



Designation: D7785 – 21

# Standard Practice for Water in Lint Cotton by Oven Evaporation Combined with Volumetric Karl Fischer Titration<sup>1</sup>

This standard is issued under the fixed designation D7785; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice covers the determination of the total amount of water (free and bound) in raw and lint cotton at moisture equilibrium from conditioning in the standard atmosphere for testing textiles.

NOTE 1—For other methods of determination of moisture in lint cotton that do not specify conditioning to moisture equilibrium, refer to Test Methods [D2495](#) and [D1348](#).

1.2 This practice requires the use of oven evaporation to remove all of the water in the fiber matrix, volumetric Karl Fischer (KF) titration to determine water content and water regain, and control current potentiometry to detect the end point.

1.3 This practice is not intended for use with potentiometric (zero current) and coulometric Karl Fischer titrators (see Test Methods [D1533](#), [D4377](#) and [E1064](#)), nor is this practice intended to be used with methanol extracts of cotton (See Test Methods [D1348](#)).

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

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary warnings see [9.1](#).

1.6 *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.*

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee [D13](#) on Textiles and is the direct responsibility of Subcommittee [D13.11](#) on Cotton Fibers.

Current edition approved Jan. 1, 2021. Published February 2021. Originally published in 2012. Last previous edition approved in 2012 as D7785-12. DOI: 10.1520/D7785-21.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- [D123 Terminology Relating to Textiles](#)
- [D1193 Specification for Reagent Water](#)
- [D1348 Test Methods for Moisture in Cellulose \(Withdrawn 2017\)<sup>3</sup>](#)
- [D1441 Practice for Sampling Cotton Fibers for Testing](#)
- [D1533 Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration](#)
- [D1776 Practice for Conditioning and Testing Textiles](#)
- [D2495 Test Method for Moisture in Cotton by Oven-Drying](#)
- [D4377 Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration \(Withdrawn 2020\)<sup>3</sup>](#)
- [D7139 Terminology for Cotton Fibers](#)
- [E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods](#)
- [E203 Test Method for Water Using Volumetric Karl Fischer Titration](#)
- [E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)
- [E1064 Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration](#)

## 3. Terminology

### 3.1 Definitions:

3.2 For all terminology relating to [D13.11](#), Cotton Fibers, refer to Terminology [D7139](#).

3.3 The following terms are relevant to this standard: bound water, free water, test specimen, water content, water regain.

3.4 For definitions of all other textile terms see Terminology [D123](#).

## 4. Summary of Practice

4.1 A 0.1 g test specimen is sealed in a glass vial, positioned on the sample turntable and lowered into the single sample

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

oven where dry nitrogen transports the water vapor that is rapidly evaporated from the fibers into a titration cell. The amount of water is determined by volumetric titration with Karl Fischer reagent with the end point determined by electrodes that measure a sharp change in potential when the iodine in the reagent is reduced by sulfur dioxide in the presence of water. The volume of reagent required to titrate the wet gas stream is converted into the amount of water in the test specimen. For the reaction mechanism and the chemicals involved with this practice, see [Appendix X1](#).

## 5. Significance and Use

5.1 The water content of raw or lint cotton determined by this practice, calculated from the required volume of reagent, may be greater, equal to or less than the moisture content measured by standard oven drying methods. These differences may be of significance in commercial transactions (**1-3**)<sup>4</sup> (see also [Appendix X2](#)). Water content by this method is not to be considered the same attribute as moisture content.

5.2 Standard test methods using volumetric and coulometric Karl Fischer reagent are two of the most widely used procedures for the determination of water.

5.3 The volumetric method is typically used for the routine determination of water in the mass percent range of concentrations. If samples contain very low levels of water, the coulometric technique should be considered (see Test Methods [D1533](#), [E1064](#)).

5.4 This practice for testing the water content of cotton can be used for acceptance testing of commercial shipments of lint cotton, manufacturing control and calibration of fast, indirect sensors to measure water.

5.5 Information on the water content of cotton is desirable since the physical properties of cotton are significantly affected by its water content. Variations in the amount of water present, or its regain, affect the mass and hence the market value of a lot of material.

5.6 The observed volume of Karl Fischer reagent used in this practice to analyze a specimen represents the water in the absence of side reactions in an oven supplied with air (**3**).

NOTE 2—Side reactions in cotton that confound the actual weight loss due to water have been demonstrated in two laboratory ovens and a thermogravimetric analysis oven supplied with air (**3**). This results in an approximation regarding the actual amount of water in cotton based on mass loss by drying. If the moisture content by oven drying agrees with the water content measured by Karl Fischer titration, the one-to-one correspondence may be coincidental due to the presence of both negative and positive biases in moisture content values.

## 6. Interferences

6.1 Compounds such as aldehydes, ketones, free halogens, most acids, and oxidizing agents may interfere in this titrimetric method. A detailed discussion of interfering substances can be found in the treatise on aquametry (**4**). Detailed investigations of sources of bias in industrial samples analyzed by an

automatic oven evaporator combined with the coulometric Karl Fischer method demonstrate the need to validate the procedures (**5,6**).

6.2 To establish that the compounds listed in [6.1](#) do not cause a significant change in water results in this practice, information was obtained by a series of observations during the performance of these tests.

6.2.1 A drifting endpoint, an indicator of interferences in cotton, was not observed with the reagents used in this practice (see Test Method [D1533](#)).

6.2.2 The recovery of spiked additions of water to the sample matrix was not significantly different from 100 % (see Test Method [E203](#)).

6.2.3 The slope of the plot of water content of cotton versus oven module temperature in the range of 150 to 160°C was not significantly different from zero (**7**). A nonzero slope is indicative of biases due to interferences.

6.2.4 Studies have shown that the selectivity of this practice for water in cotton, relative to the interferences in cotton, is not significantly influenced by these interfering substances (**2,8**).

6.2.4.1 In practice, the first step in the selectivity determination is to set up a low temperature laboratory oven at 50°C to pre-dry the test specimen. The sealed Karl Fischer sample vial containing 0.1 g cotton (see [10.2.1](#)) is placed in this oven and the vial is purged overnight with dry nitrogen at 60 mL/min to separate water from the test specimen. The low temperature distillation technique evaporates off the moisture without removing interfering substances.

6.2.4.2 Confirmation of complete water removal from each test specimen is checked or confirmed by recording a near infrared reflectance spectrum taken *in vitro*.

NOTE 3—Near infrared reflectance spectra taken through the bottom of the sealed glass specimen vial by an external probe immediately after Karl Fischer analysis will show the absence of the strong water band in cotton at 1400 nm (**1,3**).

6.2.4.3 The recommended Karl Fischer titration procedure (see Section [14](#)) is carried out on two different treatments of specimens of the same cotton in sealed vials: (a) the anhydrous cotton and (b) the fibers before drying.

6.2.4.4 The volumes of reagent consumed are reported as: (a) the equivalent water content of the interferences (%) in the pre-dried cotton and (b) the water content (%) of the cotton.

6.2.4.5 The selectivity of this practice for water in cotton is computed from the two water measures in [6.2.4.4](#).

## 7. Apparatus

7.1 *Volumetric Karl Fischer Titrator*—with dual platinum electrodes with the following accessories:

7.1.1 *Titration Vessel*—Consists of a sealed vessel containing the platinum electrodes, several tubes in the cell with different applications and a vent in the top of the cell. The vent is connected to a drying tube.

7.1.2 *Titration Vessel Tubes*—The four required tubes in this practice extend below the liquid level in the cell. In one tube flows the wet nitrogen stream. Another tube is connected to the mechanical burette that automatically dispenses the iodine reagent. The two remaining tubes are used for pumping in fresh solvent and pumping out spent solvent.

<sup>4</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

7.1.3 *Outlet Gas Pressure Line, with Flow Controller*—The gas transfer line between the oven block and titration vessel shall be heated to prevent water condensation in the line. The gas flow is selected to ensure the water vapor is absorbed by the solvent.

7.1.4 *Magnetic Stirrer*—The glass or PTFE covered magnetic stirring bar should spin at a sufficient rate to create a solution vortex in the titration vessel that helps to capture the carrier gas stream and allow for absorption of water by the solvent in the cell.

7.2 *Turntable – Oven Assembly*—with oven module that heats one specimen at a time with the following accessory:

7.2.1 *Needle Guide – Double Wall Needle*—The needle guide moves the double wall needle to penetrate the septum of a sealed vial on the turntable, moves the vial with penetration needle inside the vial in and out of the oven block, and removes the needle from the vial. The double wall needle shall be long enough to penetrate the septum but not penetrate the sample matrix.

7.3 *Glass Vials and Canning Jars*—with crimp type PTFE septum caps to seal the specimen vials and wide-mouth type jars with lids and rings to seal the jars used to store specimen vials before Karl Fischer analysis.

## 8. Reagents and Materials

8.1 *Purity of Reagents*—Unless otherwise stated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>5</sup>

8.2 *Purity of Waters*—Unless otherwise indicated, references to water shall mean reagent water as defined by Type II and III of Specifications D1193.

8.3 *Volumetric Karl Fischer Reagents*—Solvent and titration reagents are available commercially.

8.3.1 *Solvent Reagent*—The working medium or solution in the titration vessel. Consists of a mixture of organic chemicals.

8.3.2 *Titration Reagent*—Consists of a mixture of iodine (the key ingredient) and other organic chemicals.

8.4 *Water Standards*—Solutions are available commercially to calibrate the combination of the oven and volumetric titrator. Consist of water (units are mg water/mL water standard) and a mixture of organic chemicals. The response of the volumetric titrator itself, without the oven, shall be checked with pure water (see 8.2).

8.5 *Dry Nitrogen*—References to dry nitrogen shall mean compressed nitrogen gas containing <1 ppm water (volume/volume) and used without further purification.

## 9. Hazards

9.1 Commercially available Karl Fischer solvent and titration reagents used in this practice may contain potentially

hazardous chemicals, such as iodine, sulfur dioxide, chloroform, imidazole and substituted imidazole, hydriodic acid, diethylene glycol monoethyl ether, or other organic materials. Commercially available water standards used in this practice may contain propylene carbonate, xylene, or other organic materials. Wear chemically resistant gloves when handling the materials. Care must be exercised to avoid unnecessary inhalation of organic vapors or direct contact with the skin or eyes.

NOTE 4—Carefully read and follow manufacturer's instructions and Material Safety Data Sheets when using commercially available reagents and water standards.

## 10. Sampling, Test Specimens, and Test Units

10.1 The preferred method for sampling cotton fibers for testing is Practice D1441.

10.2 *Test Specimens*—Three test specimens shall be taken at random from each laboratory sample.

10.2.1 *Test Specimen Size*—The test specimen size is  $0.1 \pm 0.0003$  g.

## 11. Preparation of Apparatus

11.1 Thoroughly clean and dry the titration vessel and reassemble according to the manufacturer's recommendations.

11.2 Turn the oven and volumetric titrator on and allow software and hardware initializations to be completed. Check that the oven temperature is set at 150°C.

11.3 Fill reagent reservoirs with appropriate reagents according to the manufacturer's instructions.

11.4 Open the cylinder of compressed dry nitrogen.

## 12. Calibration and Standardization

12.1 All successive steps must be done in the general laboratory area of the Karl Fischer instrumentation.

NOTE 5—The cotton fiber atmospheric conditioning requirements do not apply to the oven evaporator and Karl Fischer titrator because it is beneficial to operate the system at lower humidity.

12.2 Tightly crimp five of the PTFE septum caps onto the empty glass sample vials. Place one of the empty vials in the "conditioning position" of the sample rack on the oven turntable. Place the remaining four vials into the first four sample positions of the oven rack. The first position is to initialize the system (initializing blank) and remove any extraneous water. The following three are the blank vials for the titration of atmospheric water in the vials.

12.3 Follow the manufacturer's software instructions to condition or purge the complete tubing system with dry nitrogen flowing through the conditioning vial, which is maintained on the rack at room temperature.

12.3.1 With the conditioning vial on the rack, the titration vessel is conditioned with the volumetric Karl Fischer titrator until the reaction mixture in the titration vessel is dry.

12.4 Each of the three blank vials, in turn, with programmed conditioning of the reaction medium in between, is moved into the oven at  $150 \pm 0.1^\circ\text{C}$  and the gas flow transports atmospheric moisture in the vial into the titration cell for the blank determination.

<sup>5</sup> 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 L6d, Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

12.5 The preset routine titrates the blank vials with Karl Fischer reagent according to the manufacturer's program. The mean volume of the Karl Fischer reagent, mL, required to titrate the blank vials is recorded on hard disk.

12.6 Standardize the Karl Fischer reagent using a commercially available water standard solution.

12.6.1 Rinse a syringe of suitable capacity with the water standard by drawing a small volume of the liquid into the syringe, rotating the syringe, and ejecting into a waste container. Repeat the procedure and fill the syringe with the standard solution and remove air bubbles.

12.6.2 Wipe the needle with paper tissue to remove any residual water from the needle and determine the weight of syringe plus water standard to the nearest 0.1 mg.

12.6.3 Dispense a volume of standard solution into an empty glass sample vial that is equivalent to 7 % water (mean value based on available data) in 0.1 g of conditioned cotton. Reweigh the syringe and enter the mass of water standard into the computer. Crimp the PTFE septum tightly onto the vial.

12.6.4 Repeat 12.6.3 and prepare a total of three crimped vials containing the water standard solution.

12.6.5 Place the three water standard vials into the first available sample positions of the oven rack.

12.7 Activate the start button to titrate with Karl Fischer reagent according to the manufacturer's program. The mean volume of Karl Fischer reagent, mL, millilitres, required to titrate the water standard vials is recorded on hard disk.

12.8 *Calculation*—Calculate the water equivalent,  $E$ , of the KF reagent, in milligrams of water per millilitre of reagent, as follows:

$$\begin{aligned} \text{water equivalent} &= \text{mg water titrated/mL reagent} \\ &= \text{g water standard} \times 10 / (\text{mL reagent, standard} - \text{blank}) \\ E &= S \times 10 / (A - B) \end{aligned} \quad (1)$$

where:

$S$  = grams of water standard containing a nominal 10 mg water/g standard (density = 1 g/mL),

$A$  = millilitres of reagent required for titration of the standard, and

$B$  = millilitres of reagent required for titration of the blank.

12.8.1 The final value for  $E$  should be based on the mean of a minimum of three replicates.

12.8.2 The replicate values for  $E$  should have a relative standard deviation of not greater than 1 %. The standardization should be repeated if a greater relative standard deviation is determined.

NOTE 6—If desired, more detailed instrument calibrations, Karl Fischer volumetric titrator alone and the oven connected to the titrator, are possible (2). Accuracy and linearity confirmations may be performed.

### 13. Conditioning of Laboratory Cotton Samples and Containers

13.1 Use gloves in handling the cottons and containers.

13.2 Condition approximately 10 g of each cotton laboratory sample.

13.3 Condition clean, dry Karl Fischer glass vials and septum caps. Condition clean, dry 225 mL wide mouth glass jars with screw cap lids.

13.4 Condition cottons and containers in an appropriate manner, according to Practice D1776 for at least 24 h.

### 14. Procedure

14.1 All successive steps must be done in the conditioned laboratory with conditioned cotton and containers (see Section 13).

14.1.1 Place a septum cap over the glass vial to be used in the "conditioning position" of the sample rack, the initializing blank, and over the three blank vials.

14.1.2 Label septum caps with a suitable code representing the cotton laboratory sample and three corresponding specimens taken from the sample. Place the labeled septum cap over a KF vial.

14.1.3 From the laboratory sample, the 0.1 g test specimens are randomly selected, weighed to a mass range of 0.0997 to 0.1003 g and placed in the corresponding vials.

14.1.4 The septa are crimped tightly on to the empty vials and the test specimen vials. The weighing and crimping should be completed in a short span of time to minimize variability in the conditioning atmosphere.

14.1.5 Pack the crimped empty vials in a wide mouth jar and screw the lid on tightly.

14.1.6 Pack the crimped specimen vials in wide mouth glass jars and screw the lids on tightly. The empty and test specimen vials should remain in the jars until Karl Fischer testing.

NOTE 7—When the KFT testing room is a different area than the conditioning room, the tightly crimped Karl Fischer cotton specimen vials are packed into the larger clean jars. The jars shall be transferred to the testing room and opened immediately before testing.

14.2 Follow the manufacturer's software instructions for the automated method to determine water in the cottons.

14.2.1 Open the wide mouth jars and place the sealed empty and sample vials on the sample rack.

14.2.2 Verify that the Karl Fischer solvent in the titration vessel has been titrated to dryness. The endpoint is indicated visually by the light brown color of the solvent or by an audible alarm, or both, depending on the equipment manufacturer.

14.2.3 Each blank vial and each test specimen vial, in turn, with programmed conditioning of the reaction medium in between, is moved into the oven set at  $150 \pm 0.1^\circ\text{C}$  and the gas flow transports the driven-off water in the cotton into the titration cell.

14.2.4 The water vapor is titrated until the controlled current potentiometric endpoint is reached and the volume of titrant is recorded.

14.2.5 After analysis of six test specimens, follow the manufacturer's instructions to pump the spent solvent out of the titration cell and pump in fresh solvent.

### 15. Sample Calculations

15.1 Most commercially available volumetric Karl Fischer titrators automatically calculate the water content in percent. If not or to check the automatic results, calculate the mass of water in a cotton specimen as follows: