



Standard Test Method for Demulsibility Characteristics of Lubricating Oils¹

This standard is issued under the fixed designation D 2711; 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 measurement of the ability of oil and water to separate from each other. It is intended for use in testing medium and high-viscosity lubricating oils.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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:

D 96 Test Method for Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure)²

D 1193 Specification for Reagent Water³

D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)²

3. Summary of Test Method

3.1 *For Oils That Do Not Contain Extreme Pressure (EP) Additives*—A 405-mL sample of the oil and 45 mL of distilled water are stirred together for 5 min at 82°C (180°F) in a special graduated separatory funnel. After a 5-h settling period following the stirring, a percentage of the water in the oil and the volumes of water and emulsion separating from the oil are measured and recorded.

3.2 *For Oils That Contain Extreme Pressure (EP) Additives*—A 360-mL sample of the oil and 90 mL of distilled water are stirred together for 5 min at 82 °C (180 °F) in a special graduated separatory funnel. After a 5-h settling period following the stirring, percentage of water in the oil and the volumes of water and emulsion separating from the oil are measured and recorded.

4. Significance and Use

4.1 This test provides a guide for determining the demulsi-

bility characteristic of lubricating oils that are prone to water contamination and may encounter the turbulence of pumping and circulation capable of producing water-in-oil emulsions.

5. Apparatus

5.1 *Stirrer*,⁴ constructed from parts shown in Fig. 1 and Fig. 2.

5.2 *Special Graduated Separatory Funnel*,⁴ as shown in Fig. 3.

5.3 *Heating Bath*, sufficiently large and deep to permit the immersion of at least two test separatory funnels in the bath liquid up to their 500-mL graduation mark. The bath shall be capable of maintaining a temperature of $82 \pm 1^\circ\text{C}$ ($180 \pm 2^\circ\text{F}$) and shall be so equipped to hold the separatory funnels securely in a position so that the vertical axis of the stirrer corresponds to the center line of the separatory funnel during the mixing of the oil and water.

5.4 *Centrifuge*, as described in Method A of Test Method D 96 and Test Method D 1796.

5.5 *Centrifuge Tubes*, long-form, 195 to 203 mm (8 in.) as described in Fig. 1 of Test Methods D 96 and D 1796.

6. Materials

6.1 *Cleaning Solvent*—Any suitable solvent capable of cleaning and effectively removing any oil or fluid from the stirrer and graduated cylinder. 1,1,1-Trichloroethane has been found suitable for use in this test method.

NOTE 1—**Warning:** 1,1,1-Trichloroethane, Harmful if inhaled or swallowed. Eye irritant. High concentration can cause unconsciousness or death.

NOTE 2—In cases in which the use of 1,1,1 trichloroethane is unacceptable, some laboratories are using heptane or mineral spirits as alternative solvents. The effect on the precision of this test method when using an alternate solvent has not been determined.

6.2 *Water*—Type II reagent grade water conforming to Specification D 1193.

7. Preparation of Apparatus

7.1 Clean the graduated cylinder by removing any film of oil (or fluid) with cleaning solvent followed by a wash first with acetone (see Note 3) and then with tap water, immerse the cylinder in a suitable cleaning solution. Rinse thoroughly with tap water and then with nonreferee reagent water.

⁴ Available from Research Appliance Co., Moose Lodge Rd., Dept. T, Cambridge, MD 21613.

¹ 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.L on Industrial Lubricants.

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² *Annual Book of ASTM Standards*, Vol 05.01.

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

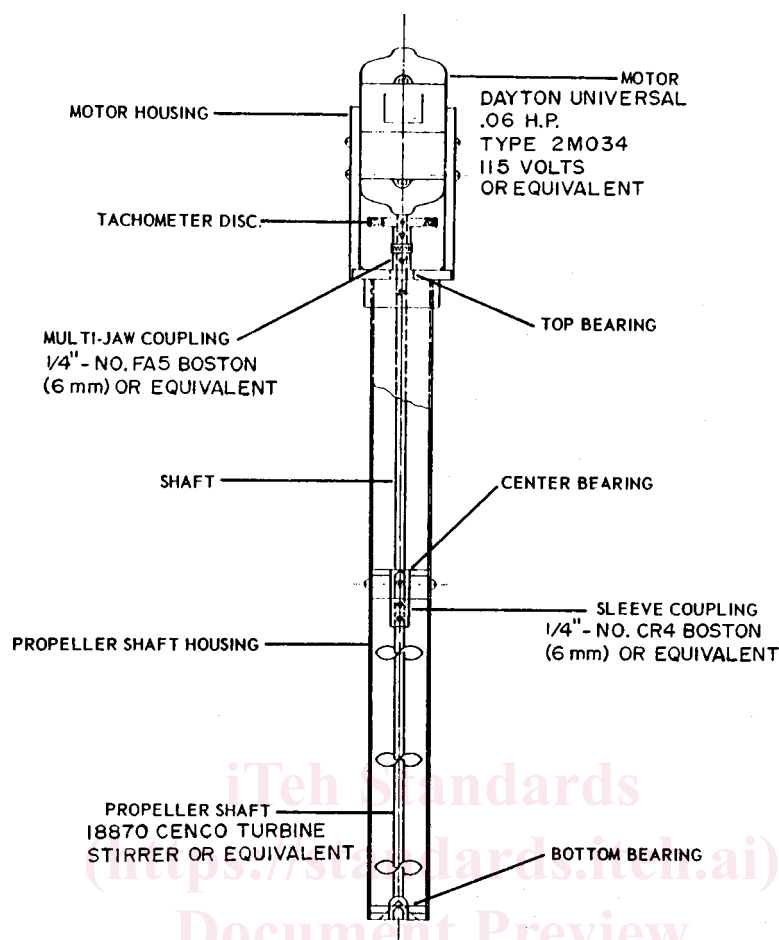


FIG. 1 Stirrer

NOTE 3—**Warning:** Extremely flammable vapors may cause flash fires.

7.2 Clean the stirrer by appropriate means, using the cleaning solvent (6.1). Allow the stirrer to air dry completely prior to use in the test.

8. Procedure

8.1 Heat the bath liquid to $82 \pm 1^\circ\text{C}$ ($180 \pm 2^\circ\text{F}$) and maintain this temperature throughout the test.

8.2 Measure the oil under test, at room temperature, directly into the separatory funnel to a volume of 405 mL. Place the separatory funnel and oil in the constant-temperature bath and bring it to a temperature of 82°C (180°F). Add 45 mL of distilled water, measured at room temperature, to the oil. Immerse the stirrer in the oil and position it carefully as follows: lower the stirrer until it touches the bottom of the funnel, then raise it approximately 25 mm (1 in.). Ensure that the vertical axis of the stirrer corresponds with the vertical center line of the funnel. Slowly bring the stirrer motor to a speed of 4500 ± 500 rpm within 25 to 30 s, and operate for a total of 5 min, including the start-up time. Then withdraw the stirrer from the oil-water mixture but not entirely out of the separatory funnel. Allow the stirrer to drain for 5 min, then remove from the separatory funnel and clean.

8.3 Five hours after stirring has stopped, withdraw a 50-mL sample from the center of the funnel and approximately 50 mm (2 in.) below the surface of the oil-water mixture, using a

50-mL pipet. Discharge the contents of the pipet into a centrifuge tube and determine the water present in the sample using Test Methods D 96 or D 1796. Record the results as "percent water in the oil."

8.4 With minimum delay, after sampling for the "percentage of water in the oil," remove the separatory funnel from the bath and draw off any free water that has separated from the oil-water mixture into a 50-mL graduated cylinder. Allow this water to reach room temperature, measure, and record the volume.

8.5 After removing the free water from the separatory funnel, reduce the volume of the fluid remaining to 100 mL by carefully siphoning the fluid off the top (end of siphon should not be more than 20 mm below the surface of the fluid at any time) down to the 100-mL graduation mark on the separatory funnel. Drain the remaining 100 mL of fluid (oil, water, and emulsion) directly into a centrifuge tube.

8.6 Centrifuge the tube and its contents for a period of 10 to 15 min at a relative centrifugal force of 700. Record the volume of water and emulsion separated by centrifuging.

8.7 Perform a minimum of two determinations on each oil sample by repeating procedure 8.1-8.6. If the difference in the two sets of results obtained is outside the repeatability limits of Procedure A, discard them and obtain two additional sets of results.

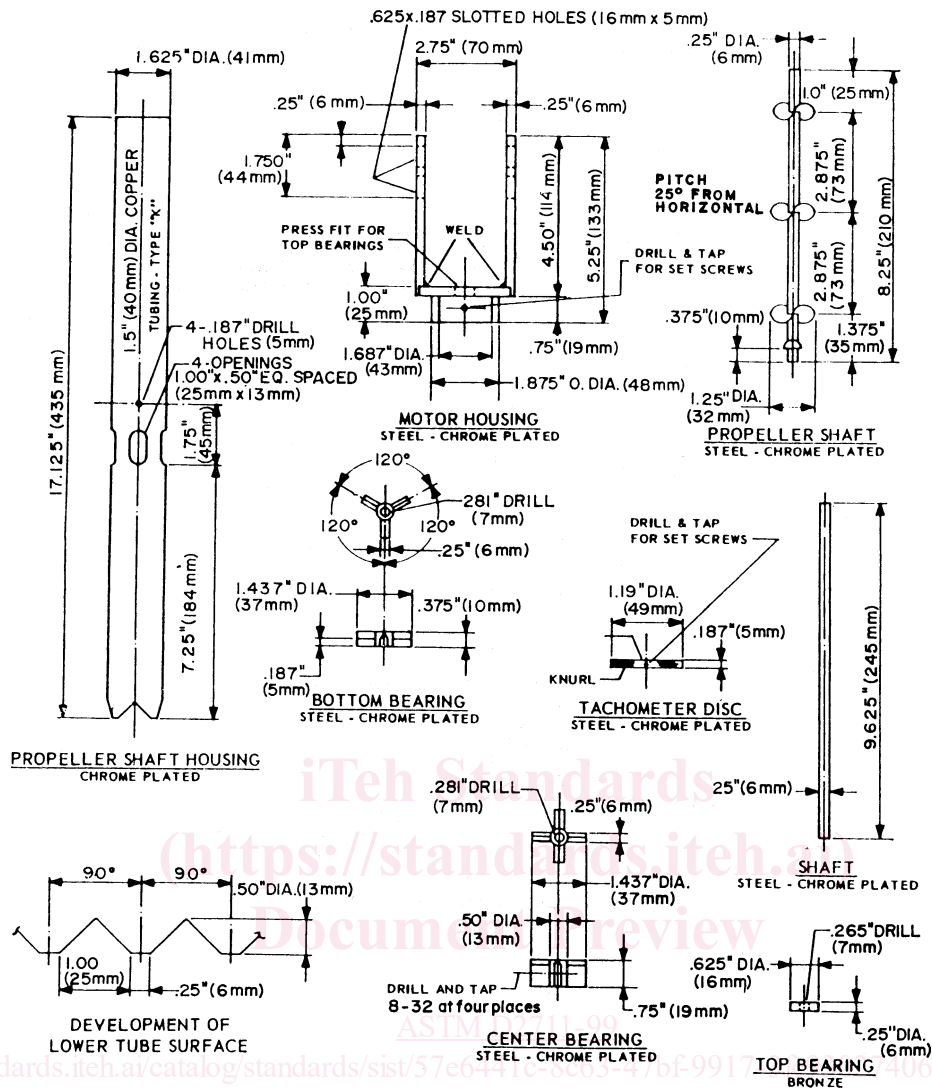


FIG. 2 Detailed Construction of Stirrer

9. Procedure B for Oils That Contain EP Additives

9.1 Heat the bath liquid to 82 ± 1 °C (180 ± 2 °F) and maintain this temperature throughout the test.

9.2 Measure the oil under test, at room temperature, directly into the separatory funnel to a volume of 360 mL. Place the separatory funnel and oil in a constant-temperature bath and bring to a temperature of 82 °C (180 °F). Add 90 mL of distilled water, measured at room temperature, to the oil. Immerse the stirrer in the oil and position it carefully as follows: lower the stirrer until it touches the bottom of the funnel, then raise it approximately 25 mm (1 in.). Ensure that the vertical axis of the stirrer corresponds with the vertical center line of the funnel. Slowly bring the stirrer motor to a speed of 2500 ± 250 rpm within 25 to 30 s, and operate for a total of 5 min, including the start-up time (Note 4). Then withdraw the stirrer from the oil-water mixture but not entirely out of the separatory funnel. Allow the stirrer to drain for 5 min, then remove it from the separatory funnel and clean.

9.3 Five hours after stirring has stopped, withdraw a 50-mL sample from the center of the funnel approximately 50 mm (2 in.) below the surface of the oil-water mixture, using a 50-mL

pipette. Discharge the contents of the pipet into a centrifuge tube and determine the water present in the sample using Test Methods D 96 or D 1796. Record the results as "percent water in the oil".

9.4 Immediately after sampling for the "percentage of water in the oil", remove the separatory funnel from the bath and draw off any free water that has separated from the oil-water mixture into a 100-mL graduated cylinder. Allow this water to reach room temperature, measure, and record the volume.

9.5 After removing the free water from the separatory funnel, reduce the volume of the fluid remaining to 100 mL by carefully siphoning the fluid off the top (end of siphon should not be more than 20 mm below the surface of the fluid at any time) down to the 100-mL graduation mark on the separatory funnel. Drain the remaining 100 mL of fluid (oil, water and emulsion) directly into a centrifuge tube.

9.6 Centrifuge the tube and its contents for a period of 10 to 15 min at a relative centrifugal force of 700. Record the volume of water and emulsion separated by centrifuging.

9.7 Perform a minimum of two determinations on each oil sample by repeating procedure 9.1-9.3. If the difference in the