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TEXTILE TECHNOLOGY

Review of Standard Test Methods for Moisture in Lint Cotton

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ABSTRACT

Almost all of the standard test methods for measuring moisture in lint cotton utilize the oven-drying technique. The loss in weight is taken as the amount of water and is expressed as a percentage of the mass of either the moist or dried material. There is a need to review the standard methods and compare terminology, procedures, the quantities that are calculated and measured and the known biases, and available accuracy and precision data. This review covers the standard test methods – voluntary, national and international – for moisture in lint cotton in eleven countries that represent all six continents. Examples of the moisture terminology presented in this review include: oven dry, bone dry, moisture equilibrium and moisture free. Procedural examples demonstrate wide ranges in the oven method specifications: sample analyzed, 1 to > 50 g; conditioning prior to drying, from none to optional to yes; standard testing environment (70º F and 65 % RH), from none to optional to required; and oven temperature, generally 105º C to 110º C. To help understand the various moisture quantities that are calculated in the methods and provide for direct comparability, simple formulas are presented in terms of the quantities measured and the known biases. In the reviewed standards, these biases are assumed equal to zero, ignored, or the standard method eliminates a specific bias. In terms of round trials, there is no quantitative accuracy information; however, the biases are readily admitted in the standards. Between-laboratories precision is generally poor or is lacking.

The properties of cotton fiber are strongly affected by moisture content because it is a hygroscopic fiber and absorbs or desorbs moisture from the surrounding atmosphere. In general, the fibers that absorb the greatest amount of moisture are the ones whose properties change the most. The main types of properties affected are: dimensional, mechanical and electrical (Saville, 1998). Furthermore, because the moisture content in cotton may vary from 5 to 10%, the actual moisture content can have a significant effect on the mass of the material. This factor has commercial importance when cotton fibers and yarns are bought and sold by weight.

On a more fundamental level, the molecular structure of the cotton fiber includes hydroxyl groups that attract water molecules that hydrogen bond to the cellulose. This bonding changes the properties and characteristics of the fiber. Possible locations of water molecules on cotton fibers have been explored based on fiber size, crystallite size, known crystal structures of cellulose hydrates, and cross-sections of swollen cotton fibers (French et al, 2004). A 5 % moisture content corresponds to roughly 760 monolayers of water if it all goes on the external surface of the fiber, or about 0.3 monolayers if it were able to access all surfaces of the crystallites that are indicated by x-ray diffraction studies.

The impact of moisture on HVI measurements and processing issues are driving the current research in moisture test methods. Additionally, new moisture methods are being developed for online analysis. However, all test methods used in routine analysis must be traceable to an accurate reference method (s).

The USA does not have a national standard method for measuring moisture in cotton. In this country, preferred standard test methods for moisture in cotton fibers are the American Society for Testing and Materials (ASTM) standards (e.g., ASTM 2495, 2001). These methods are almost all based on oven drying. The U.S. cotton industry questions the reliability of the oven-drying test and has requested that this laboratory develop improved standard test methods of moisture in cotton. As a prerequisite to initiating laboratory work on better test methods, we conducted a literature review of the standard test methods.

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Backe (2002) published an excellent review related to cotton moisture from harvesting through textile processing. However, the specific area of the standard test methods, including accuracy and precision, is not included in that review. More recently, Byler (2006) published a comprehensive review of the effect of moisture content and the addition of moisture in seed cotton before ginning on fiber length. The standard test methods, including ease and cost of analysis, are not included in his review.

This is the first review of developments in the standard test methods for moisture in lint cotton (loose cotton fibers, raw or processed) over the period 1930-2007. The primary areas reviewed include the standard test methods – voluntary, national and international – and the sources of error in the procedures. International standard publications were translated into English by commercial translators.

As a final note, we also focus on emerging trends and current issues related to the standard test methods by oven-drying. (As a reviewer of this paper pointed out, the scope of the review does not include the necessary actions, such as sealing a sample in an airtight container, to properly manage cotton samples during transportation, storage, use and transformation due to processing. Additionally, the reviewer noted that no comparative data was reported in this bibliographic study on the same set of cottons analyzed for moisture in round-robin studies with all of the standard methods.)

A table of acronyms is provided (Table 1) as a quick reference to the acronyms of national standards bodies. A table of useful websites is also provided (Table 2). The NIST website gives a comprehensive list of national standards bodies of various countries.

STANDARD TEST METHODS BY COUNTRY BASED ON OVEN DRYING

Standard methods by country for determining moisture in lint cotton using oven-drying techniques that are reviewed in this paper are listed in Table 3. Representative countries were selected from all six continents. The test methods are grouped into four categories: countries without national standards, countries with national standards, countries with national standards superseded by ISO 6741 and global.

Standards developed by ASTM (U.S. headquartered) have gained international acceptance and serve as de facto global standards. The input to these standards comes from international participants, in some cases on an individual basis, in others on an organizational basis.

COUNTRIES WITHOUT NATIONAL STANDARDS

These nations must rely on standards developed by voluntary standards organizations (e.g., ASTM), national standards from other countries, or the global oven drying method (ISO 6741, 1989). Within a nation, there may be a mix of standards used in the industry (i.e., more than one ASTM method).

Table 1. List of acronyms of national standards bodies

Acronym	Organization
AATCC[z]	American Association of Textile Chemists and Colorists
AFNOR	Association Francaise de Normalization
ASTM[z]	American Society for Testing and Materials
BS/BSI	British Standards/British Standards Institute
DIN[z]	National Standards of Germany/Deutsches Institut fur Nirmung
EN/CEN	European Standards/Comite European de Normalization
EOS	Egyptian Organization for Standardization and Quality Control
GB[z]	National Standards/People's Republic of China
ISO[z]	International Organization for Standardization
NIST	National Institute of Standards and Technology
SANS/SABS	South African National Standards/South African Bureau of Standards
UNI[z]	Italian Organization for Standardization/Ente Nazionale Italino di Unifcazione

[z] Same acronym for standard and sponsoring organization.

Table 2. Useful websites

Website	Link
www.aatcc.org	AATCC standards
www.afnor.fr/	French standards (NF)
www.astm.org	ASTM standards
www.bsonline.bsi-global.com	British Standards (BS) Catalogue
www.beuth.de	National Standards (DIN) of Germany in German
www.cenorm.be/	Catalogue of European Standards (EN)
www.eos.org.eg/en_home.html	Egyptian standards (ES)
www.ccc-us.com	China Compulsory Certification (CCC); National Standards (GB) of the People's Republic of China in Chinese and English
www.iso.org	Catalogue of ISO textile fiber standards
www.nist.gov/oiaa/stnd-org	List of national standards bodies by NIST
www.sabs.co.za	Catalogue of South African National Standards (SANS)
www.uni.com/en/	Italian standards (UNI)
www.standards.com.au	Australian standards

Standards developed by voluntary organizations such as ASTM are subject to the current interests of subcommittee members, who work in the industry. At any given time, a particular standard may be active (may purchase the document from ASTM), withdrawn (cannot purchase document but still use it if you have access to a copy), or in revision for resubmission and approval.

USA

In this country, the voluntary standards for moisture in lint cotton most often cited are the ASTM methods (Table 3): two are active, D 2495 and D 1348, and one is withdrawn, D 2654. The relevant moisture terminology in these methods is defined in Table 4 (e.g., oven dry, moisture content and regain). A summary of the essentials in the oven-drying methods (Tables 5 and 6) allows for a quick view of the specifications in the methods. The simple formulas to compute the amounts of moisture in lint cotton by the various procedures are all different (Table 7). Moisture acronyms for the formulas are given in Table 8. Available accuracy and precision results are tabulated for ease of reference (Table 9).

The title page of all standards, whether voluntary, national or global, generally includes information following the designated standard number to indicate the year of original approval or adoption, year of last revision, and year of current edition. To standardize the presentation of this information in the text below, we introduce the standard number in bold print followed by the year of original publication and year of current edition. By contrast, the standard number is listed in the tables without publication dates.

Table 3. Standard methods by country for determining moisture in lint cotton using oven drying method

Country	Standard Number
countries without national standards	
USA	ASTM D 2495
	ASTM D 1348
	ASTM D 2654
	USDA Handbook 422
	AATCC 20A
Australia	ASTM/ISO/other standards
Brazil	ASTM/ISO/other standards
India	ASTM/ISO/other standards
S. Africa	ASTM/ISO/other standards
countries with national standards	
China	GB/T6102.1
Germany	DIN 53800
countries with national standards replaced by ISO 6741	
Egypt	ES 2448-1
France	NF G08-003
Italy	UNI 9213-1
United Kingdom	BS EN 4784-1
global standard	
international	ISO 6741-1,2,3,4

Table 4. Moisture terminology in international standards test methods by oven drying

Method	Term	Definition
ASTM D 2495	oven dry	cotton heated under prescribed conditions of temperature and humidity until there is no further significant change in its mass
	moisture content	the amount of water in cotton determined under prescribed conditions and expressed as a percentage of the mass of the moist material
	moisture regain	the amount of water in cotton determined under prescribed conditions and expressed as a percentage of the mass of the dried material
ASTM D 1348	bone dry	cotton heated under prescribed conditions of temperature and a stream of dry air passed through the oven
	moisture content at bone dry conditions	the amount of water in cotton heated under prescribed conditions of temperature and zero relative humidity and expressed as a percentage of the mass of the moist material
	moisture regain at bone dry condition	the amount of water in cotton heated under prescribed conditions of temperature and zero relative humidity and expressed as a percentage of the mass of the bone dry material
ASTM D 2654	moisture equilibrium	the condition reached by cotton when it no longer takes up moisture from, or gives up moisture to, the surrounding atmosphere
	moisture free	a descriptive term for cotton exposed to oven drying followed by desiccated air at room temperature until there is no further significant change in mass or has been treated by a distillation process using a suitable solvent
	moisture pickup	the moisture content of cotton in equilibrium with the standard atmosphere, after treatment to render it moisture free, at 70 ± 2ºF and 65 ± 2 % RH
	moisture regain after scouring	the moisture regained by cotton at specified equilibrium conditions after scouring and drying in a vacuum oven
Chinese GB/T6102.1	moisture	the water and other volatiles lost during the drying test of raw cotton
ISO 6741-1	oven dry mass of the consignment	mass of the consignment at time of sampling corrected by the sample ratio of oven dry mass to original (moist) mass

ASTM D 2495. Originally published by ASTM in 1961 as D 2495-61 T; current edition published July 2001 (ASTM D 2495, 2001). This particular ASTM standard is cited more often than D 1348 and D 2654. The purpose of the method is to determine the moisture content and regain at a specified time or under prevailing conditions outside the laboratory (i.e., at various stages of processing). Consequently, samples are not preconditioned or conditioned after they are taken.

The oven is controlled at 105 ± 2º C. The air entering the fan-forced ventilation oven must come from the standard atmosphere for testing textiles (70 ± 2º F and 65 ± 2 % RH). A 5 g sample is required for lint cotton.

This test method offers two procedures for oven drying and weighing, one using an oven balance and the other an external balance with cooling in a dessicator before weighing. There are critical differences between the two options. Procedure 1 calls for an oven balance and drying ≥ 1 hr or 15 min intervals until there is < 0.1% change in sample mass between successive weighings. Procedure 2 calls for an external balance and ≥ 8 hr of oven drying. Then the sample container is closed while still in the oven and placed in a desiccator to cool. During cooling, the dessicator lid is removed and the cover of the sample container (weighing bottle with ground-glass stopper) is raised slightly to equalize the air pressure. If the container is not opened during cooling, the partial vacuum created inside the weighing bottle may make it impossible to open without breakage, and cause an error in the weight due to the vacuum in the bottle.

We should note that Procedure 2 increases oven drying time by 7 hr (700 %) compared to Procedure 1. There is no additional sample drying in the desiccator due to the fact that the sample container is closed. It follows that the expected relative mass of the oven dried sample for the two procedures is OD ≤ D (Tables 7 and 8). The resultant formulas for moisture content and regain are: Equations 1 and 2 (Procedure 1) and Equations 3 and 4 (Procedure 2). The other quantities included in the formulas, e_1 and e_2, are the known biases in this method and are put in the equations to emphasize that the biases do exist. In practice, however, both are assumed equal to zero or ignored.

Table 5. ASTM standard oven-drying methods summary (lint cotton)

			Oven characteristics			
Condition prior to drying	Sample analyzed (g)	Std. testing[z] atm.	Temp. (°C)	Fan forced vent.	Oven bal.	Drying time
ASTM D 2495. SCOPE - TEST METHOD FOR MOISTURE IN LINT COTTON - DELIVERED TO THE LABORATORY - BY 2 DIFFERENT PROCEDURES						
Procedure 1. Specimen weighed in oven:						
N	5	Y	105 ± 2	Y	Y	Dry ≥ 1 hr or 15 min intervals until < 0.1% change in sample mass
Procedure 2. Specimen weighed outside oven:						
N	5	Y	105 ± 2	Y	N	Dry ≥ 8 hr. Cool the closed sample container in a desiccator to room temperature, weigh. Repeat at 1 hr periods until mass changes < 0.1 %.
ASTM D 1348. SCOPE - TEST METHODS FOR MOISTURE IN CELLULOSE BY 2 DIFFERENT PROCEDURES						
Procedure 1. Specimen weighed in oven (dry air passed through oven):						
option	10 - 50	N	105 ± 3	-	Y	Dry 2 hr in oven, weigh and repeat steps at ½ hr periods until mass loss in successive weightings < 0.005g.
Procedure 2. Specimen weighed outside oven (dry air passed through oven):						
option	10	N	105 ± 3	-	N	Dry 2 hr in oven. Cool the closed sample container in a desiccator to room temperature, weigh. Repeat at 1hr intervals until mass loss between successive weightings < 0.005g.
ASTM D 2654. SCOPE - TEST METHODS FOR MOISTURE BY 4 DIFFERENT PROCEDURES						
Procedure 1. Using ambient air for oven drying:						
N	10	N	105 ± 2	Y	Y	Dry until change in sample mass < 0.01 % at 2 hr intervals
Procedure 2. Using air from standard atmosphere for oven drying:						
option	10	Y	105 ± 2	Y	Y	Same as Procedure 1
Procedure 3. Moisture content and pickup at moisture equilibrium:						
Y	10	Y	105 ± 2	Y	Y	Same as Procedure 1
Procedure 4. Moisture regain at moisture equilibrium (after scouring and dried in vacuum oven):						
Y	2-5	Y	65°C in vac.	-	-	Vacuum dry, cool, weigh until change < 0.01 %. Condition overnight, repeat steps at 2 hr intervals until change < 0.01 %.

[z] 70° ± 2°F, 65 ± 2% RH

The precision information on this method (Table 9) was last updated in 1969. For the bias information on the method, there is an admitted small bias (e_1) due to moisture in the standard atmosphere entering the oven, which lowers results. Also, ASTM refers the reader to Test Method D 2654 for more bias information and admits to another bias (e_2) due to other volatiles lost during oven drying.

ASTM D 1348. Originally approved by ASTM in 1954; current edition published October 2003 (ASTM D 1348, 2003). The purpose of the method is to determine the moisture content and regain at bone-dry conditions by passing a stream of dry air through the oven during oven drying. The important moisture related terms are (Table 4): bone dry, moisture content and moisture regain at bone dry condition. Samples may be conditioned after they are taken (Table 5). The oven is controlled at 105 ± 3° C. A standard testing atmosphere and fan-forced ventilation are not necessary since dry air is passed through the oven during drying.

Two procedures are available. One procedure calls for a 10 – 50 g sample and an oven balance. The other uses a 10 g sample and a desiccator following oven drying. For both procedures, the recommended drying time is 2 hr followed by weighing. The steps are repeated until mass loss between successive weighings is < 0.005 g. In the dessicator procedure, the weighing bottles are closed while still in the oven and placed in the desiccator. The bottles are momentarily opened to equalize the pressure during the cooling process, and then weighed when cool.

Since the oven drying time in both procedures is essentially the same, the expected moisture content and regain values for each procedure should not differ significantly. Thus, for both procedures, the same formulas (Tables 7 and 8) are used to compute moisture content (MCBD, Equation 5) and moisture

Table 6. Other standard oven-drying methods summary (lint cotton)

			Oven characteristics			
Condition prior to drying	Sample analyzed (g)	Std. testing[z] atm.	Temp. (°C)	Fan forced vent.	Oven bal.	Drying time
USDA Handbook 442. SCOPE - TEST METHOD FOR MOISTURE IN LINT COTTON DELIVERED TO THE LABORATORY						
N	20	Y	105-110	Y	Y	Dry ≥ 1hr or 15 min intervals until change < 0.1 %.
AATCC 20A. SCOPE - TEST METHOD FOR MOISTURE CONTENT IN TEXTILE MATERIAL						
N	≥ 1	N	105-110	N	N	Dry 1.5 hr. Cool the closed sample container in desiccator, weigh. Repeat heating, cooling and weigh at 30 min intervals until mass constant to < 0.001 g.
Chinese GB/T6102.1. SCOPE – TEST METHOD FOR MOISTURE REGAIN IN RAW COTTON DELIVERED TO THE LABORATORY:						
N	50	option	105 ± 3	Y	Y	Dry and weigh every 15 min until the difference < 0.05 % in successive weightings.
ISO 6741-1. SCOPE – TEST FOR OVEN DRY MASS OF THE CONSIGNMENT IN COTTON DELIVERED TO THE LABORATORY:						
N	≥ 50	Y	105 ± 2	Y	option	Dry and weigh every 5 min until the difference < 0.05 % in 2 successive weightings.

[z] 70° ± 2°F, 65 ± 2% RH

regain (MRBD, Equation 6). Note that bias e_1 is non-existent because dry air enters the oven during the drying period. In the scope of this ASTM standard test method, the reader is reminded that the oven drying approach is to be used where a sample does not contain non-aqueous volatile material at 105° C (i.e., bias e_2 is also nonexistent to prevent elevated results). We do not know the year of the last update for the reported *within* and *between laboratory* precision (Table 9).

ASTM D 2654. Originally published by ASTM in 1967 as D 2654-67 T; last edition published August 1989 (ASTM D2654, 1989) and withdrawn in 1998. These comprehensive test methods for moisture in textiles in addition to fibers, covers four different procedures to eliminate the e_1 and e_2 biases in the oven drying approach. Due to the uniqueness of the specific procedures in D 2654, ASTM D 2495 makes numerous referrals to D 2654. For example, the reader is referred to the 1997 Annual Book of ASTM Standards, Vol 07.01, to locate D 2654 and use those procedures to get more accurate results. For these reasons, we include a review of D 2654 in this paper.

The important moisture related terms are (Table 4): moisture equilibrium, moisture free, moisture pickup, and moisture regain after scouring. There is a wide range in specifications (Table 5) across the four procedures (examples): conditioning prior to drying and standard testing atmosphere, none to required; oven temperature, 105° C to 65° C in vacuum; fan-forced ventilation in oven and oven balance, not used to required; and sample weight, 2 to 10 g. A summary of the four procedures followed by intended use is given below.

Table 7. Moisture related formula in international standards methods by oven drying[z]

ASTM D 2495	
$MC = \frac{OS - (OD - e_1 + e_2)}{OS} x100$	**[Eq. 1]**
$MR = \frac{OS - (OD - e_1 + e_2)}{OD - e_1 + e_2} x100$	**[Eq. 2]**
$MCD = \frac{OS - (D - e_1 + e_2)}{OS} x100$	**[Eq. 3]**
$MRD = \frac{OS - (D - e_1 + e_2)}{D - e_1 + e_2} x100$	**[Eq. 4]**
ASTM D 1348	
$MCBD = \frac{OS - (OBD_1 + e_2)}{OS} x100$	**[Eq. 5]**
$MRBD = \frac{OS - (OBD_1 + e_2)}{OBD_1 + e_2} x100$	**[Eq. 6]**
ASTM D 2654	
$MCE = \frac{OSE - (OD - e_1 + e_2)}{OSE} x100$	**[Eq. 7]**
$MPE = \frac{OSE - (OD - e_1 + e_2)}{OD - e_1 + e_2} x100$	**[Eq. 8]**
$RVD = \frac{RDSV - DSV}{DSV} x100$	**[Eq. 9]**
Chinese GB/T6102.1	
$CMR = \frac{OS - (COD - e_1)}{COD - e_1} x100$	**[Eq. 10]**
ISO 6741-1	
$C = M\left(\frac{OD - e_1 + e_2}{OS}\right)$	**[Eq. 11]**

[z] **In practice, the biases e_1 and e_2 are not in the equations and are ignored or assumed equal to zero.**

Procedures 1 and 2, for moisture content and pickup, are based on drying in ovens, which have different air supplies. Procedure 3, for moisture-equilibrium content or pickup, uses air from the standard atmosphere for conditioning and drying. Procedure 4, for moisture regain, is based on removal of surface materials followed by oven drying in a vacuum oven, and regain of moisture by conditioning. Intended uses: Procedure 1 is intended to give an idea of moisture content, not an accurate measure. It is designed as a tool to reject materials that contain too much moisture. Procedure 2 gives the trade a means to accept a shipment based on dry weight. Procedure 3 generates results specifically at moisture equilibrium with the standard atmosphere. Procedure 4 arrives at the actual moisture regain after surface scouring.

The formulas (Tables 7 and 8) used to compute the various amounts of moisture are: Procedures 1 and 2 (ambient air supply and standard atmosphere supply), Equations 1 and 2; Procedure 3 (at moisture equilibrium), Equations 7 and 8; and Procedure 4 (moisture regain after scouring), Equation 9. Note in the formula that moisture pickup in Procedures 1 and 2 is referred to as regain in Equations 1 and 2. All results are subject to biases e_1 and e_2 (Table 9) except Procedure 4, (see Equation 9). The *within* and *between laboratory* precision for Procedure 1 was generated in 1963.

USDA Agriculture Handbook No. 422. In order to tailor the ASTM methods for ginning research, this handbook on standard procedures for moisture and other fiber tests, was developed at the U.S. Cotton Ginning Laboratory in Stoneville, MS (Shepherd, 1972). This method was cited in recent literature on moisture testing (Byler, 2004). The purpose of the handbook is twofold. First, provide laboratory technicians in ginning research with a set of specific procedures. Second, to standardize the procedures at laboratories concerned with cotton ginning investigations.

The important moisture related term for lint cotton in this method is moisture content, similar to that in ASTM D 2495 (Table 4). The scope calls for determining the amount of moisture in lint cotton delivered to the laboratory, so there is no conditioning prior to analysis (Table 6). Note that the oven is controlled at 105° C to 110° C, which converts to 107.5 ± 2.5° C (Byler, 2004). No rationale is given to explain why these temperature ranges differ from the ASTM procedures in Table 5. The air entering the fan-forced ventilation oven must come from the standard testing atmosphere; an oven balance is required. A 20 g sample is used and calls for drying ≥ 1 hr or until the change in successive weighings at 15 min intervals < 0.1 % of sample mass.

Table 8. Moisture acronyms in international standards test methods by oven drying

QUANTITIES CALCULATED	
ASTM D-2495	
MC	**moisture content**
MR	**moisture regain**
MCD	**moisture content after oven drying and cool closed sample container in a desiccator**
MRD	**moisture regain after oven drying and cool closed sample container in a desiccator**
ASTM D-1348	
MCBD	**moisture content after oven drying at zero % relative humidity**
MRBD	**moisture regain after oven drying at zero % relative humidity**
ASTM D-2654	
MCE	**moisture content at moisture equilibrium**
MPE	**moisture pickup at moisture equilibrium**
RVD	**moisture regain at moisture equilibrium after solvent extraction and vacuum drying in an oven**
Chinese GB/T 6102.1	
CMR	**moisture regain in raw cotton**
ISO 6741-1	
C	**oven dry mass of the consignment (weight)**
QUANTITIES MEASURED (G)	
OS	**mass of original (moist) sample**
OD	**mass of oven dry sample**
D	**mass of sample after oven drying and cool closed container in a desiccator**
OBD	**mass of oven bone-dried sample**
OSE	**mass of original (moist) sample at moisture equilibrium**
DSV	**mass of dried sample after solvent extraction and vacuum oven drying**
RDSV	**mass of sample at moisture equilibrium after solvent extraction and vacuum oven drying**
M	**mass of original (moist) consignment (flexible weight unit)**
OTHER QUANTITIES (G)	
e_1 **(bias)**	**mass of moisture that remains in sample after oven drying**
e_2 **(bias)**	**mass of other volatiles lost during oven drying**

Table 9. Accuracy and precision of standard oven drying methods for lint cotton.

		Precision (%)	
Method	**Accuracy**	***Within Laboratory***	***Between Laboratory***
D 2495 (1969 test data) Procedure 1	**bias: ambient air supplied to oven**	**oven bal./5 reps/0.27**	**oven bal./5 reps/0.90**
Procedure 2	**bias: ambient air supplied to oven**	**desiccator /5 reps/0.22**	**desiccator/5 reps/0.85**
D 1348 Procedure 1	**bias: other volatiles at 105ºC**	**± 0.14 at the 95% confidence level**	**± 0.2 at the 95% confidence level**
Procedure 2	**bias: other volatiles at 105ºC**	**± 0.14 at the 95% confidence level**	**± 0.2 at the 95% confidence level**
D 2654 Procedure 1 (1963 test data)	**bias: ambient air & other volatiles**	**± 0.20 (12 reps)**	**± 0.45 (12 reps)**
Procedure 2	**bias: ambient air & other volatiles**	**To be determined**	**To be determined**
Procedure 3	**bias: ambient air & other volatiles**	**To be determined**	**To be determined**
Procedure 4	**no known bias**	**To be determined**	**To be determined**
USDA Handbk 442	**-[z]**	**± 0.3 (10 reps)**	**-**
AATCC	**-**	**-**	**-**
GB/T6102.1	**-**	**-**	**-**
ISO 6741-1	**-**	**-**	**-**

[z] Not stated in standard method.

The formula for moisture content in this method is Equation 1, also used in ASSTM D 2495. No accuracy information is presented (Table 9). Based on 10 replications, the *within laboratory* precision is 0.3 %.

AATCC 20A. Originally published by AATCC in 1957; current edition published in 2002 (AATCC 20A, 2002). The purpose of this method that relates to moisture is to determine the moisture content of textiles (fibers, yarn and fabric). The term moisture content is equivalent to that in ASTM 2495 (Table 4). Samples are not conditioned prior to testing (Table 6). The oven is controlled at 105º C to 110º C. A standard testing atmosphere, fan-forced ventilation oven and an oven balance are not required.

The procedure calls for at least a 1 g sample. After oven drying for 1.5 hr, the sample container is closed, placed in a desiccator to cool, and then weighed. Heating, cooling, and weighing are repeated at 30 min intervals until the weight is constant to < 0.001 g. Equation 1 (Table 7) is used to compute moisture content. No accuracy or precision results are reported for this method (Table 9).

AUSTRALIA, BRAZIL, INDIA AND S. AFRICA

Although these four countries produce cotton, they represent other nations that do not have their own national standards to measure moisture in lint cotton (Table 3). For convenience, they are listed in alphabetical order. Probably the most widely recognized and used are the ASTM and ISO methods.

In addition to examining the national standards websites, we collaborated with cotton researchers in each of these four countries to confirm the absence of a national standard for moisture in lint cotton. An example of an excellent website that can be used to download and examine the catalogue of South African National Standards (SANS) is provided by the South African Bureau of Standards (SABS): www.sabs.co.za.

COUNTRIES WITH NATIONAL STANDARDS

CHINA

GB/T6102.1. Published by Standards Press of China in 1985 as BG/T6102.1-1985; current edition published September 2006 (GB/T6102.1, 2006). To obtain this National Standard of the People's Republic of China in Chinese and English, visit website: www.ccc-us.com.

China is the major importer of US cotton. In contrast to the ASTM methods used in the USA, moisture is officially defined in the GB standard (Table 4) as "The water and the other volatile substances lost during the drying test on raw cotton". The purpose of the method is to determine moisture regain in raw cotton delivered to the laboratory. Therefore, samples are not conditioned after they are taken (Table 6).

The oven is controlled at 105° ± 3° C. The air entering the fan-forced ventilation oven should come from standard conditions defined as: 20° ± 2° C and 65 ± 3% RH. If the test is run under non-standard conditions, an appendix is provided to correct the result to standard atmosphere. A 50 g sample is run.

The Chinese moisture regain (CMR) is calculated by Equation 10 (Tables 7 and 8). Due to the definition of CMR, the only bias in the formula is e_1. There is no accuracy and precision statement for this method (Table 9).

GERMANY

DIN 53800-1. Although very similar to ISO 6741-1, there is no official relationship between ISO 6741-1 and DIN 53800-1 (2003). The global ISO 6741-1 method is reviewed below rather than the related German standard.

COUNTRIES WITH NATIONAL STANDARDS REPLACED BY ISO 6741-1

Example countries that are in this category and the national standard numbers include: Egypt (ES 2448-1, 2005), France (NF G08-001, 1987**),** Italy (UNI 9213-1, 1990**)** and United Kingdom (BS EN 4784-1, 1988). All three European countries are members of CEN and ISO (Table 1) and use the ISO global standard in addition to their own European Standards (EN).

GLOBAL

ISO 6741-1. Originally published by ISO in 1987; current edition published in 1989. ISO 6741 (1989) consists of the following parts, under the general title "Textiles – Fibres and yarns – Determination of commercial mass of consignments": Part 1: Mass determination and calculations; Part 2: Methods for obtaining laboratory samples; Part 3: Specimen cleaning procedures; and Part 4: Values used for the commercial allowances and the commercial moisture regains. Only Part 1 is reviewed in this paper. The purpose of the standard method is to determine a correct invoice weight, after drying and perhaps cleaning, where the textile material is bought and sold by weight. Included in the corrected invoice, weight is an allowance for moisture, sometimes called the official or standard regains. The USA has a standard regain value of 7.0 % for natural cotton yarn compared to 8.5% for the UK (Saville, 1998).

The important moisture related term is oven dry mass of the consignment (Table 4) for cotton delivered to the laboratory. Consequently, samples are not conditioned before analysis (Table 6). The oven is controlled at 105 ± 2° C. The air entering the fan-forced ventilation oven should come from the standard atmosphere for testing textiles; an oven balance is optional. A large sample is required, ≥ 50 g. If no oven drying duration is specified for the consignment, the sample is dried and weighed every 5 min until the difference in sample mass between two successive weighing is < 0.05 %.

There are two other options in this global standard that are noteworthy. First, if the air entering the oven is not the standard atmosphere for testing, the oven-dry mass may be corrected for non-standard conditions. Second, the procedure is outlined for use when the commercial mass is to be adjusted to a specified non-volatiles extractible content.

The formula to compute the oven dry mass of the consignment is Equation 11 (Table 7). No accuracy or precision information is presented.

STANDARD TEST METHOD BASED ON KARL FISCHER

ASTM D 1348. Originally approved by ASTM in 1954; current edition published October 2003 (ASTM D 1348, 2003). Procedures 1 and 2 are based on oven drying and were reviewed previ-

ously; Procedure 3 is based on Karl Fischer. The purpose of the method is to determine the moisture content – free water and water of hydration – in cellulose (including lint cotton) by titration with Karl Fischer reagent. As stated in the scope of this procedure, it is especially useful with samples containing nonaqueous material volatile at 105° C to 110° C, since such substances interfere in the oven-drying methods.

The general equation in the chemical reaction is

$$H_2O + I_2 + R'OH + 3RN \rightarrow (RNH)SO_4R' + 2(RNH)I$$

where *RN* is an organic base and *R'OH* is an alcohol. Interferences of classes of compounds are eliminated by chemical reactions to form inert compounds prior to titration or by careful selection of the solvent (alcohol) in the Karl Fischer reagent. The preferred reagent for titration is Hydranal® (Hoffmann and Schoffski, 2005), sometimes referred to as a one component method because all the needed chemicals are in the titrant.

The important moisture related terms are (Table 10): percent moisture and dry basis regain. Samples

Table 10. ASTM D 1348 standard test method by Karl Fischer titration.

MOISTURE TERMINOLOGY			
Term	**Definition**		
percent moisture	**the amount of moisture (free water and of hydration) in cellulose after alcohol extraction and titration with Karl Fischer reagent**		
dry basis regain	**the amount of moisture in cellulose determined by Karl Fischer and expressed as a percentage of the extracted material after drying**		
METHODS SUMMARY[y]			
Condition prior to analysis	**Sample analyzed (g)**	**Extraction time (min)**	**Oven drying**
option	**≈ 2.75**	**15 - 20**	**Dry extracted sample at 120° C for 30 min, cool, weigh**
MOISTURE RELATED FORMULA (direct titration of aliquot) and ACRONYMS			
$PM = \frac{(C-B)xTxR + e_3}{OS} x100$ **[Eq. 12]**			
$DBRKF = \frac{PM}{100 - PM} x100$ **[Eq. 13]**			
$DBRZ = \frac{OS - Z}{Z} x100$ **[Eq. 14]**			
PM	**percent moisture**		
DBRKF	**dry basis regain, Karl Fischer titration, %**		
DBRZ	**dry basis regain, after extraction and oven drying, %**		
C	**ml of Karl Fischer reagent required for titration of the sample aliquot**		
B	**ml of Karl Fischer reagent required for titration of 50 ml reagent**		
T	**g of water equivalent to 1 ml of the reagent**		
R	**aliquot factor**		
OS	**(see Table 8)**		
Z	**g of dried cellulose**		
***e*3 (bias)**	**mass of bound water that remains in the fiber after alcohol extraction; in practice, this bias (Equations 12 and 13) is ignored**		
ACCURACY AND PRECISION			
		Precision (std dev, %)	
Accuracy		***Within Laboratory***	***Between Laboratory***
-[z]		**0.41**	**-**

[y] Methods titration options: titrate entire supernatant or aliquot, direct or back titration.

[z] Not stated in standard method

may be conditioned after they are taken. A 2 to 3 g sample is recommended for lint cotton. The sample is extracted with anhydrous methanol. Generally an aliquot is transferred to the titration cell rather than the entire supernatent. The end point is best determined electrometrically. Dry basis regain calls for drying the extracted fibers in an oven at 120° C for 30 min, cool and weigh. No accuracy results are reported (Table 10); the *within laboratory* precision is 0.41 % (standard deviation).

The formulas (Table 10) used to compute percent moisture and dry basis regain are given by Equations 12, 13 and 14. Results by Equations 12 and 13 are subject to bias e_3, the mass of bound water that remains in the fiber after alcohol extraction.

COMPARABILITY OF STANDARD TEST METHODS BY OVEN-DRYING

The standard test methods used in five of the eleven countries presented in this review are not national standards and must rely on either voluntary standards (e.g., ASTM) or the global procedure (ISO 6741-1). These countries are: Australia, Brazil, India, S. Africa and the USA. Two of the eleven countries have national standards: China and Germany. The remaining four had national standards superceded by the global ISO method: Egypt, England, France and Italy.

There are wide ranges in the oven method specifications in the reviewed standard methods. The sample analyzed varies from 1 to > 50 g. Parameters such as conditioning prior to drying, a standard testing atmosphere, fan-forced ventilation or an oven balance may not be required, or are optional or necessary. Generally the oven temperature is set at 105° C to 110° C. Specified oven drying time ranges from not specified (i.e., based on oven performance) to > 8 hr.

The definition of *moisture* has not been standardized across the reviewed standard test methods. Associated terminology presented in this review includes: oven dry, moisture content, moisture regain, bone dry, moisture content at bone dry conditions, moisture regain at bone dry conditions, moisture equilibrium, moisture free, moisture pickup, moisture regain after scouring, and oven dry mass of the consignment.

Consequently, an unusually large number of different moisture quantities are calculated in the test methods. To help understand these differences and provide for direct comparability between standards, simple formulas are compiled in this review. The formulas are presented in terms of the quantities actually measured and the known biases. In the standard procedures, these biases are assumed to be equal to zero, ignored, or the procedure itself eliminates a specific bias.

In terms of round trials, there is no quantitative accuracy information; indeed, the known biases are readily admitted in the standards. Between laboratories precision is generally poor or lacking. The exception is ASTM D 1348 (Procedures 1 and 2) in which dry air rather than ambient or conditioned air is passed through the oven.

SOURCES OF ERROR IN OVEN DRYING METHODS

This section includes the review of six papers presented in chronological order since the work builds on other studies. Publication year ranged from 1930 to 2004; earlier studies not covered in this review date back to 1909.

For the oven-drying procedure, the first inherent source of bias in the method (Davidson and Shorter, 1930) is residual moisture that remains in the cotton (e_1, see Table 8) since the absolute humidity of the ambient air is not zero. The second is substances, other than water, driven out of the sample (e_2, Table 8). For raw cotton, they estimated that the amount of error due to each cause, may be of the order of 0.2 %. To obtain the most accurate results at the Shirley Institute, samples were dried in an enclosed space containing the drying agent, phosphorus pentoxide, at room temperature. Since the temperature is not raised, other changes in the sample are not likely to occur. However, four to six weeks were necessary to dry 8-g samples of cotton.

Stephenson (1938) found that the transfer of the sample from the oven to an external balance increased the dry weight by as much as 0.2 %. Also, because warmer air is less dense than cooler air, the cotton sample weight was lower than the true weight. A difference of 7°C was found to exist between the center of the cotton and that of the surrounding air. On a large sample weighing 500 grams, the error was 0.4 %, while with 250-g samples, an error of 0.6 % was noted. The total error of the dry weights varied from a negative bias of 0.5 % to a positive 0.2 %.

Balls (1950) published a survey paper about random errors in testing Egyptian bales for mois-

ture by the oven-drying technique. Four types of random error were considered: in the oven test itself; parallel sampling of the same bale by two independent technicians; bales in the same lot; and the pressure in the bale at the press head. The standard deviation of the test itself was quite good, 0.11 %, based on testing ten 1 lb samples. The main source of error in the test is probably due to poor circulation of hot air within the cotton mass. The other three random error results, relative to the oven test standard deviation were (dimensionless): parallel sampling, 2.3; between bales, 4.0; and press head, 4.3.

Terrell (1967a,b), who served on ASTM Committee D-13 on Textiles, summarized the available history of the work done with cotton, wool and synthetic fibers, yarn and fabric to measure moisture content by oven drying (Sub-committees A-3, B-1 and B-3). Only those results related to cotton fibers are included in this review, unless otherwise noted.

In his first paper (Terrell, 1967a) summarized seven ASTM reports. *First*, an ASTM study in 1946 used five different ovens to measure the moisture in cotton fibers, which had been scoured prior to testing to exclude all volatile matter other than moisture. The averaged moisture content results, based on 15 to 20 specimens, ranged from 6.95% to 7.88% (a spread of 0.93%), and a between oven standard deviation of 0.4. Within-oven standard deviations ranged from 0.07% to 0.17%. *Second,* in 1948 a task group concluded that a temperature of 127° C is necessary to remove all water and subsequently a loss of 0.3% to 0.6% of volatile matter other than water. *Third*, in 1949 a task group determined that distillation in an immiscible solvent was the best reference method. *Fourth,* in 1951 a task force reported that octane and toluene distillation gave the most accurate results and that without an accurate extraction method all oven-drying methods are suspect. *Fifth*, later in 1951 another study, based on split samples in which one set was extracted before oven drying, indicated that almost as much non-aqueous volatiles were removed at 105°C as at 135°C. *Sixth*, in a 1952 study inter-laboratory differences were found to be significant; and *seventh*, in a 1953 report, differences between 5 labs were again found to be significant using drying at 105° C.

In the second paper (Terrell, 1967b), an inter-laboratory test in 1964 involved a polyester, a polyamide, cotton, viscose, and wool. Four laboratories participated. Each laboratory ran a total of six specimens of each of the five materials. The laboratory average for moisture content of cotton fibers ranged from 4.07 % to 4.89 %, with a range of 0.82 % and a *between laboratory* standard deviation of 0.36 %. An analysis of variance of results for the total experiment gave a disappointing between lab component of variance about nineteen times greater than the *within laboratory* variance component. A similar study conducted in 1963 with only wool revealed a *between laboratory* component of variance more than twenty times greater than the *within laboratory* variance component. Terrell concluded that moisture content of textile materials as determined with the oven-drying method is affected by significant sources of error, which had not been satisfactorily identified nor eliminated.

Near infrared analysis (Taylor, 1988) was employed to measure the moisture content of raw cotton and correlated to moisture as determined by two calibration methods. One method was the ASTM 2495 oven drying procedure and the other by desiccation over calcium sulfate at room temperature for eight days. Volatile loss by desiccation at room temperature was less than comparable oven drying data. NIR moisture calibration coefficients of determination (R^2) for each calibration method were (N = 26): desiccator drying, 0.990; and oven drying, 0.986. Standard error of prediction and NIR bias (N = 68), respectively, for the calibrations were: desiccator drying, 0.268 % and 0.51 %; and oven drying, 0.326 % and -0.08 %. Re-drying the desiccated cottons in the oven resulted in an additional averaged weight loss of about 0.7 %, which confirmed the differences in bias.

Byler (2004) noted that the USDA Handbook No. 42 by Shepherd (1972) calls for 20-g specimens of lint in the oven-drying method, which is more than is available in many occasions. The use of smaller specimens was considered. Based on the variance of the moisture content data, seven 1-g specimens produced the same variance as three 20-g specimens after 1 h of oven drying, consuming 88 % less material. There was not a statistically significant difference in means based on 1 and 20 g specimens. However, the 1-g sample procedure required more time and effort to achieve a similar variance. Additionally, oven-drying data was observed after 2.5 hr in the oven and the results suggested that the longer time in the oven drove off additional weight, either water or other volatiles.

EMERGING TRENDS AND CURRENT ISSUES OF OVEN DRYING

EMERGING TRENDS

The overall trends in voluntary, national and global standard test methods for moisture in lint cotton by oven drying include: publication of new editions of the methods without updating the required accuracy and precision statements, admitting to the biases e_1 (mass of moisture that remains in specimen after oven drying and e_2 (mass of other volatiles loss during oven drying), and modifications in the procedures without supporting information on means and variance of results. Examples of this presented in this review include: ASTM 2495 (2001) and the Chinese GB/T6102.1 (2006). This particular ASTM procedure was last revised in 2001 yet the statistical statement is based on a 1969 inter-laboratory test; biases e_1 and e_2 are declared. The Chinese method actually defines moisture as "the water and other volatiles lost during the drying test …". A major revision in the Chinese method compared to the 1985 version is the cancellation of a specified drying time with the time of drying determined by the performance of the oven itself. No results are presented comparing the 1986 and 2006 versions.

CURRENT ISSUES

Clearly, the major issue is the need for accurate information in the standard test methods that addresses the biases e_1 and e_2. This must be obtained with current varieties of USA and international cottons. Intra and inter-laboratory comparisons presented in this review also indicated some precision issues that remain unresolved. A reviewer of this paper emphasized that unless RH measurements are calibrated by a standard method, there may be a systematic error in comparing inter-laboratory measurements.

The issue of variability in RH measurements is being addressed by this laboratory. To detect any changes in RH in the standard conditioned laboratory, a 1 g sample in an open weighing bottle is kept in near proximity to the analytical balance. This provides a direct way to determine if a change in environmental conditions makes a measurable difference in the recorded weights in the oven drying technique (Montalvo and Von Hoven, 2008).

If there is to be a true standard reference method, acceptable accuracy and precision results are a prerequisite, particularly if indirect methods are to be developed based on these standards. The error from the indirect methods will only compound with those of the reference method.

The published work on accuracy of the oven test methods, as presented in this review, ceased in the 1960's. The paper by Taylor (1988) is the only exception. To confirm these findings, we reviewed four other sources of information dealing with reviews and recent developments in the testing of textiles (Morton and Hearle, 1993; Slater, 1993; Steadman, 1997; and Saville, 1998). No additional papers were found regarding accuracy and precision of the oven-drying test.

CONCLUSIONS

The standard test methods for measuring moisture in lint cotton in eleven countries were reviewed. These countries include Australia, Brazil, China, England, Egypt, France, Germany, India, Italy, S. Africa, and the USA. Examples of these standards presented in this review include voluntary methods (e.g., ASTM 2495), national (Chinese GB/T6102.1) and global (ISO 6741-1). All are based on oven-drying except one, ASTM D 1348 (Procedure 3), by Karl Fischer titration of an aliquot after alcohol extraction of the water.

The oven standards readily admit to two biases without specifying their expected magnitudes. These biases are moisture that remains in the sample due to moisture in the air supply entering the oven and other volatiles lost during drying. Examples of standard procedures presented in this review to eliminate the biases include ASTM D 1348 (Procedures 1 and 2), which uses dry air passed through the oven to eliminate the first bias. ASTM D 2654 (Procedure 4), after scouring and drying in a vacuum oven to eliminate the second bias, measures moisture regain at moisture equilibrium. A third bias, bound water that remains in the sample, occurs with ASTM D 1348 (Procedure 3) by Karl Fischer titration.

There is a general lack of recently published information on the accuracy and precision of the standard methods reviewed, especially inter-laboratory results, with the current varieties of cotton grown in this country and abroad. This review demonstrates that more research is needed to develop more accurate and precise standard methods for moisture in lint cotton. After reviewing all the methods, and based on our research of various methods, we conclude that measurement by oven evaporation and

Karl Fischer titration is a useful alternative to the standard oven drying techniques (Montalvo and Von Hoven, 2008). In the new technique, the sample is placed in a closed glass container and heated in an oven. Moisture released is transported by a dry carrier gas into the Karl Fischer reaction cell where it is titrated with iodine. The end point is determined by platinum indicating electrodes.

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