Designation: ~~D1401 – 19~~ D1401 – 21

Standard Test 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 U.S. 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 mm^2/s to 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 °C ± 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. 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see Section 7.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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 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
D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.C0 on Turbine Oils.

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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.

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

2.2 *ISO Standards:*[3]
BS EN ISO 3696:1995 Water for analytical laboratory use—Specification and test methods

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, see Terminology D4175.

3.1.2 *emulsion, n*—a suspension of fine globules of one or more liquids in another liquid.

3.1.3 *interface, n*—a common boundary between different phases of a substance.

3.1.4 *phase separation, n*—the formation of two layers, an aqueous constituent and a hydrophobic constituent, separated by either a common boundary or a layer of emulsion; typically, the aqueous portion will be the lower phase, but for certain fluids whose relative densities are greater than that of water, the aqueous portion will be the upper phase.

4. Summary of Test Method

4.1 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.

5. Significance and Use

5.1 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.

6. Apparatus

6.1 *Cylinder,* 100 mL, graduated in 1.0 mL divisions (divisions starting at 1.0 mL or 5.0 mL), 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 mm to 260 mm. The graduation shall not be in error by more than 1 mL at any point on the scale.

6.2 *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.

6.3 *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 rpm ± 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

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[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.

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.)

NOTE 1—Soldering material used to join the stirrer and paddle may deteriorate or corrode over time and lead to additional emulsion formation (which may be very stable and impact quality). Careful inspection of the union or use of a stirring paddle and shaft made of a single piece of metal is recommended.

6.4 *Spatula or Wiper,* with or without inert rod support, composed of a material such as rubber, that is resistant to the oil or fluid.

7. Reagents

7.1 *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.

7.2 *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.

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

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

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

8. Sampling

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

9. Preparation of Apparatus

9.1 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 (7.5). 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.

9.1.1 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 2—Alternatively, 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 9.1.1.

9.2 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.

10. Procedure

10.1 Heat the bath liquid to 54 °C ± 1 °C, 82 °C ± 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

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[4] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar 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.

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.

10.2 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 mL 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 that would avoid the corrections is to make the initial volumetric measurements at the test temperature. Oils with high surface tension can create a highly curved meniscus with the reagent water resulting in a reagent water level falling below the 40 mL line after the oil is added. For those types of oils, the original reading of 40 mL (without the addition of the oil) is valid and shall be used. Do not adjust the reagent water volume.

10.3 A 1 % sodium chloride (NaCl) solution or synthetic seawater, as described in Practice D1141 or Test Method D665, may be used in place of distilled water when testing certain oils or fuels used in marine applications.

10.4 Secure the cylinder in place directly under the stirring paddle. Lower the paddle into the cylinder until the stop engages at the required depth. Start the stirrer and a stop watch simultaneously and adjust the stirrer, as required, to a speed of 1500 rpm ± 15 rpm. At the end of 5 min, stop the stirrer and raise the stirring assembly until it is just clear of the graduate. Wipe the paddle with a spatula or wiper, allowing the liquid thus removed to drop back into the cylinder. At 5 min intervals, or at the specification time limit identified for the product being tested, depending on the type of heating bath utilized, lift the cylinder out of the bath or inspect the sample through the glass panel of the heating bath, and record the volumes of the oil (or fluid), water, and emulsion layers. If necessary, additional lighting, such as a backlight or an indoor flood light, may be used to aid the analyst in the inspection of the sample.

10.5 Several samples may be placed in the bath at the beginning of the first analysis. While the first sample is being observed, additional samples may be stirred. At no time during a sample inspection shall an additional sample be added to the heating bath.

11. Report

11.1 *Record Measurements at 5 min Intervals*—Report the following information:
(1) Type of water used.
(2) Test temperature.
(3) Time, in minutes, to reach 3 mL or less of emulsion.
(4) Time, in minutes, to reach 37 mL of water.
(5) Time, in minutes, to reach a complete break of 0 mL emulsion, 40 mL of oil, and 40 mL of water.

11.1.1 In addition to reporting the time, report the volume of each layer in millilitres. In all cases, report results as follows:

$$\text{mL oil} - \text{mL water} - \text{mL emulsion}\ (\text{time,min}) \tag{1}$$

The test may be aborted after 30 min when testing at 54 °C and 60 min at 82 °C.

11.1.2 When the test method is performed to determine if the sample meets a specification, report the test temperature. Report the time when either:
(1) The product passes the product separability requirements against which it is being tested, or
(2) The test limit for water separability is exceeded (usually 3 mL emulsion or less for 30 min at 54 °C and 60 min at 82 °C).

In addition to reporting the time, report the volume of each layer in millilitres. In both cases, report results as follows:

$$\text{mL oil} - \text{mL water} - \text{mL emulsion}\ (\text{time,min}) \tag{2}$$

11.1.3 Some samples can produce only two layers. In situations where the aqueous layer is ≥37 mL, report the second layer as the oil layer. However, if the volume of the aqueous layer is <37 mL, then the volume of the second layer is >43 mL and is to be reported as emulsion.

11.1.4 For results where the middle layer has an ill-defined interface but the aqueous layer shows ≥37 mL of water separated, report a volume of emulsion equal to the missing volume of water. If the volume of the aqueous layer <37 mL, ill-defined interfaces should be established at the largest volume of emulsion.

11.2 For uniformity, test results may be reported in the manner shown in the examples provided in 11.1.

40-40-0 (20)	Complete separation occurred in 20 min. More than 3 mL of emulsion had remained at 15 min.
39-38-3 (20)	Complete separation had not occurred, but the emulsion reduced to 3 mL so the test was ended.
39-35-6 (60)	More than 3 mL of emulsion remained after 60 min—39 mL of oil, 35 mL of water, and 6 mL of emulsion.
41-37-2 (20)	Complete separation had not occurred but the *emulsion layer* reduced to 3 mL or less after 20 min.
43-37-0 (30)	The *emulsion layer* reduced to 3 mL or less after 30 min. The *emulsion layer* at 25 min exceeded 3 mL, for example, 0-36-44 or 43-33-4.

11.3 The appearance of each layer may be described in the following terms:

11.3.1 *Oil (or Oil Rich) Layer:*

11.3.1.1 Clear.

11.3.1.2 Hazy (Note 3).

11.3.1.3 Cloudy (or milky) (Note 3).

11.3.2 *Water or Water-Rich Layer:*

11.3.2.1 Clear.

11.3.2.2 Lacy or droplets present, or both.

11.3.2.3 Hazy (Note 3).

11.3.2.4 Cloudy (or milky) (Note 3).

11.3.3 *Emulsion:*

11.3.3.1 Loose and lacy (Note 4).

11.3.3.2 Cloudy (or milky) (Note 5).

11.3.3.3 Creamy (like mayonnaise) (Note 5).

11.3.3.4 Combinations of 11.3.3.1 – 11.3.3.3.

NOTE 3—A hazy layer is defined as being translucent and a cloudy layer opaque.

NOTE 4—A loose or lacy layer can be defined as having large globules that are not tightly spaced.

NOTE 5—The principal difference between cloudy and creamy emulsions is that the cloudy emulsion is quite fluid and probably unstable while the creamy emulsion has a thick consistency and is probably stable. A cloudy emulsion will readily flow from an inclined graduate while a creamy emulsion will not.

11.4 The appearance of the oil/emulsion and water/emulsion interfaces may be described in the following terms:

11.4.1 Well-defined, sharp.

11.4.2 Ill-defined, droplets.