Designation: D1401 – 12

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

This standard is issued under the fixed designation D1401; 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*

1.1 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 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 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 may also be used. A1% 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 warning statements, see Section 6.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D665 Test Method for Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water
D1141 Practice for the Preparation of Substitute Ocean Water
D1193 Specification for Reagent Water
D2711 Test Method for Demulsibility Characteristics of Lubricating Oils
D4057 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, or 1% sodium chloride (NaCl) solution or synthetic seawater are stirred for 5 min in a graduated cylinder at 54°C or 82°C, 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

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.C0.02 on Corrosion and Water/Air Separability.

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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* volume information, refer to the standard's Document Summary page on the ASTM website.

[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**

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 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	120 ± 1.5
Width, mm	19 ± 0.5
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 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 *Spatula or Wiper,* with or without inert rod support, composed of a material such as rubber, that is 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 *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 D1193 or Grade 3 in BS EN ISO 3696:1995.

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

6.4 *Toluene,* (**Warning—** Flammable.) Solvents with similar cleaning and solvency characteristics may be substituted for toluene.

6.5 *Detergent,* free rinsing, anionic detergent with a pH of 9.5 to 11.

7. Sampling

7.1 The test is very sensitive to small amounts of contamination. Take samples in accordance with Practice D4057.

8. Preparation of Apparatus

8.1 Clean the graduated cylinder by removing any film of oil (or fluid) with a thorough rinsing with toluene or solvent with similar cleaning and solvency characteristics followed by a wash first with acetone and then with tap water. Clean the glassware with a suitable detergent (6.5). Rinse with tap water. Soak the cylinder in the detergent for at least 24 h. Rinse thoroughly with tap water and then with reagent water.

8.1.1 Check the glassware for cleanliness by adding reagent water to the cylinder and inverting it to drain the water. If the water drains with a smooth sheeting action and leaves behind no droplets, the glassware is clean for use. An additional soak in a mildly acidic solution may be necessary.

NOTE 1—Alternately, new glassware may be used, provided that they are thoroughly rinsed with reagent water and meet the cleanliness defined by the sheeting action as described in 8.1.1.

8.2 Clean the stirring paddle and shaft with absorbent cotton or tissue wet with toluene or solvent with similar cleaning and solvency characteristics and air dry. Care shall be taken not to bend or misalign the paddle assembly during the cleaning operation.

9. Procedure

9.1 Heat the bath liquid to 54 ± 1°C, 82 ± 1°C or specified test temperature and maintain it at that temperature throughout the test. Add reagent water to the graduated cylinder to reach the 40-mL mark when at test temperature. Typically 39.5 mL of water at room temperature will expand to the 40-mL mark once the cylinder is placed in the bath at 54°C; 39 mL if heating the sample to 82°C. Invert the sample several times in the original container. Do not pour, shake, or stir samples to any greater extent than necessary to prevent air entrainment. Pour the oil (or fluid) under test into the same cylinder until the top level of the oil reaches the 80-mL mark on the cylinder when at test temperature.

9.2 If initial volumetric measurements are made at room temperature, expansion occurring at the elevated test temperature will have to be considered. For example, there will be a total volumetric expansion of about 2 to 3 mL at 82°C. Corrections to each volume reading at 82°C, therefore, should be made so that the total of the volume readings made for oils (or fluid), water, and emulsion does not exceed 80 mL. An alternative procedure which would avoid the corrections is to make the initial volumetric measurements at the test temperature.

[4] *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.