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Standard Test Method for Water Separability of Petroleum Oils and Synthetic Fluids[1]

This standard is issued under the fixed designation D 1401; 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.

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

1. Scope

~~1.1This test method covers measurement of the ability of petroleum oils or synthetic fluids to separate from water.~~

~~NOTE1—Although developed specifically for steam-turbine oils having viscosities of 28.8–90 cSt (mm~~

1.1 This test method covers measurement of the ability of petroleum oils or synthetic fluids to separate from water. Although developed specifically for steam-turbine oils having viscosities of 28.8–90 mm^2/s at 40°C, this test method ~~can~~may be used to test oils of other types having various viscosities and synthetic fluids at other test temperatures. It is recommended, however, that the test temperature be raised to 82 ± 1°C when testing products more viscous than 90 ~~cSt (mm~~mm^2/s~~) at 40°C. For higher viscosity oils where there is insufficient mixing of oil and water, Test Method D2711, is recommended.~~

~~Other test temperatures such as 25°C can also be used.~~

~~When testing synthetic fluids whose relative densities are greater than that of water, the procedure is unchanged, but it should be noted that the water will probably float on the emulsion or liquid.~~

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

~~1.3~~*/s at 40°C. For higher viscosity oils where there is insufficient mixing of oil and water, Test Method D 2711 is recommended. Other test temperatures such as 25°C may also be used. A 1% sodium choloride (NaCl) solution or synthetic seawater may be used in place of distilled water when testing certain oils or fuels used in marine applications.*

1.2 When testing synthetic fluids whose relative densities are greater than that of water, the procedure is unchanged, but it should be noted that the water will probably float on the emulsion or liquid.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.* ~~For specific precautionary statements, see 6.3-6.5~~For specific warning statements, see Section 6.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 665 Test Method for Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water

D 1141 Practice for the Preparation of Substitute Ocean Water

D 1193 Specification for Reagent Water

D 2711 Test Method for Demulsibility Characteristics of Lubricating Oils

D 4057 ~~Practice for Manual Sampling of Petroleum and Petroleum Products~~ Practice for Manual Sampling of Petroleum and Petroleum Products

2.2 *ISO Standards:*[3]

BS EN ISO 3696:1995 Water for analytical laboratory use – Specification and test methods

3. Summary of Test Method

3.1 A test specimen consisting of a 40-mL sample and a 40-mL quantity of distilled ~~water (Note 3)~~ water, or 1% sodium chloride (NaCl) solution or synthetic seawater are stirred for 5 min in a graduated cylinder at 54°C ~~(Note 1) in a graduated cylinder.~~or 82°C,

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* ~~, Vol 05.01.~~volume information, refer to the standard's Document Summary page on the ASTM website.

~~[3] Annual Book of ASTM Standards, Vol 11.02.~~

[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

***A Summary of Changes section appears at the end of this standard.**

depending upon the viscosity of the test specimen or sample specification. The time required for the separation of the emulsion thus formed is recorded either after every 5 min or at the specification time limit. If complete separation or emulsion reduction to 3 mL or less does not occur after standing for 30 min or some other specification time limit, the volumes of oil (or fluid), water, and emulsion remaining at the time are reported.

4. Significance and Use

4.1 This test method provides a guide for determining the water separation characteristics of oils subject to water contamination and turbulence. It is used for specification of new oils and monitoring of in-service oils.

5. Apparatus

5.1 *Cylinder*, 100-mL, graduated from 5 to 100 mL in 1.0-mL divisions, made of glass, heat-resistant glass, like borosilicate glass, or a chemical equivalent. The inside diameter shall be no less than 27 mm and no more than 30 mm throughout its length, measured from the top to a point 6 mm from the bottom of the cylinder. The overall height of the cylinder shall be 225 to 260 mm. The graduation shall not be in error by more than 1 mL at any point on the scale.

5.2 *Heating Bath*, ~~sufficiently large and deep to permit the immersion of at least two test cylinders in the bath liquid up to their 85-mL graduations. The bath shall be capable of being maintained at a temperature of 54 ± 1°C (Note 1), and shall be fitted with clamps which hold the cylinder in a position so that the longitudinal axis of the paddle corresponds to the vertical center line of the cylinder during the stirring operation. The clamps shall hold the cylinder securely while its contents are being stirred.~~ , sufficiently large and deep to permit the immersion of at least two test cylinders in the bath liquid up to their 85-mL graduations. The bath shall be capable of being maintained at a test temperature to within ±1°C. The cylinder shall be secured in a position so that the longitudinal axis of the paddle corresponds to the vertical center line of the cylinder during the stirring operation. It is recommended that the bath be constructed with at least one transparent side that allows for clear visual inspection of the oil (fluid), water, and emulsion layer volumes while the cylinder remains immersed in the bath.

5.3 *Stirring Paddle*, made of chromium-plated or stainless steel and conforming to the following dimensions:

~~Length, mm (in.)~~	~~120 ± 1.5 (4¾ ± 1/16)~~
Length, mm	120 ± 1.5
~~Width, mm (in.)~~	~~19 ± 0.5 (¾ ± 1/64)~~
Width, mm	19 ± 0.5
~~Thickness, mm (in.)~~	~~1.5 (1/16)1.5 ± 0.15~~
Thickness, mm	1.5 ± 0.15
Radius of curvature of paddle corners, mm	1.6 max

It is mounted on a vertical shaft of similar metal, approximately 6 mm ~~(¼ in.)~~ in diameter, connected to a drive mechanism which rotates the paddle on its longitudinal axis at 1500 ± 15 rpm. The apparatus is of such design that, when the cylinder is clamped in position and the paddle assembly is lowered into the cylinder, a positive stop engages and holds the assembly when the lower edge of the paddle is 6 mm from the bottom of the cylinder. During the operation of the stirrer, the center of the bottom edge of the paddle shall not deviate more than 1 mm from the axis of rotation. When not in operation, the paddle assembly can be lifted vertically to clear the top of the graduated cylinder. (**Warning—**Paddle edges may be very sharp. Handle with care.) (**Warning—**A protective shield may be used to cover the rotating shaft of the stirrer.)

5.4 *Glass Rod (Policeman)*, covered with a material such as rubber, resistant to the oil or fluid.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] 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.

6.2 ~~*Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water as defined by Type II in Specification D1193~~Reagent Water—Unless otherwise indicated, reference to water shall be understood to mean distilled, deionized water as defined by Type I or Type II in Specification D 1193 or Grade 3 in BS EN ISO 3696:1995.

6.3~~*Cleaning Solvents, Light-Hydrocarbon* , such as precipitation naphtha (**Warning**—Health hazard) for petroleum oils. Use other appropriate solvents for dissolving synthetic fluids.~~

6.4 ~~*Acetone*, (**Warning**—Health hazard, Flammable.)~~

6.5 ~~*Cleaning Reagent*, Cleaning by either hot NOCHROMIX (**Warning** —Corrosive. Health hazard oxidizer), or a 24-h soak at room temperature in MICRO solution gave acceptable, statistically equivalent results in round-robin testing.~~

6.3 *Acetone*, (**Warning—** Health hazard, Flammable.)

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