



Standard Test Method for Stability of Water-in-Oil Emulsions Under Low to Ambient Temperature Cycling Conditions¹

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1. Scope

1.1 This test method covers the indication of the stability of water-in-oil emulsions when subjected to temperature cycling changes between ambient temperature and -18°C (0°F).

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.*

2. Referenced Documents

2.1 ASTM Standards:

D7144 Practice for Collection of Surface Dust by Microvacuum Sampling for Subsequent Metals Determination²

3. Summary of Test Method

3.1 A 100-mL sample contained in a graduated 100-mL cylinder is placed in a cooling box at -18°C (0°F) for 16 h and then allowed to stand at room temperature for 8 h. This cycling procedure is repeated to give a total of nine cycles, except that the fifth cycle is one of 64 h at -18°C (0°F), 8 h at room temperature. At the completion of the cycling procedure the sample is examined for the amount of free oil and free water separated. In addition, water contents of the sample at specified levels in the upper and lower layers of the sample are also obtained.

4. Significance and Use

4.1 This test method indicates the ability of the emulsion to withstand mild to moderately severe winter conditions of use

and storage. Generally this significance would be limited to conditions where the emulsion reaches a minimum temperature of -18°C (0°F).

5. Apparatus

5.1 *Cold Box*—A thermostatically controlled freezer or cold box capable of being maintained at $-18 \pm 1.5^{\circ}\text{C}$ ($0 \pm 3^{\circ}\text{F}$).

5.2 *Graduated Cylinder*—A stoppered 100-mL glass cylinder graduated in 1.0-mL increments. The stopper should have a vent groove to prevent vacuum or pressure build-up during the test.

5.3 *Pipet*, 10-mL glass.

5.4 *Microsyringe*, 0.05-mL glass with a fixed needle, No. 19 gage, point style No. 3.

5.5 *Glass Vials*, approximately 30-mL size.

6. Preparation of Sample

6.1 To ensure uniformity of the sample, it should be mixed thoroughly before removing the quantity required for the test. Vigorous hand shaking or mechanical mixing for 3 to 5 min is recommended for litre-size containers or less.

6.2 Special difficulties arise in mixing and withdrawing representative samples from large containers. This is due to the nature of water-in-oil emulsions that are two-phase systems as distinct from truly homogeneous systems. Vigorous mechanical stirring for an extended period is the best means of achieving homogeneity. However, for unstable emulsions that have been in storage for some time there is no satisfactory way to obtain a representative sample.

7. Procedure

7.1 After careful preparation of the sample to ensure homogeneity (described in Section 6), transfer 100 mL to a graduated 100-mL cylinder. Insert the vented stopper.

7.2 Place the cylinder in the cold box at $-18 \pm 1.5^{\circ}\text{C}$ ($0 \pm 3^{\circ}\text{F}$) for a period of 16 h. Withdraw the cylinder from the cold box and allow to stand at room temperature, $21 \pm 3^{\circ}\text{C}$ ($70 \pm 5^{\circ}\text{F}$), for a period of 8 h.

7.3 Repeat the cycling procedure as in 7.2 for three more cycles.

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.N0.02 on Industrial Applications.

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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.