Designation: D7785 – 12

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

1. Scope

1.1 This test method 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 test method 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 test method is not intended for use with potentiometric (zero current) and coulometric Karl Fischer titrators (see Test Methods D1533, D4377 and E1064), nor is this test method 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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary warnings see 9.1.

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
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
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 Test Method

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 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 test method, see Appendix X1.

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[1] This test method 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.

5. Significance and Use

5.1 The water content of raw or lint cotton determined by this test method, 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**)[3] (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 test method 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 test method 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 test method, 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 test method (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 test method 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 test method 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 test method 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.

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.

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

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.[4]

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 test method 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 test method 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 ± 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 ± 0.1°C and the gas flow transports atmospheric moisture in the vial into the titration cell for the blank determination.

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

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

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:

$$\text{water equivalent} = \text{mg water titrated/mL reagent}$$

$$= \text{g water standard} \times 10/(\text{mL reagent, standard} - \text{blank})$$

$$E = S \times 10/(A - B) \quad (1)$$

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 1.0003 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 ± 0.1°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 potentiometic 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:

$$M = (C - B) \times E \times 0.001 \quad (2)$$

where:

M = grams of water titrated in the specimen,

C = millilitres of reagent required for titration of the specimen,

B = millilitres of reagent required for titration of the blank, and

E = water equivalent, in milligrams of water per millilitre of KF reagent.

15.2 Calculate the water content of the cotton specimen as follows:

$$\text{Water content, mass \%} = (M/W) \times 100 \qquad (3)$$

where:

w = grams of specimen

15.3 Calculate the water regain relative to the dry mass of the specimen as follows:

$$\text{Water regain, mass \%} = [M/(W - M)] \times 100 \qquad (4)$$

15.4 Calculate the water content or regain of each specimen to the nearest 0.01 % and the average of the specimens to the nearest 0.01 %.

15.4.1 Alternately, calculate the water content or regain directly by one formula rather than two as follows:

$$\text{Water content, mass \%} = (C - B) \times E \times 0.1/W \qquad (5)$$

$$\textit{Water regain, mass \%} = 1/[W/(C - B) \times E \times 0.1 - 0.01] \qquad (6)$$

15.5 Water regain may be calculated from water content and water content may be calculated from water regain as follows:

$$R = [T/(100 - T)] \times 100 \qquad (7)$$

$$T = [R/(100 + R)] \times 100 \qquad (8)$$

where:

R = water regain, and
T = water content.

16. Report

16.1 Report the following information:

16.1.1 State that the specimens were tested as directed in this method (ASTM Test Method D7785). Describe the material or product sampled and the method of sampling used. If the purpose of this test method is to determine the water content or water regain under prevailing conditions outside the conditioned laboratory, report the variances as specified in 16.1.3.

16.1.2 Report the water content or water regain of each specimen to the nearest 0.01 % and the average of the specimens to the nearest 0.01 %.

16.1.3 If the test specimens and containers were not conditioned to moisture equilibrium as specified in 13.4, incorporate in the report a statement as to the particular conditions used in the test.

17. Precision and Bias

17.1 *Precision*—The precision of this test method is based on a study conducted in 2010. A single laboratory tested eight different cotton materials, on two days, under two conditions (cleaned and raw). Every "test result" represents an individual determination, and the single analyst reported triplicate test results on the materials on each day. The precision statement was determined through statistical examination of 96 results. Except for the use of only a single laboratory, Practice E691 was followed for the design and analysis of the data.[5]

TABLE 1 Percent Water – Day 1 – Raw

Sample ID	Average Water Content	Repeatability Standard Deviation	Repeatability Limit (r)
Sample 1 Raw	8.14	0.066	0.185
Sample 2 Raw	8.45	0.337	0.944
Sample 3 Raw	8.34	0.086	0.240
Sample 4 Raw	7.92	0.039	0.109
Sample 5 Raw	8.09	0.030	0.084
Sample 6 Raw	8.06	0.183	0.508
Sample 7 Raw	8.01	0.052	0.142
Sample 8 Raw	8.22	0.055	0.154
Average	8.15	0.106	0.296

TABLE 2 Percent Water – Day 1 – Cleaned

Sample ID	Average Water Content	Repeatability Standard Deviation	Repeatability Limit (r)
Sample 1 Cleaned	8.08	0.026	0.074
Sample 2 Cleaned	8.03	0.066	0.184
Sample 3 Cleaned	7.88	0.080	0.223
Sample 4 Cleaned	7.80	0.010	0.028
Sample 5 Cleaned	7.81	0.072	0.203
Sample 6 Cleaned	8.09	0.058	0.163
Sample 7 Cleaned	7.89	0.081	0.226
Sample 8 Cleaned	8.21	0.092	0.257
Average	7.97	0.061	0.170

TABLE 3 Percent Water – Day 2 – Raw

Sample ID	Average Water Content	Repeatability Standard Deviation	Repeatability Limit (r)
Sample 1 Raw	8.31	0.127	0.324
Sample 2 Raw	8.39	0.120	0.336
Sample 3 Raw	8.35	0.010	0.028
Sample 4 Raw	7.95	0.020	0.057
Sample 5 Raw	8.05	0.033	0.091
Sample 6 Raw	8.14	0.119	0.334
Sample 7 Raw	8.18	0.021	0.058
Sample 8 Raw	8.23	0.189	0.539
Average	8.12	0.078	0.220

TABLE 4 Percent Water – Day 2 – Cleaned

Sample ID	Average Water Content	Repeatability Standard Deviation	Repeatability Limit (r)
Sample 1 Cleaned	8.01	0.138	0.387
Sample 2 Cleaned	7.94	0.060	0.168
Sample 3 Cleaned	7.94	0.013	0.036
Sample 4 Cleaned	7.82	0.028	0.079
Sample 5 Cleaned	7.86	0.022	0.063
Sample 6 Cleaned	8.05	0.036	0.099
Sample 7 Cleaned	8.10	0.080	0.225
Sample 8 Cleaned	8.06	0.040	0.113
Average	7.97	0.052	0.146

17.1.1 *Repeatability (Single Analyst) limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

17.1.1.1 Repeatability limits are listed in Tables 1-4.

17.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D13-1133.

difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

17.1.2.1 Reproducibility limits cannot be determined from single laboratory results.

17.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

17.1.4 Any judgment in accordance with 17.1.1 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. Data from just a single laboratory reporting results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Consider the repeatability limit as a general guide, and the associated probability of 95 % as only a rough indicator of what can be expected.

17.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

18. Keywords

18.1 Karl Fischer reagent; lint cotton; pyridine-free; volumetric Karl Fischer titration; water content; water regain

APPENDIXES

X1. REACTION MECHANISM

X1.1 The reaction mechanism in this test method involves several chemicals as follows:

$$SO_2 + R'OH + RN = [RNH]SO_3R' \text{ (slow step)}$$

$$H_2O + I_2 + [RNH]SO_3R' + 2RN = [RNH]SO_4R' + 2[RNH]I$$

where:

RN = an organic base, and
R'OH = an alcohol.

X2. RATIONALE FOR WATER DETERMINATION

X2.1 The standard oven-drying procedures are used worldwide to measure moisture content and moisture regain in cotton by drying in ambient air at 105 – 110°C. The weight of the *lost volatiles* is the measure of moisture content. The known biases have been reported in scientific papers dating back to the 1930s (**9**).

X2.2 The bias phenomena had not been studied in depth – how or why they occur – until 2007, when USDA scientists in New Orleans, at the request of the cotton industry, picked up where scientists left off more than half a century ago. Knowledge of the actual moisture or water content is of financial importance and can help to make certain that U.S. cotton industry segments compete profitably.

X2.3 USDA scientists elucidated the underlying bias mechanisms in standard oven-drying, confirmed the known biases and discovered new biases (**1,3**). Some water remains in the cotton. Particulate matter (trash, dust, broken and immature fibers) is removed from the cotton. Oxidation and decomposition reactions of cellulose and impurities produce non-aqueous volatile material of low vapor pressure. A decomposing odor is detected. The chemical reactions are eliminated in a dry nitrogen atmosphere and hysteresis does not occur in this environment.

X2.4 Test Method D7785 for measuring water in cotton by KFT will enable the calibration, packaging and distribution of standard reference cottons for water content and water regain.

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