



Standard Test Method for Trace Quantities of Water in Aromatic Hydrocarbons and Their Mixtures by Coulometric Karl Fischer Titration¹

This standard is issued under the fixed designation D7375; 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 describes the use of the Coulometric Karl Fischer (KF) titration for the determination of water in aromatic hydrocarbons and their mixtures, derivatives and related chemicals.

1.2 This test method is applicable to samples with water concentrations from 10 to 400 mg/kg.

1.3 In determining the conformance of the test results using this method to applicable specifications, results shall be rounded off in accordance with the rounding-off method of Practice E29.

1.4 Values stated in SI units are to be regarded as the standard. The preferred units are milligrams per kilogram (mg/kg).

1.5 *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 a specific hazard statement see Section 8.

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

D3437 Practice for Sampling and Handling Liquid Cyclic Products

D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E203 Test Method for Water Using Volumetric Karl Fischer Titration

¹ This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.04 on Instrumental Analysis.

Current edition approved Jan. 1, 2008. Published February 2008. DOI: 10.1520/D7375-08.

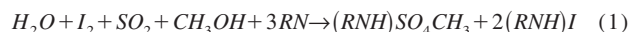
² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

2.2 Other Standard:

OSHA Regulations, 29 CFR paragraphs 1910 and 1910.1200³

3. Summary of Test Method

3.1 In the Karl Fischer reaction, water will react with iodine in the presence of sulfur dioxide, alcohol and an organic base according to the following equation:



where:

RN = organic base.

3.2 In the coulometric titration test method, the sample is injected into an electrolytic cell where the iodine required for the reaction with water is produced by an anodic oxidation of iodide. With this technique, no standardization of reagents is required.

4. Significance and Use

4.1 High water concentrations can have a detrimental effect on many uses of aromatic hydrocarbons, their mixtures and related compounds.

4.2 Water can inhibit desired reactions when aromatic hydrocarbons, their mixtures and related compounds are used in formulations and chemical processes.

4.3 Water can cause corrosion problems and detrimental effects during the manufacture and processing of aromatic hydrocarbons, their mixtures and related compounds.

5. Interferences

5.1 Certain compounds or classes of compounds interfere with the accurate determination of water by the Karl Fischer test method. They include aldehydes, ketones, free halogens, ferric salts and strong oxidizing and reducing agents.

5.2 Free halogens can oxidize the iodate in the KF reagents to form iodine: this causes erroneously low water values.

³ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>.

5.3 A more detailed discussion of KF interferences can be found in Test Method [E203](#) and other sources.⁴

6. Apparatus

6.1 *Coulometric Titrator*—Consisting of a single or dual bath electrolytic cell, dual platinum electrode, magnetic stirrer and control unit.

6.1.1 *Dual Bath Electrolytic Cell*—The dual bath coulometric titration cell consists of a sealed vessel containing both an anode and cathode compartment (a Fritted Cathode Cell). The anodic compartment usually contains a solution consisting of sulfur dioxide, iodide and an amine in an alcoholic based solvent. The cathodic compartment contains similar reagents optimized for the cathodic reduction. When a sample containing water is injected into the anode compartment, the electrolytic cell generates its own supply of iodine from the iodide present. The iodine reacts stoichiometrically with the water and the completion of the reaction is detected with a dual platinum-sensing electrode. The coulombs of electricity required to generate the necessary amount of iodine is then translated by the microprocessor in the control unit into the amount of water that was present in the sample.

6.1.2 *Single Bath Electrolytic Cell*—The single bath electrolytic cell consist of a sealed vessel with an anode and cathode electrode (a Frittless Cathode Cell), a dual platinum sensing electrode and is filled with a single bath reagent. When a sample containing water is injected into the vessel, the electrolytic cell generates its own supply of iodine from the iodide present. The iodine reacts stoichiometrically with the water and the completion of the reaction is detected with a dual platinum-sensing electrode. The coulombs of electricity required to generate the necessary amount of iodine is then translated by the microprocessor in the control unit into the amount of water that was present in the sample.

6.2 *Sampling Syringes*—5 and 10-mL gastight syringes.

6.3 *Standard Gastight Syringe*—10-μL size.

6.4 *Silicone Rubber Blocks Silicone Rubber Septa*, or equivalent.

6.5 *Drying Oven*—Air circulating capable of maintaining a drying temperature of 110°C.

6.6 *Desiccator* with desiccant.

6.7 *Analytical Balance*—Capable of weighing to the nearest 0.0001 g accurately.

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 American

Chemical Society where such specifications are available.⁵ Other grades may be used, providing 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 reagent water conforming to Type II of Specification [D1193](#).

7.3 *Anode Reagent*—For dual bath titration use reagent recommended by manufacturer of titrator.

7.4 *Cathode Reagent*—For dual bath titration use reagent recommended by manufacturer of titrator.

7.5 *Single Bath Reagent*—For single bath titration use reagent recommended by manufacturer of titrator.

7.6 *Toluene*—Reagent grade.

7.7 *Dry Molecular Sieve Type 4A*—4-8 mesh.

7.8 *Dried Toluene Solution*—Fill a narrow top 1 L glass bottle with approximately 2 in. of Molecular Sieve Type 4A, 4-8 Mesh and fill the bottle with reagent grade toluene taking care to minimize or eliminate the headspace. Agitate/mix the toluene and molecular sieve in the bottle and let the solution stand for 2 days in a flammable storage cabinet or fume hood before using.

7.9 *Quality Assurance (QA) Samples*—Samples that are aromatic hydrocarbons or standards of known water content. These (QA) samples are to be used to check the validity of the testing process as described in Section 11. An ample supply of QA sample material should be available for the intended period of use and must also be homogenous and stable under the anticipated storage time and conditions.

8. Hazards

8.1 Consult current OSHA regulations and suppliers Material Safety Data Sheets (MSDS) for materials used in this test method.

9. Sampling

9.1 Obtain a test sample in accordance with Practice [D3437](#). Since aromatic hydrocarbons normally contain low concentrations of water, care must be taken to eliminate the introduction of water from sampling equipment and atmospheric moisture.

9.2 Without taking the proper sampling precautions, more error is typically introduced into the determination of water through sampling techniques than in the titration process itself.

9.3 Dry sample bottles and closures in an oven at 110°C for 1 to 2 h. Place caps on the bottles immediately after removing from the oven.

9.4 Transfer the sample to the bottles as quickly as possible. Adjust the liquid level to achieve as close to zero headspace as

⁴ Mitchell, J. Jr. and Smith, D. M., *Aquametry—A treatise on Methods for the Determination of Water*, Part III—The Karl Fischer Reagent, 2nd ed., J. Wiley and Sons, Inc., New York, NY, 1977.

⁵ *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.