



# Standard Test Method for Water Separability of Petroleum Oils and Synthetic Fluids<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee ~~D-2~~ 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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## 1. Scope

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

NOTE 1—Although developed specifically for steam-turbine oils having viscosities of 28.8–90 cSt ( $\text{mm}^2/\text{s}$ ) at 40°C, this test method can be used to test oils of other types having various viscosities and synthetic fluids. It is recommended, however, that the test temperature be raised to  $82 \pm 1^\circ\text{C}$  when testing products more viscous than 90 cSt ( $\text{mm}^2/\text{s}$ ) 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 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.

1.2 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.* For specific precautionary statements, see ~~6.3, 8.1,~~ 6.3-6.5.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 665 Test Method for Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water<sup>2</sup>

~~D 1141 Specification 1141 Practice for the Preparation of Substitute Ocean Water<sup>3</sup>~~

D 1193 Specification for Reagent Water<sup>4</sup>

D 2711 Test Method for Demulsibility Characteristics of Lubricating Oils<sup>2</sup>

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>5</sup>

## 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) are stirred for 5 min at 54°C (Note 1) in a graduated cylinder. 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.

<sup>2</sup> Annual Book of ASTM Standards, Vol 05.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 11.02.

<sup>4</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>5</sup> Annual Book of ASTM Standards, Vol 05.02.

## 5. Apparatus

5.1 *Cylinder*, 100-mL, graduated from 5 to 100 mL in 1.0-mL divisions, made of glass, heat-resistant glass,<sup>6</sup> 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  $54 \pm 1^\circ\text{C}$  (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.

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

Length, mm (in.)	$120 \pm 1.5$ ( $4\frac{3}{4} \pm \frac{1}{16}$ )
Width, mm (in.)	$19 \pm 0.5$ ( $\frac{3}{4} \pm \frac{1}{64}$ )
Thickness, mm (in.)	$1.5$ ( $\frac{1}{16}$ )

It is mounted on a vertical shaft of similar metal, approximately 6 mm ( $\frac{1}{4}$  in.) in diameter, connected to a drive mechanism which rotates the paddle on its longitudinal axis at  $1500 \pm 15$  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.

## 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.<sup>7</sup> 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 D 1193.

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<sup>8</sup> (**Warning**—Corrosive. Health hazard- $\Theta$  oxidizer), or a 24-hr soak at room temperature in MICRO<sup>9</sup> solution gave acceptable, statistically equivalent results in round-robin testing.

## 7. Sampling

7.1 The test is very sensitive to small amounts of contamination. Take samples in accordance with Practice D 4057.

## 8. Preparation of Apparatus

8.1 Clean the graduated cylinder by removing any film of oil (or fluid) with cleaning solvent followed by a wash first with acetone and then with tap water. The glassware shall be further cleaned with a suitable cleaning reagent. Rinse thoroughly with tap water and then with reagent water. Inspect the cylinders for any residue or water droplets adhering to the inside walls. Both conditions indicate a need for additional cleaning.

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

## 9. Procedure

9.1 Heat the bath liquid to  $54 \pm 1^\circ\text{C}$  (Note 1) and maintain it at that temperature throughout the test. Add reagent water (Note 2, 2 and Note 3) to the graduated cylinder up to the 40-mL mark and then add to the same cylinder a representative sample of the

<sup>6</sup> Borosilicate glass has been found satisfactory for this purpose.

<sup>7</sup> *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 *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.

<sup>8</sup> NOCHROMIX available from

<sup>8</sup> The sole source of supply of NOCHROMIX known to the committee at this time is Godax Laboratories Inc., 720-B Erie Ave., Takoma Park, MD 20912. 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<sup>1</sup>, which you may attend.

<sup>9</sup> MICRO available from

<sup>9</sup> The sole source of supply of MICRO known to the committee at this time is International Products Corp., P.O. Box 70, Burlington, NJ 08016. 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<sup>1</sup>, which you may attend.

oil (or fluid) under test until the top level of the oil reaches the 80-mL mark on the cylinder. Place the cylinder in the bath and allow the contents to reach bath temperature. Heating time may vary with type of equipment and can reach up to 30 min.

NOTE 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.

NOTE 3—A 1 % sodium chloride (NaCl) solution or synthetic sea water, as described in Specification Practice D 1141 or Test Method D 665, can be used in place of distilled water when testing certain oils or fuels used in marine applications.

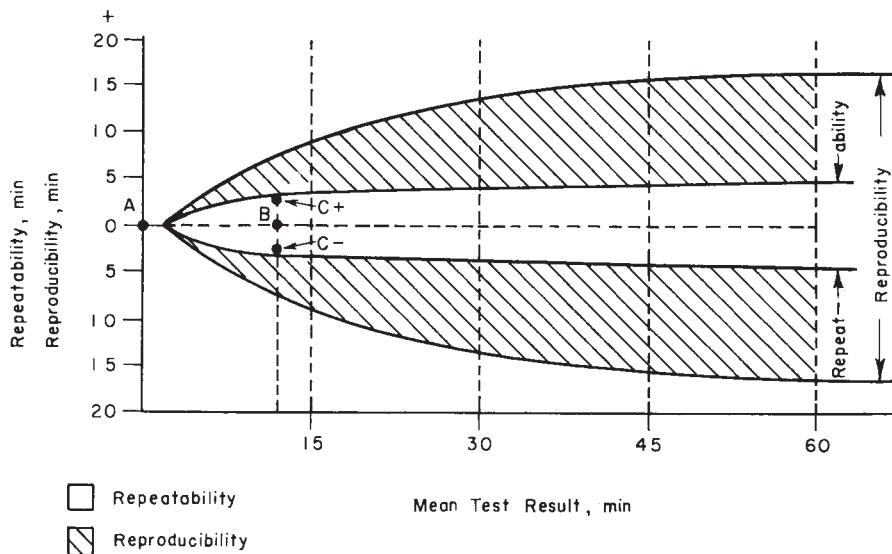
9.2 Clamp the cylinder in place directly under the stirring paddle. Lower the paddle into the cylinder until the stop engages at the required depth. Start the stirrer and a stop watch simultaneously and adjust the stirrer, as required, to a speed of  $1500 \pm 15$  rpm. At the end of 5 min, stop the stirrer and raise the stirring assembly until it is just clear of the graduate. Wipe the paddle with a policeman (Note 4), allowing the liquid thus removed to drop back into the cylinder. Remove the cylinder from the retaining clamps and transfer it carefully to another section of the bath. At 5-min intervals, or at the specification time limit identified for the product being tested, lift the cylinder out of the bath (see Note 5), inspect, and record the volumes of the oil (or fluid), water, and emulsion layers.

NOTE 4—The policeman should be made of material resistant to the oil or fluid.

NOTE 5—It is not necessary to lift the cylinders out of the bath for inspection if the heating bath is 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.

## 10. Report

10.1 Recording Measurements at 5-min Intervals—Record the time (at 5-min intervals) until either (a1) the product passes the water separability requirements it is being tested against, or (b2) 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). The maximum volume to be reported as the oil layer is 43 mL (see Note 5.6). For uniformity, report the test results may be reported in the manner shown in the following examples:



*Use of Chart*—Calculate the mean test result in minutes. Enter chart at the zero point, A, on the ordinate and move to the right on the abscissa to point B. Compute and locate the deviation points C+ and C-, from the mean test result. If the deviation points fall within the repeatability area, then the results are within the precision of the test.

*Example*—An oil has emulsion of 40-40-0 (10 min) and 40-40-0 (15 min). The mean test result is 12.5 min (B) and the deviation from the mean is +2.5 (C+) and -2.5 (C-).

These points fall within the repeatability area.

Use this graph similarly for the reproducibility of means of different laboratories.

**FIG. 1 Chart for Determining Test Precision**

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 <i>emulsion layer</i> reduced to 3 mL or less after 20 min.
43-37-0 (30)	The <i>emulsion layer</i> reduced to 3 mL or less after 30 min. The <i>emulsion layer</i> at 25 min exceeded 3 mL, for example, 0-36-44 or 43-33-4.

10.1.1 Recording Measurements at the Specification Time Requirement Only—Record the volumes for oil (or fluid), water, and emulsion layers at the specification time limit and determine whether (1) the product passes the water separability requirements it is being tested against, 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). The maximum volume to be reported as the *oil layer* is 43 mL (see Note 6). For uniformity, test results may be reported in the manner shown in the examples provided in 10.1.

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

10.2.1 *Oil (or Oil Rich) Layer*:

10.2.1.1 Clear.

10.2.1.2 Hazy (~~Note 6~~-7).

10.2.1.3 Cloudy (or milky) (~~Note 6~~-7).

10.2.1.4 Combinations of 10.2.1.1-10.2.1.3.

10.2.2 *Water or Water-Rich Layer*:

10.2.2.1 Clear.

10.2.2.2 Lacy or bubbles present, or both.

10.2.2.3 Hazy (~~Note 6~~-7).

10.2.2.4 Cloudy (or milky) (~~Note 6~~-7).

10.2.2.5 Combinations of 10.2.2.1-10.2.2.4.

10.2.3 *Emulsion*:

10.2.3.1 Loose and lacy.

10.2.3.2 Cloudy (or milky) (~~Note 7~~-8).

10.2.3.3 Creamy (like mayonnaise) (~~Note 7~~-8).

10.2.3.4 Combinations of 10.2.3.1-10.2.3.3.

**NOTE 56**—Certain oils may produce a hazy oil layer. In situations where the measurement of the oil and water layer indicates essentially complete separation, the upper layer should be reported as oil. If there are two layers and if the upper layer is more than 43 mL, this layer should be considered the emulsion layer.

**NOTE 67**—A hazy layer is defined as being translucent and a cloudy layer opaque.

**NOTE 78**—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.

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

10.3.1 Well-defined, sharp.

10.3.2 Ill-defined, bubbles.

10.3.3 Ill-defined, lace.

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

## 11. Precision and Bias

11.1 *Precision*—The precision of this test method was obtained on steam-turbine oils having viscosities of 28.8 to 90 cSt (mm<sup>2</sup>/s) at 40°C and using time to 3 mL or less emulsion as the test completion. This precision is expressed graphically in Fig. 1. The graph shows the maximum allowable deviation in minutes for repeatability and reproducibility (95 % confidence) from mean emulsion test results for these oils. It may not be applicable to other oils or fluids.

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

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