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Standard Test Method for Water in Petroleum Products and Bituminous Materials by Distillation¹

This standard is issued under the fixed designation D 95; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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

^{e1} NOTE—Footnote 1 and the Referenced Documents were corrected editorially to comply with the ASTM-API joint standard contract in October 2002. Warning notes were editorially moved into the standard text in October 2002.

1. Scope

1.1 This test method covers the determination of water in the range from 0 to 25 % volume in petroleum products, tars, and other bituminous materials by the distillation method.

NOTE 1—Volatile water-soluble material, if present, may be measured as water.

1.2 The specific products considered during the development of this test method are listed in Table 1. For bituminous emulsions refer to Test Method D 244. For crude oils, refer to Test Method D 4006 (API MPMS Chapter 10.2).

NOTE 2—With some types of oil, satisfactory results may be obtained from Test Method D 1796 (API MPMS Chapter 10.6).

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

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 hazard statements, see Section 5.

2. Referenced Documents

2.1 ASTM Standards:

D 244 Test Methods for Emulsified Asphalts²

D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure) (API MPMS Chapter 10.6)³

D 4006 Test Method for Water in Crude Oil by Distillation (API MPMS Chapter 10.2)⁴

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products (API MPMS Chapter 8.1)⁴

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products (API MPMS Chapter 8.2)⁴

D 5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (API MPMS Chapter 8.3)⁵

E 123 Specification for Apparatus for Determination of Water by Distillation⁶

2.2 API Standards:⁷

MPMS Chapter 8.1 Manual Sampling of Petroleum and Petroleum Products (ASTM Practice D 4057)

MPMS Chapter 8.2 Automatic Sampling of Petroleum and Petroleum Products (ASTM Practice D 4177)

MPMS Chapter 8.3 Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products (ASTM Practice D 5854)

MPMS Chapter 10.2 Determination of Water in Crude Oil by the Distillation Method (ASTM Test Method D 4006)

MPMS Chapter 10.6 Test Method for Water and Sediment in

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and the API Committee on Petroleum Measurement, and is the direct responsibility of Subcommittee D02.02/COMQ, the joint ASTM-API Committee on Static Petroleum Measurement.

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² Annual Book of ASTM Standards, Vol 04.03.

³ Annual Book of ASTM Standards, Vol 05.01.

⁴ Annual Book of ASTM Standards, Vol 05.02.

⁵ Annual Book of ASTM Standards, Vol 05.03.

⁶ Annual Book of ASTM Standards, Vol 14.02.

⁷ Published as Manual of Petroleum Measurement Standards. Available from the American Petroleum Institute (API), 1220 L St., NW, Washington, DC 20005.

Fuel Oils by the Centrifuge Method (Laboratory Procedure) (ASTM Test Method D 1796)

3. Summary of Test Method

3.1 The material to be tested is heated under reflux with a water-immiscible solvent, which co-distills with the water in the sample. Condensed solvent and water are continuously separated in a trap, the water settling in the graduated section of the trap and the solvent returning to the still.

4. Significance and Use

4.1 A knowledge of the water content of petroleum products is important in the refining, purchase, sale, and transfer of products.

4.2 The amount of water as determined by this test method (to the nearest 0.05 volume %) may be used to correct the volume involved in the custody transfer of petroleum products and bituminous materials.

4.3 The allowable amount of water may be specified in contracts.

5. Solvent-Carrier Liquid

5.1 A solvent-carrier liquid appropriate to the material being tested (see Table 1) shall be used.

5.1.1 *Aromatic Solvent*—The following aromatic solvents are acceptable:

5.1.1.1 *Industrial Grade Xylene*—(Warning—Flammable. Vapor harmful.)

5.1.1.2 A blend of 20 volume % industrial grade toluene and 80 volume % industrial grade xylene. (Warning—Flammable. Vapor harmful.)

5.1.1.3 *Petroleum or Coal Tar Naphtha*, free of water, yielding not more than 5% distillates at 125°C (257°F) and not less than 20% at 160°C (320°F) and with a relative density (specific gravity) not lower than 0.8545 at 15.56/15.56°C (60/60°F). (Warning—Extremely flammable. Harmful if inhaled. Vapors may cause fire.)

5.1.2 *Petroleum Distillate Solvent*—A petroleum distillate solvent, 5% boiling between 90 and 100°C (194 and 212°F) and 90% distilling below 210°C (410°F), shall be used. Percent may be determined by mass or by volume. These solvents are available from most chemical companies under the name of stoddard solvent or ligroine. (Warning—Flammable. Vapor harmful.)

5.1.3 *Volatile Spirits Solvent*—The following volatile spirits solvents are acceptable:

5.1.3.1 *Petroleum Spirit*, with a boiling range from 100 to 120°C (212 to 248°F). (Warning—Flammable. Vapor harmful.)

TABLE 1 Type of Solvent-Carrier Liquid Versus Material to Be Tested

Type of Solvent-Carrier Liquid	Material to be Tested
Aromatic	asphalt, tar, coal tar, water gas tar, road tar, cut-back bitumin, liquid asphalt, tar acid
Petroleum distillate	road oil, fuel oil, lubricating oil, petroleum sulfonates
Volatile spirits	lubricating grease

5.1.3.2 *Iso-octane*, of 95% purity or better. (Warning—Extremely flammable. Harmful if inhaled. Vapors may cause fire.)

5.2 *Solvent Blank*—The water content of the solvent shall be determined by distilling an equivalent amount of the same solvent used for the test sample in the distillation apparatus and testing as outlined in Section 9. The blank shall be determined to the nearest scale division and used to correct the volume of water in the trap in Section 10.

6. Apparatus

6.1 *General*—The apparatus comprises a glass or metal still, a heater, a reflux condenser, and a graduated glass trap. The still, trap, and condenser may be connected by any suitable method that produces a leakproof joint. Preferred connections are ground joints for glass and O-rings for metal to glass. Typical assemblies are illustrated in Fig. 1, Fig. 2, and Fig. 3. The stills and traps should be chosen to cover the range of materials and water contents expected. On assembly, care should be taken to prevent the joints from freezing or sticking. This may be prevented by the application of a very thin film of stopcock grease.

6.2 *Still*—A glass or metal vessel with a short neck and suitable joint for accommodating the reflux tube of the trap shall be used. Vessels of 500, 1000, and 2000-mL nominal capacity have proved satisfactory.

6.3 *Heater*—A suitable gas burner or electric heater may be used with the glass still. A gas ring burner with ports on the inside circumference shall be used with the metal still. The gas ring burner shall be of such dimensions that it may be moved up and down the vessel when testing materials that are likely to foam or solidify in the still.

6.4 *Glassware*—Dimensions and descriptions of typical glassware for use in this test method are provided in Specification E 123.

NOTE 3—Instead of standardizing on a particular apparatus specification with respect to dimensions and style, a given apparatus will be deemed satisfactory when accurate results are obtained by the standard

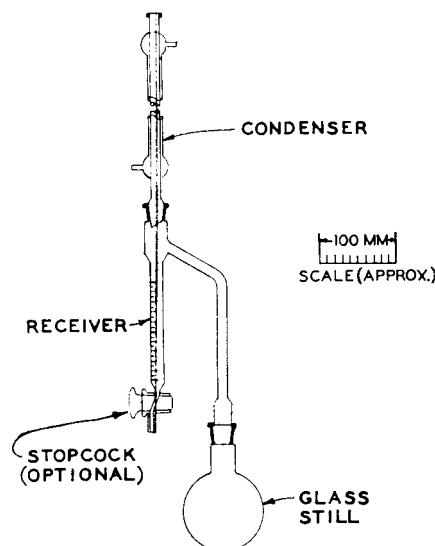


FIG. 1 Typical Assembly with Glass Still