Designation: D7785 – 21

Standard Practice for Water in Lint Cotton by Oven Evaporation Combined with Volumetric Karl Fischer Titration[1]

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 (ε) 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D123 Terminology Relating to Textiles
D1193 Specification for Reagent Water
D1348 Test Methods for Moisture in Cellulose (Withdrawn 2017)[3]
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)[3]
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

[1] This practice is under the jurisdiction of ASTM Committee D13 on Textiles and is the direct responsibility of Subcommittee D13.11 on Cotton Fibers.

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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] The last approved version of this historical standard is referenced on 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**)[4] (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.

[4] The boldface numbers in parentheses refer to a list of references at the end of this standard.