



Designation: D4006 – 22



Manual of Petroleum Measurement Standards (MPMS), Chapter 10.2

Standard Test Method for Water in Crude Oil by Distillation¹

This standard is issued under the fixed designation D4006; 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.

1. Scope*

1.1 This test method covers the determination of water in crude oil by distillation.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see 7.1 and A1.1.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation (API MPMS Chapter 10.5)

D473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method (API MPMS Chapter 10.1)

D665 Test Method for Rust-Preventing Characteristics of

Inhibited Mineral Oil in the Presence of Water

D1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure) (API MPMS Chapter 10.6)

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products (API MPMS Chapter 8.1)

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products (API MPMS Chapter 8.2)

D4928 Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration (API MPMS Chapter 10.9)

E123 Specification for Apparatus for Determination of Water by Distillation

2.2 API Standards:

MPMS Chapter 1 Terms and Definitions Database

MPMS Chapter 8.1 Manual Sampling of Petroleum and Petroleum Products (ASTM Practice D4057)

MPMS Chapter 8.2 Automatic Sampling of Petroleum and Petroleum Products (ASTM Practice D4177)

MPMS Chapter 10.1 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method (ASTM Test Method D473)

MPMS Chapter 10.4 Determination of Water and/or Sediment in Crude Oil by the Centrifuge Method (Field Procedure)

MPMS Chapter 10.5 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation (ASTM Test Method D95)

MPMS Chapter 10.6 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure) (ASTM Test Method D1796)

MPMS Chapter 10.9 Test Method for Water in Crude Oils by Coulometric Karl Fischer Titration (ASTM Test Method D4928)

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175 and the API MPMS Chapter 1 Terms and Definitions Database.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, 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 Hydrocarbon Measurement for Custody Transfer (Joint ASTM-API). This test method has been approved by the sponsoring committees and accepted by the Cooperating Societies in accordance with established procedures.

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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.

*A Summary of Changes section appears at the end of this standard

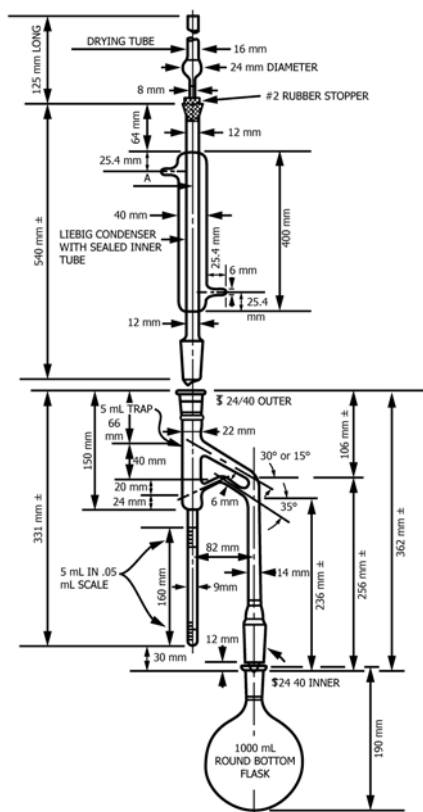


FIG. 1 Distillation Apparatus

4. Summary of Test Method

4.1 The sample is heated under reflux conditions 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 settles in the graduated section of the trap, and the solvent returns to the distillation flask.

5. Significance and Use

5.1 A knowledge of the water content of crude oil is important in the refining, purchase, sale, or transfer of crude oils.

5.2 This test method may not be suitable for crude oils that contain alcohols that are soluble in water. In cases where the impact on the results may be significant, the user is advised to consider using another test method, such as Test Method D4928 (API MPMS Chapter 10.9).

6. Apparatus

6.1 The preferred apparatus, shown in Fig. 1, consists of a glass distillation flask, a condenser, a graduated glass trap, and a heater. Other types of distillation apparatus are specified in Specification E123. Any of these apparatus will be acceptable for this test method provided it can be demonstrated that they operate within the precision established with the preferred apparatus.

6.1.1 *Distillation Flask*—A 1000 mL round-bottom, glass, distillation flask fitted with a 24/40 female taper joint shall be used. This flask receives a 5 mL calibrated, graduated water

trap with 0.05 mL graduations. The trap will be fitted with a 400 mm Liebig condenser. A drying tube filled with desiccant (to prevent entrance of atmospheric moisture) is placed on top of the condenser.

6.1.2 *Heater*—Any suitable gas or electric heater that can uniformly distribute heat to the entire lower half of the flask may be used. An electric heating mantle is preferred for safety reasons.

6.1.3 The apparatus used in this test will be accepted when satisfactory results are obtained by the calibration technique described in Section 9.

7. Solvent

7.1 *Xylene*—reagent grade (**Warning**—Extremely flammable. Vapor harmful. See Annex A1.) A solvent blank will be established by placing 400 mL of solvent in the distillation apparatus and testing as outlined in Section 10. The blank will be determined to the nearest 0.025 mL and used to correct the volume of water in the trap as in Section 11.

7.2 The xylene used in this procedure is generally a mixture of ortho, meta, and para isomers and may contain some ethyl benzene. The typical characteristics for this reagent are:

Color (APHA)	not more than 10
Boiling range	137 °C to 144 °C
Residue after evaporation	0.002 %
Sulfur compounds (as S)	0.003 %
Substances darkened by H ₂ SO ₄	Color pass test
Water (H ₂ O)	0.02 %
Heavy metals (as Pb)	0.1 ppm
Copper (Cu)	0.1 ppm
Iron (Fe)	0.1 ppm
Nickel (Ni)	0.1 ppm
Silver (Ag)	0.1 ppm

8. Sampling, Test Samples, and Test Units

8.1 Sampling is defined as all steps required to obtain an aliquot of the contents of any pipe, tank, or other system and to place the sample into the laboratory test container.

8.1.1 *Laboratory Sample*—Only representative samples obtained as specified in Practice D4057 (API MPMS Chapter 8.1) and Practice D4177 (API MPMS Chapter 8.2) shall be used for this test method.

8.1.2 *Preparation of Test Samples*—The following sample handling procedure shall apply in addition to those covered in 8.1.1.

8.1.2.1 The sample size shall be selected as indicated below based on the expected water content of the sample:

Expected Water Content, weight or volume %	Approximate Sample Size, g or mL
50.1–100.0	5
25.1–50.0	10
10.1–25.0	20
5.1–10.0	50
1.1–5.0	100
0.5–1.0	200
less than 0.5	200

8.1.2.2 If there is any doubt about the uniformity of the mixed sample, determinations should be made on at least three test portions and the average result reported as the water content.

8.1.2.3 To determine water on a volume basis, measure mobile liquids in a 5 mL, 10 mL, 20 mL, 50 mL, 100 mL, or