Designation: D1401 – 12$^{\varepsilon 1}$

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.

ε^1 NOTE—Subsection 11.1 was corrected editorially in October 2015.

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. 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.

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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.C0.02 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**

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 mL 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 mm 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 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 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 °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

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[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.

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 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 which would avoid the corrections is to make the initial volumetric measurements at the test temperature.

9.3 A1 % 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.

9.4 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.

9.5 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.

10. Report

10.1 *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.

10.1.1 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.

10.1.2 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:

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

10.1.3 Some samples may produce a hazy oil layer without an emulsion layer. In situations where the water layer is ≥37 mL, report the upper layer as the oil layer. If there are two layers, and the water layer is <37 mL, that is, the upper layer is >43 mL, report the upper layer as the emulsion layer.

10.2 For uniformity, test results may be reported in the manner shown in the examples provided in 10.1.

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.

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

10.3.1 *Oil (or Oil Rich) Layer:*
10.3.1.1 Clear.
10.3.1.2 Hazy (Note 2).
10.3.1.3 Cloudy (or milky) (Note 2).
10.3.1.4 Combinations of 10.3.1.1 – 10.3.1.3.
10.3.2 *Water or Water-Rich Layer:*
10.3.2.1 Clear.
10.3.2.2 Lacy or bubbles present, or both.
10.3.2.3 Hazy (Note 2).
10.3.2.4 Cloudy (or milky) (Note 2).
10.3.2.5 Combinations of 10.3.2.1 – 10.3.2.4.
10.3.3 *Emulsion:*
10.3.3.1 Loose and lacy.
10.3.3.2 Cloudy (or milky) (Note 3).
10.3.3.3 Creamy (like mayonnaise) (Note 3).
10.3.3.4 Combinations of 10.3.3.1 – 10.3.3.3.

NOTE 2—A hazy layer is defined as being translucent and a cloudy layer opaque.

NOTE 3—The principal difference between cloudy and creamy emulsions is that the former is quite fluid and probably unstable while the latter has a thick consistency and is probably stable. A cloudy emulsion will readily flow from an inclined graduate while a creamy emulsion will not.

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

10.4.1 Well-defined, sharp.
10.4.2 Ill-defined, bubbles.
10.4.3 Ill-defined, lace.

10.5 Report the test temperature if other than 54 °C and the aqueous medium if other than distilled water.

11. Precision and Bias[5]

11.1 *Precision*—An interlaboratory study performed in 2008 by 13 laboratories (9 using glass baths and 4 using metal baths). At 54 °C, the following samples were included: Turbine Oil, Mineral Turbine Oil ISO 32, 150 N Base oil (in duplicate), Mineral Circulating Oil ISO 32 , Synthetic Gear Oil ISO 32 , Industrial Oil ISO 68, Industrial Oil ISO 68, Synthetic Compressor ISO 46, Multi-Purpose Oil ISO 32. At 82 °C, the following samples were included: 600 NS Base oil , Synthetic Gear Oil ISO 320 (in duplicate), Synthetic Paper Machine Oil ISO 220, Industrial Oil ISO 100, Hydraulic oil ISO 150, Synthetic Compressor ISO 100, Multi-Purpose Oil ISO 100, Circulating Oil, Circulating Oil. The mineral oil samples were Group I or Group II based. The synthetic samples were primarily PAO based but may have contained some amount of a Group V oil, however no specific details on any co-base oil was disclosed. This precision is for the determination of the time in minutes to 3 mL of emulsion or 37 mL of water.

11.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty.

at 54 °C	15 min
at 82 °C	10 min

11.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

at 54 °C	20 min
at 82 °C	25 min

11.2 *Bias*—The procedure in this test method for measuring water separability has no bias because the value for water separability is defined only in terms of the test method.

12. Keywords

12.1 emulsion; petroleum oils; steam-turbine oils; synthetic fluids; water separability

APPENDIX

(Nonmandatory Information)

X1. OPTIONAL METHOD USING DYE TO DIFFERENTIATE OIL, WATER, AND EMULSION LAYERS

X1.1 Scope

X1.1.1 This optional procedure describes a method to use a dye in Test Method D1401 testing to help differentiate the oil, water, and emulsion layers. The modifications to the most current method follow.

X1.2 Summary of Method

X1.2.1 Optionally, add one to two drops of red oil-soluble dye on top of the oil layer. Continue as per procedure. The oil layer will be red, the emulsion layer will be pink, and the water layer will be clear. Report as per procedure.

X1.3 Apparatus

X1.3.1 *General Purpose Polyethylene or Glass Transfer Pipet with Fine or Regular Sized Tip*—To be used for adding the dye.

X1.4 Reagents

X1.4.1 *Dye*[6]—2.5 % liquid oil-soluble red dye (42 % "C.I. solvent red 164" in 58 % petroleum hydrocarbon) in 97.5 % additive-less 110N diluent oil.

X1.5 Procedure

X1.5.1 Follow the current Test Method D1401 procedure. After the oil addition, optionally add one to two drops (approximately 0.02 g to 0.05 g) of the dye using a general purpose pipet on top of the oil layer to help with differentiating the layers. Continue as per procedure. Please refer to Research Report RR:D02-1745 for the background study on the dye.[7]

X1.6 Report

X1.6.1 If the dye was used, note this in remarks.

X1.7 Precision and Bias

X1.7.1 The red dye was *not* used in the ILS for the precision statement detailed in RR:D02-1665.[5]

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1665.

[6] The sole source of supply of the pre-blended dye known to the committee at this time is trademarked and coded UNISOL Liquid RED 2640, United Color Manufacturing, Inc., 660 Newtown Yardley Rd., # 205, Newtown, PA 18940, www.unitedcolor.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1745.

SUMMARY OF CHANGES

Subcommittee D02.C0.02 has identified the location of selected changes to this standard since the last issue (D1401 – 10) that may impact the use of this standard.

(1) Added Appendix X1.

(2) Added new research report.

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