on tank oxygen is recommended.

6.9 *Cleaning Reagent*, cleaning by a 24-h soak at room temperature either in Nochromix<sup>15</sup> (**Warning**—Corrosive. Health Hazard.) or in Micro<sup>16</sup> solution.

## 7. Sampling

7.1 Samples for this test can come from tanks, drums, small containers, or even operating equipment. Therefore, use the applicable apparatus and techniques described in Practice D 4057.

7.2 For one single determination the minimum required sample size is 300 mL.

## 8. Preparation of Apparatus

8.1 *Cleaning Catalyst*—Immediately prior to winding a catalyst coil, clean a 3.00 ± 0.01-m length of iron wire and an equal length of copper wire with wads of absorbent cotton wet with *n*-heptane and follow by abrasion with abrasive cloth until a fresh metal surface is exposed. Then wipe with dry absorbent cotton until all loose particles of metal and abrasive have been removed. In subsequent operations handle the catalyst wires with clean gloves (cotton, rubber, or plastic) to prevent contact with the skin.

8.2 *Preparation of Catalyst Coil*—Twist the iron and copper wires tightly together at one end for three turns and then wind them simultaneously alongside each other on a threaded mandrel (Fig. 3), inserting the iron wire in the deeper thread. Remove the coil from the mandrel, twist the free ends of the iron and copper wires together for three turns, and bend the

<sup>14</sup> Carbon steel wire, soft bright annealed and free from rust, of Grade 1008 as described in Specification A 510 is satisfactory. Similar wire conforming to BS 1829 is also satisfactory. If these steels are not available, other equivalent steels may be used, provided they are found to be satisfactory in comparative tests using Test Method D 943.

<sup>15</sup> Nochromix is available from Godax Laboratories, Inc., 720-B Erie Ave., Takoma Park, MD 20912.

<sup>16</sup> Micro is available from International Products Corp., P.O. Box 70, Burlington, NJ 08016.

twisted ends to conform to the shape of the spiral coil. The overall length of the finished coil should be  $225 \pm 5$  mm ( $8.9 \pm 0.2$  in.). If necessary, the coil may be stretched to give the required length. (Note 2 and Note 3.)

NOTE 3—The finished catalyst coil is a double spiral of copper and iron wire,  $225 \pm 5$  mm ( $8.9 \pm 0.2$  in.) overall length and 15.9 to 16.5 mm (0.625 to 0.650 in.) inside diameter. The turns of wire are evenly spaced, and two consecutive turns of the same wire are 3.96 to 4.22 mm (0.156 to 0.166 in.) apart, center to center. The mandrel shown in Fig. 3 is designed to produce such a coil. Using this mandrel, the iron wire is wound on a thread of 14.98-mm (0.590-in.) diameter, while the copper wire is wound on a thread of 15.9-mm (0.625-in.) diameter. The smaller diameter is to allow for *springback* of the steel wire after winding, so as to give 15.9-mm consistent inside diameter. Use of a very soft annealed steel wire may allow use of identical thread diameters for the two wires. Any arrangement that leads to the coil configuration described above is satisfactory.

8.3 *Catalyst Storage*—The catalyst coil may be stored in a dry, inert atmosphere prior to use. A suitable procedure for catalyst storage is given in Appendix X1. Before use it should be inspected to assure that no corrosion products or contaminating materials are present. For overnight storage (less than 24 h) the coil may be stored in *n*-heptane.

8.3.1 *n*-Heptane used for catalyst storage must be free of traces of water and corrosive materials. Redistilled *n*-heptane conforming to 6.5 and stored in a tightly sealed bottle is suitable.

8.4 *Cleaning New Glassware*—Wash new oxygen delivery tubes, condensers, and test tubes with a hot detergent solution and rinse thoroughly with tap water. Clean the interiors of the test tubes, exteriors of the condensers, and both interiors and exteriors of the oxygen delivery tubes with cleaning reagent. Rinse thoroughly with tap water until all cleaning solution is removed. Rinse all parts with reagent water and allow to dry at room temperature or in an oven. The final reagent water rinse may be followed by an isopropyl alcohol rinse, or acetone rinse, optionally followed by dry air blowing, to hasten drying at room temperature.

8.5 *Cleaning Used Glassware*—Immediately following termination of a test, drain the oil completely from the test tube. Rinse all the glassware with *n*-heptane to remove traces of oil, wash with a hot detergent solution using a long-handled brush, and rinse thoroughly with tap water. If deposits still adhere to the glassware, a method that has been found useful is to fill the test tubes with detergent solution, insert the oxygen delivery tubes and condensers, and place the tubes in the bath at test temperature. Several hours soaking in this manner often serves to loosen all adhering deposits except iron oxide. Subsequent rinsing with hot ( $50^{\circ}\text{C}$ ) hydrochloric acid will serve to remove iron oxide. After all deposits are removed, rinse all glassware with a cleaning reagent. Rinse thoroughly with tap water until all acid is removed. Rinse all parts with reagent water and allow to dry at room temperature or in an oven. The final reagent water rinse may be followed by an isopropyl alcohol rinse, or acetone rinse, optionally followed by dry air blowing, to hasten drying at room temperature. Store glassware in a dry dust-free condition until ready to use.

8.6 *Cleaning Used Sampling Tube*—Immediately following termination of a test, drain the oil completely from the sampling tube. Rinse the tube with *n*-heptane to remove traces

of oil and any tenacious organic residues. Repeat the rinsing procedure with *n*-heptane and blow dry with air or nitrogen.

## 9. Procedure

9.1 Adjust the heating bath to a temperature high enough to maintain the oil in the oxidation test cell at the required temperature of  $95 \pm 0.2^{\circ}\text{C}$ .

9.2 Fill the empty oxidation test tube with 300 mL of the oil sample to the graduation mark. Slide the catalyst coil over the inlet of the oxygen delivery tube. If the wires are uneven at one end of the coil, position the coil so that this end is down. Place the oxygen delivery tube with the coil into the test tube. Place the condenser over the oxygen delivery tube and test tube. Place the sampling tube holder over the oxygen delivery tube. Insert the syringe sampling tube through the syringe sampling tube spacer, and into the sampling tube holder, as shown in Fig. 5. Position the bottom end of the sampling tube inside the catalyst coil. Insert a stopper in the Luer-Lok fitting end of the sampling tube. Immerse the test tube in the heating bath. Adjust the heating bath liquid level so that the tube is immersed in the liquid to a depth of  $355 \pm 10$  mm. Connect the condenser to the cooling water. The temperature of the outlet water should not exceed  $32^{\circ}\text{C}$  at any time during the test.

NOTE 4—As an alternative to using the sampling tube holder and sampling tube spacer, the sampling tube may be secured in position by taping or clamping to the oxygen delivery tube using a suitable adhesive tape or clamp. The sampling tube is taped or clamped to the oxygen delivery tube at a distance of approximately 25 mm (1 in.) above the top of the condenser. The bottom of the sampling tube is positioned at  $152 \pm 6$  mm ( $6 \pm 0.25$  in.) from the bottom curved end of the oxygen delivery tube.

9.3 Connect the oxygen delivery tube to the oxygen supply (see 6.8) through the flowmeter using new polyvinyl chloride flexible tubing no more than 600 mm in length. Before using, the interior of the new tubing should be rinsed with *n*-heptane and blown dry with air. Adjust the rate of flow to  $3 \pm 0.1$  L and continue flow for 30 min.

9.4 Raise the condenser unit from the oxidation cell and add 60 mL of reagent water through the opening thus provided. The test is considered to start at this point.

9.5 Throughout the duration of the test, maintain the temperature of the oil-water mixture (sample temperature) at  $95 \pm 0.2^{\circ}\text{C}$  in each test cell with oxygen flowing. Accomplish this by maintaining the bath at the temperature that is found necessary to give the required  $95^{\circ}\text{C}$  sample temperature. The temperature of the bath is always higher than the sample temperature due to the cooling effect of the oxygen gas flow, and depends on heating bath medium, capacity, circulation, and on the number of tests cells in the bath. Measure the sample temperature by a thermometer positioned in the oxidation cell by a thermometer bracket, as in Fig. 6 (Note 5). Make temperature measurements only with new, undepleted, oil samples, and preferably with dummy cells used specifically for temperature measurement. When an actual test sample is used, remove the thermometer immediately after temperature measurement is complete. Check the temperature in this way in various parts of a multiple-cell bath to verify uniformity of temperature control. Once the required bath temperature is found, maintain at that temperature  $\pm 0.2^{\circ}\text{C}$ .