



Standard Test Method for Determination of Water in Liquid Petroleum Products by Karl Fischer Reagent¹

This standard is issued under the fixed designation D1744; 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 the concentration from 50 to 1000 mg/kg in liquid petroleum products.

1.2 Values stated in SI units are to be regarded as the standard. Inch-pound units are provided for information only.

1.3 *This standard does not purport to address all of the safety problems, 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 Section 7.

2. Referenced Documents

2.1 *ASTM Standards*:²

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

3. Summary of Test Method

3.1 The material to be analyzed is titrated with standard Karl Fischer reagent to an electrometric end point.

4. Significance and Use

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

5. Interferences

5.1 Free alkali, oxidizing and reducing agents, mercaptans, certain basic nitrogenous substances, or other materials that

react with iodine, interfere. One part per million of sulfur as mercaptan causes an error in the titration, equivalent to approximately 0.2 mg/kg of water.

6. Apparatus

6.1 Assemble the apparatus as described in the [Annex A1](#). (See [Note 1](#).)

NOTE 1—Any apparatus that will give equal or better precision is acceptable.

7. Reagents and Materials

7.1 *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.³ Other grades can 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity. The solvents used must have low-water content, for example, less than 500 mg/kg mass.

7.3 *Karl Fischer Reagent, Solution* ([Note 2](#)), *Stock*—(**Warning**—Toxic.) For each litre of solution, dissolve 85 ± 1 g of iodine in 270 ± 2 mL of pyridine in a dry, glass-stoppered bottle. Add 670 ± 2 mL of methanol (99.9 %) ([Note 3](#)). Cool the mixture in an ice bath to below 3.9°C. Bubble gaseous sulfur dioxide (SO₂) ([Note 4](#)) through concentrated sulfuric acid (H₂SO₄, rel dens 1.84) (see warning information below) into the cooled mixture. Continue the addition of SO₂ until the volume is increased 50 ± 1 mL. Alternatively, add 50 ± 1 mL of freshly drawn liquid SO₂ in small increments to the precooled mixture in an ice bath. Mix well and set aside for at

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.02 on Hydrocarbon Measurement for Custody Transfer (Joint ASTM-API).

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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.

³ *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.