Designation: D 3707 – 89 (Reapproved 1999)

An American National Standard

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Storage Stability of Water-in-Oil Emulsions by the Oven Test Method[1]

This standard is issued under the fixed designation D 3707; 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.

1. Scope

1.1 This test method covers the stability of water-in-oil emulsions when held at a constant elevated temperature.

1.2 *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.*

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1744 Test Method for Water in Liquid Petroleum Products by Karl Fischer Reagent[2]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[3]

3. Summary of Test Method

3.1 A 100-mL sample contained in a graduated 100-mL cylinder is placed in a thermostatically controlled oven at 85°C (185°F) for 48 or 96 h. The sample is then 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 stability of the emulsion during storage and normal usage.

5. Apparatus

5.1 *Convection Oven*, meeting the requirements of Specification E 145.

5.2 *Graduated Cylinder*—A stoppered 100-mL glass cylinder graduated in 1-mL increments.[4] The stopper should have a vent groove to prevent pressure buildup 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 which 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 A—48-h Test

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

7.2 Place the cylinder in a convection oven maintained at 85 ± 1°C (185 ± 2°F) for a period of 48 h. Take care to position the graduated cylinder in the oven generally in the center and raise at least 75 mm from the bottom of the oven, to ensure temperature uniformity.

NOTE 1—When carrying out a number of tests at the same time, arrange the cylinders to avoid developing temperature variations in the oven due to inadequate convection. The number of tests carried out at the same time should, for the same reason, also be restricted.

7.3 Withdraw the cylinder from the oven and allow to stand at room temperature, 21 ± 3°C (70 ± 5°F), for a period of 1 h.

7.4 Observe and record:

7.4.1 The amount of oil separated, percent volume and

7.4.2 The amount of water separated, percent volume.

7.5 By means of a 10-mL pipet, withdraw aliquots in the following order:

7.5.1 With the pipet tip located exactly at the *80*-mL mark, slowly withdraw a 10-mL sample and transfer to a small glass vial. This is designated as the *upper layer* sample.

7.5.2 With the pipet tip located exactly at the *15*-mL mark,

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[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.N0.02 on Industrial Applications.

Current edition approved Oct. 27, 1989. Published December 1989. Originally published as D 3707 – 78. Last previous edition D 3707 – 83.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] A "crows-foot" cylinder, British Standard BS 658, may be used.

slowly withdraw a 10-mL sample and transfer to a small glass vial. This is designated as the *lower layer* sample.

NOTE 2—In the event that the amount of separated water equals or exceeds 10 % by volume then the determination of the water content of the *lower layer* is an optional procedure.

7.6 Determine the water content, as percent weight, of the upper layer and lower layer samples, after shaking, by the procedure given in the Annex. Use a 0.05-mL sample transferred by means of a glass microsyringe.

8. Procedure B—96-h Test

8.1 This procedure is identical to that described under Section 7 except that the time period in the convection oven (7.2) is changed from 48 h to 96 h.

9. Report

9.1 Since the test can be performed on the basis of 48 h in the oven (Procedure A) or 96 h (Procedure B), the report is to indicate the procedure followed.

9.2 Report the results as follows:

9.2.1 Oil separation, percent volume,

9.2.2 Water separation, percent volume,

9.2.3 Upper layer water content, percent weight, and

9.2.4 Lower layer water content, percent weight.

10. Precision and Bias [5]

10.1 The precision (Note 3) of this test method is dependent on the degree of stability of the emulsion, as follows:

10.1.1 *Type I—Stable Emulsions (Procedures A and B)*:

10.1.1.1 The precision of the test method as determined by the statistical examination of interlaboratory test results is as follows.

10.1.1.2 *Repeatability*—The difference between successive 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. (See Table 1.)

10.1.1.3 *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, exceed the following values only in one case in twenty. (See Table 1.)

NOTE 3—Precision limits are based on a round-robin test program carried out in Technical Division N in 1977 using invert emulsion samples with water contents between 35 and 50 % (weight). Eleven cooperators tested five samples representing highly stable, borderline, and unstable emulsions.

10.1.2 *Type II—Highly Unstable Emulsions*—The precision limits for the 96-h test are of the same order as for Type I. The 48-h test would be less severe and less precise.

10.1.3 *Type III—Emulsions of Borderline Stability*—No precision limits can be set since the change in the sample stability with time or conditions, or both, of handling make it impractical to determine meaningful precision limits.

10.2 *Bias*—No bias statement is possible because there is no absolute value. The results are interpretable only with respect to this test.

11. Keywords

11.1 emulsions; water separation; oil separation

TABLE 1 Repeatability and Reproducibility

	Free Oil, %	Free Water, %	Difference in % Water Content Upper Layer Versus Lower Layer
Repeatability	1	1	10
Reproducibility	3	1	14

[5] The results of the cooperative test program, from which these values have been derived, are available from ASTM Headquarters. Request RR:D02-1158.

ANNEX

(Mandatory Information)

A1. MODIFIED TEST METHOD D1744 (KARL FISCHER)

A1.1 Scope

A1.1.1 This test method covers the determination of water in the concentration from 50 to 1000 ppm in liquid petroleum products.

A1.2 Significance and Use

A1.2.1 Knowledge of the water content of petroleum products can be useful to predict quality and performance characteristics of the product.

A1.3 Modified Procedure

A1.3.1 To obtain weight of sample, weigh the syringe to 0.1 mg before and after introducing the sample to the titration vessel.

A1.3.2 Use a 1 part xylene to 2 parts methanol mix as the solvent instead of a 3 parts chloroform to 1 part methanol mix.

NOTE A1.1—**Warning:** Flammable. Harmful if inhaled. Skin irritant on repeated contact. Eye irritant. Aspiration hazard.

A1.4 Precision and Bias

A1.4.1 The precision of Test Method D 1744 with this solvent system is now under study by Subcommittee D.02.03.

A1.4.2 No bias statement is possible because there is no absolute value. The results are interpretable only with respect to this test.