Designation: D8063 – 18

Standard Test Method for Water in Purified Terephthalic Acid (PTA) by Volumetric Karl Fischer Titration[1]

This standard is issued under the fixed designation D8063; 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 determination of water in purified terephthalic acid (PTA) between 0.01 and 1.00 % (w/w) by Volumetric Karl Fischer Titration.

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

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

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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]

D1193 Specification for Reagent Water

D4790 Terminology of Aromatic Hydrocarbons and Related Chemicals

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

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E203 Test Method for Water Using Volumetric Karl Fischer Titration

E300 Practice for Sampling Industrial Chemicals

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *Other Document:*[3]

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200

3. Summary of Test Method

3.1 A PTA sample is dissolved in a solvent (mixture of methanol and pyridine, in the range of 30 ~ 35 % methanol and 70 ~ 65 % pyridine) and titrated with Karl Fischer reagent, which consists of iodine, sulfur dioxide, methanol and an organic base.

3.2 In the Karl Fischer reaction, water will react with iodine according to the following equation:

$$H_2O + I_2 + SO_2 + 3RN + CH_3OH \rightarrow 2RNHI + RNHSO_4CH_3$$

3.3 The volume of Karl Fischer reagent consumed to titrate the sample is converted into the amount of water in the test PTA sample.

4. Significance and Use

4.1 The presence of water in PTA used for the production of polyester is undesirable because it reduces the purity of the PTA.

4.2 Determining the amount of water content remaining from the manufacture of PTA is often required. This test method is suitable for setting product specifications and for use as an internal quality control tool where these products are produced or are used.

5. Interference

5.1 Certain compounds or classes of compounds interfere with the accurate determination of water by the Karl Fischer

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.02 on Oxygenated Aromatics.

Current edition approved Jan. 1, 2018. Published May 2018. DOI: 10.1520/D8063-18.

[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] Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.access.gpo.gov.

test method. They include aldehydes, ketones, free halogens, ferric salts and strong oxidizing and reducing agents.

6. Apparatus

6.1 *Karl Fischer Volumetric Titrator,* consisting of a titration cell, dual platinum electrode, magnetic stirrer, dispensing burette (2 or 5 mL) and control unit. Any Karl Fischer Titrator capable of performing Volumetric Karl Fischer titrations with an electrometric end-point detector can be used in this method.

6.2 *Microlitre Syringe,* 10 µL.

6.3 *Pipette,* 10 mL.

6.4 *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.[4] Other grades may 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 *Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D1193.

7.3 *Karl Fischer Reagent (one component)*—may be prepared in the laboratory or purchased, containing imidazole or pyridine, methanol, iodine and sulfur dioxide. The use of pyridine-free reagents is recommended. These reagents are less toxic, less odorous, and more stable than those containing pyridine. The water equivalence of the Karl Fischer reagent is 1 to 2 mg/mL. (**Warning**—KF reagent contains toxic compounds. It should be dispensed in a well-ventilated area. Care must be exercised to avoid inhalation of the reagent or direct contact of the reagent with the skin.)

7.4 *Pyridine*—Less than 0.05 % (w/w) water. (**Warning**—odorous and harmful if inhaled, swallowed or absorbed through the skin. Perform operations in a hood to avoid pyridine fumes.).

7.5 *Methanol*—Less than 0.05 % (w/w) water. (**Warning**—highly flammable and harmful. May be fatal or cause blindness if swallowed or inhaled.)

7.6 *Pyridine-Methanol Mixed Solvent*—in the range of 30 ~ 35 % methanol and 70 ~ 65 % pyridine (v/v). (**Warning**—highly flammable and vapor harmful.)

7.7 *Water Standard.*

7.8 *5A Molecular Sieve*—3 ~ 5 mm i.d., used for desiccation of methanol or pyridine solvent. Heating the molecular sieve to 500ºC and cooling down in a desiccator is recommended.

NOTE 1—If water content in pyridine or methanol is higher than 0.05 %, 50 g 5A may be added to 500 mL pyridine or methanol for desiccation.

8. Hazards

8.1 Consult current federal regulations, supplier's Safety Data Sheets, and local regulations for all materials used in this test method.

9. Sampling

9.1 Use only representative samples obtained as described in Practice E300, unless otherwise specified. Care must be taken to eliminate the introduction of water from sampling equipment and atmospheric moisture.

9.2 Sample containers should be dry, sealed.

9.3 The amount of PTA sample added into the titration cell is dependent upon the quantity of water in the sample. Table 1 shows the recommended sample size. Other sample size may be used as long as the dissolution of PTA in the pyridine-methanol mixed solvent is not compromised.

10. Preparation of Apparatus

10.1 Follow the manufacturer's directions for preparation of the titration apparatus.

11. Standardization

11.1 Standardize the Karl Fischer reagent daily or as necessary.

11.2 Add an appropriate amount of pyridine-methanol mixed solvent to the clean, dry titration vessel to cover the electrodes. The volume of solvent depends on the size of the titration vessel and sample amount. The 50 mL solvent size for 1 g sample and the recommended titer of the Karl Fischer reagent listed in Table 1 has been found to be satisfactory for testing water in PTA and can be used as a guideline

11.3 All joints and connections to the cell shall be sealed in order to prevent atmospheric moisture from entering the titration cell. Select a stirring speed that is sufficient to create a well-defined vortex with the solvent.

11.4 Pre-titrate the solvent according to the instrument manufacturer's instructions so that all the moisture has been removed prior to introduction of the sample.

11.5 Standardize the Karl Fischer reagent with water by the following method:

11.5.1 Fill up a 10 uL syringe with 5 mg water (7.2). Wipe the needle with a paper tissue to remove residual water from the needle and accurately measure the weight of the total (syringe plus water) to 0.0001 g. Inject the water into the pyridine-methanol mixed solvent in the titration vessel. Make sure that the tip of the needle is below the liquid level. Reseal

TABLE 1 Recommended PTA Sample Size

Expected Water Content, %	Sample Size, g	Solvent Volume, mL	Water Equivalence of the Karl Fischer Reagent, mg/mL
>0.05	1	50	1–2

[4] *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.