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# Standard Test Method for Moisture in Wool by Oven-Drying[1]

This standard is issued under the fixed designation D 1576; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of the amount of moisture present in ordinary commercial and industrial samples of wool in all forms except grease wool, using the oven-drying technique.

1.2 Formulas for calculating the moisture content (as-received basis) and moisture regain (oven-dried basis) are given. It is always important to use the correct term which corresponds to the basis used in the calculation (see 12.2.1).

NOTE 1—The determination of moisture content for textile materials in general is covered in Test Methods D 2654, and an optimal method for determining the moisture in wool by distillation with toluene is covered in Test Method D 2462. A method for sampling wool for the determination of moisture in wool is covered in Practice D 2525. The oven-drying method has been adapted for cotton in Test Method D 2495.

1.3 *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.*

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 123 Terminology Relating to Textiles[2]

D 1060 Practice for Core Sampling of Raw Wool in Packages for Determination of Percentage of Clean Wool Fiber Present[2]

D 1776 Practice for Conditioning Textiles for Testing[2]

D 2258 Practice for Sampling Yarn for Testing[2]

D 2462 Test Method for Moisture in Wool by Distillation with Toluene[2]

D 2495 Test Method for Moisture in Cotton by Oven-Drying[2]

D 2525 Practice for Sampling Wool for Moisture[2]

D 2654 Test Methods for Moisture in Textiles[3]

D 3333 Practice for Sampling Man-Made Staple Fibers, Sliver, or Tow for Testing[4]

## 3. Terminology

3.1 *Definitions:*

3.1.1 *grease wool, n*—wool taken from the living sheep and which has not been commercially scoured.

3.1.2 *moisture content, n*—the amount of moisture in a material determined under prescribed conditions and expressed as a percentage of the mass of the moist material, that is, the original mass comprising the oven-dried substance plus any moisture present.

3.1.2.1 *Discussion*—The term "mass" is the correct designation for the property commonly designated as "weight."

A slight amount of residual moisture may not be removed from a specimen subjected to oven drying because of the relative humidity of the ambient air. The amount of moisture retained by a specimen may be estimated from published data.[5]

There may also be a slight additional loss in mass caused by the evaporation of volatile material other than water, the amount depending on the characteristics of any added oils or emulsions.

3.1.3 *moisture-free, adj*—the condition of a material that has been exposed in an atmosphere of desiccated air until there is no further significant change in its mass (see 3.1.2).

3.1.3.1 *Discussion*—Heating the material and the desiccated air to temperatures as high as 110°C increases the rate of moisture loss but does not change the final equilibrium mass of the moisture-free material.

3.1.4 *moisture regain, n*—the amount of moisture in a material determined under prescribed conditions and expressed as a percentage of the mass of the moisture-free material (see moisture content).

3.1.4.1 *Discussion*—In this test method, the material is considered to be oven-dried after drying as described in Section 10.

3.1.5 *oven-dried, adj*—the condition of a material that has been heated under prescribed conditions of temperature and humidity until there is no further significant change in its mass (see 3.1.2).

3.1.5.1 *Discussion*—An oven-dried material will retain a small amount of moisture which is dependent on the temperature and relative humidity of the atmosphere in contact with the material during the drying process. An oven-dried material will only be moisture-free when the air supplied to the drying oven has been previously desiccated.

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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 D13.13 on Wool and Wool Felt.

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[2] *Annual Book of ASTM Standards*, Vol 07.01.

[3] Discontinued; see *1997 Annual Book of ASTM Standards*, Vol 07.01.

[4] *Annual Book of ASTM Standards*, Vol 07.02.

[5] Toner, R. K., Bowen, C. F., and Whitwell, J. C., "Equilibrium Moisture Relations for Textile Fibers," *Textile Research Journal*, Vol 17, January 1947, pp. 7 to 18.

3.1.6 *pulled wool*, *n*—wool taken from the pelt of a slaughtered sheep and which has not been commercially scoured. (syn. *slipe wool*, *skin wool*).

3.1.7 *raw wool*, *n*—wool or hair of the sheep in the grease, pulled, or scoured state. (See also *scoured wool*.)

3.1.8 *recycled wool*, *n—as defined in the Wool Products Labeling Act as amended in 1980*, "the resulting fiber when wool has been woven or felted into a wool product which, without ever having been utilized in any way by the ultimate consumer, subsequently has been made into a fibrous state, or the resulting fiber when wool or reprocessed wool has been spun, woven, knitted, or felted into a wool product which, after having been used in any way by the ultimate consumer, subsequently has been made into a fibrous state."

3.1.8.1 *Discussion*—In the amended Act of 1980, the term" recycled wool" replaced the terms "reprocessed wool" and" reused wool."

3.1.9 *scoured wool*, *n*—wool from which the bulk of impurities has been removed by an aqueous or solvent washing process.

3.1.9.1 *Discussion*—Although it is no longer in its original raw state, scoured wool is generally accepted as raw wool.

3.1.10 *virgin wool*, *n—as defined in the Wool Products Labeling Act*, "the terms ′virgin' or ′new' as descriptive of a wool product, or any fiber or part thereof, shall not be used when the product or part so described is not composed wholly of new or virgin fiber which has never been reclaimed from any spun, woven, knitted, felted, braided, bonded, or otherwise manufactured or used product".

3.1.11 *wool*, *n*—the fibrous covering of the sheep, *Ovis* species.

3.1.11.1 *Discussion*—For the purposes of this method, the word *wool* is used in the generic sense, and includes both *wool* as defined in the Wool Products Labeling Act of 1939 as well as recycled wool as defined in the amended Act of 1980.

3.1.12 *wool*, *n—as defined in the Wool Products Labeling Act of 1939*, "the fiber from the fleece of the sheep or lamb, or hair of the Angora goat or Cashmere goat (and may include the so called specialty fibers from the hair of the camel, alpaca, llama, and vicuna) which has never been reclaimed from any woven or felted wool product".

3.1.13 For the definition of other textile terms used in this method, refer to Terminology D 123.

## 4. Summary of Test Method

4.1 A specimen of wool material is weighed and then dried to constant mass at 105 ± 2°C in an oven supplied with ambient air. The loss in mass is considered moisture and reported as either moisture content or moisture regain. Directions are given for the adjustment of the observed results for any change in the moisture content after sampling and before drying.

## 5. Significance and Use

5.1 Test Method D 2462 for the determination of the moisture in wool by distillation with toluene is the preferred method for testing wool for moisture for the acceptance testing of commercial shipments. If, however, the purchaser and the supplier agree, Test Method D 1576 for the determination of the moisture in wool by oven drying may be used instead. Comparative tests as directed in 5.1.1, may be advisable.

5.1.1 In case of a dispute arising from differences in reported test results when using Test Method D 1576 for acceptance testing of commercial shipments, the purchaser and the supplier should conduct comparative tests to determine if there is a statistical bias between their laboratories. Competent statistical assistance is recommended for the investigation of bias. As a minimum, the two parties should take a group of test specimens which are as homogeneous as possible and which are from a lot of material of the type in question. The test specimens should then be randomly assigned in equal numbers to each laboratory for testing. The average results from the two laboratories should be compared using Student's *t*-test for unpaired data and an acceptable probability level chosen by the two parties before testing is begun. If a bias is found, either its cause must be found and corrected or the purchaser and the supplier must agree to interpret future test results in the light of the known bias.

5.2 This test method is a simple and convenient method for routine process control, in-plant evaluation, estimation of moisture content of a lot of wool, or any other purpose for which a high degree of reproducibility is not necessary (see Section 13).

## 6. Apparatus

6.1 *Oven*, ventilated and thermostatically controlled in the temperature range of 105 ± 2°C throughout the enclosure. The oven may be of either the forced draft or the convection type.

6.2 *Weighing Containers*, of perforated metal if weighing is to be performed in the drying enclosure; or containers that can be hermetically sealed (such as glass weighing bottles) if the specimen is to be cooled in a desiccator before weighing in the ambient atmosphere.

6.3 *Sampling Containers*, capable of being sealed. Mason jars have been found to be satisfactory where the sample size is not too great. For larger samples, bags of various plastic materials may be suitable if the wall thickness is sufficient to provide a good moisture vapor barrier (at least 4 mil (approximately 0.1 mm) for polyethylene, for example).

6.4 *Balance*, having a capacity adequate for weighing specimens and containers, and a sensitivity of 0.005 g.

## 7. Sampling

7.1 *Lot Sample*—As a lot sample for acceptance testing, take at random the number of shipping containers directed in applicable material specification or other agreement between the purchaser and the supplier, such as an agreement to use Practice D 2525 for bales of fiber and containers of top or sliver or to use Practice D 2258 for beams or cases of yarn. Consider shipping containers to be the primary sampling unit.

NOTE 2—An adequate specification or other agreement between the purchaser and supplier requires taking into account the variability between shipping containers, between laboratory sampling units within a shipping container, and test specimens within a laboratory sampling unit to produce a sample plan with a meaningful producer's risk, consumer's risk, acceptable quality level, and limiting quality level.

7.2 Use extreme care to prevent gain or loss of moisture during the sampling operation and the transfer of material to

the sampling container. Weigh each portion of the sample and its container immediately after sampling. Subtract the tare mass of the container to obtain the net mass at time of sampling, M.

7.3 *Laboratory Sample*—As a laboratory sample for acceptance testing, proceed as follows:

7.3.1 For wool fiber, take laboratory samples as directed in Practice D 1060 for cored samples or Practice D 3333 for hand samples.

7.3.2 For wool sliver or top, from each shipping container in the lot sample, take one ball of top. From this ball of top, take approximately 2 m from the inside and 4 m from the outside of the ball.

7.3.3 Take laboratory sampling units which weigh a minimum of 50 g. Follow the instructions in Practice D 2525 for reduction of the laboratory samples to specimens.

NOTE 3—Condition the laboratory samples as directed in Section 9 before preparing the specimens from them.

## 8. Number of Specimens

8.1 Take a number of specimens per laboratory sampling unit that the user can expect at the 95 % probability level that the test result for a laboratory sampling unit will be no more than 0.5 percentage points above or below the true average for the laboratory sampling unit. Determine the number of specimens per laboratory sampling unit as follows:

8.1.1 *Reliable estimate of s*—when there is a reliable estimate of *s* based upon extensive past records in the user's laboratory as directed in the test method, calculate the required number of specimens per laboratory sampling unit using Eq 1:

$$n = (ts/E)2 \quad (1)$$

where:

- $n$ = number of specimens per laboratory sampling unit (rounded upward to a whole number),
- $s$ = reliable estimate of the standard deviation of individual observations on similar materials in the user's laboratory under conditions of single operator precision,
- $t$ = the value of Student's $t$ for two-sided limits, a 95 % probability level, and the degrees of freedom associated with the estimate of v, and
- $E$ = 0.5 percentage points, the allowable variation.

8.1.2 *No Reliable Estimate of s*—When there is no reliable estimate of *s* for the user's laboratory, do not use Eq 1 directly. Instead, specify the fixed number of six specimens per laboratory sampling unit. This number of specimens per laboratory sampling unit is calculated using $s = 0.60$ percentage points which is a somewhat larger value of *s* than is usually found in practice. When a reliable estimate of *s* for the user's laboratory becomes available, Eq 1 will usually require fewer than six specimens per laboratory sampling unit.

## 9. Conditioning

9.1 Condition the lot sample (or laboratory sample(s)) by exposure to moving air in the laboratory atmosphere in which the testing is to be done, until equilibrium for testing is achieved.

NOTE 4—Preconditioning and conditioning as directed in Practice D 1776 is acceptable but not necessary, since the object of the conditioning for the purpose of this test is merely to stabilize the sample, that is, to bring all parts of the sample to moisture equilibrium with the prevailing atmosphere in order that changes in moisture level will not occur while the specimens are being prepared and weighed.

9.2 Weigh the conditioned sample(s) to the nearest 0.005 g and record the net mass(es), *W*.

NOTE 5—The net mass of the conditioned sample, *W*, and the net mass at the time of sampling, *M*, will be used to convert the observed moisture content of the conditioned specimen to the moisture content at time of sampling.

9.3 From the weighed conditioned sample(s), take the appropriate size specimen(s) and weigh to the nearest 0.005 g to obtain the specimen mass *B*.

## 10. Procedure

10.1 Place the specimen(s) in the oven in a suitable container and dry to constant mass, defined as the absence of any progressive decrease in mass in excess of 0.10 % of the average as determined by three successive weighings using the procedure in either 10.1.1 or 10.1.2 to obtain the oven-dry mass of specimen, *D*.

10.1.1 If the weighings of the dried specimen(s) are to be obtained with the specimen(s) inside the oven, perform the weighings with any forced-air circulation turned off. Space the weighings so that the drying intervals between readings will be equal to 20 % of the normal cycle with a minimum interval of 5 min. Determine the normal cycle by running rate-of-drying curves for similar specimens using the equipment under the same conditions that will be used for ordinary testing. Continue readings of mass until the conditions specified in 10.1 are achieved.

10.1.2 If the weighings of the dried specimen(s) are to be obtained outside the oven, dry the specimen(s) in a container provided with a tight-fitting cover with this cover removed while in the oven. At the end of the drying period, cover the container and remove it from the oven. Place the covered container in a desiccator, loosen the cover, and cool the specimen and container to approximately room temperature. When cooling is completed, set the cover firmly on the container and weigh the container, cover and specimen. Replace the container and specimen in the oven, remove the cover, and dry for an additional 30 % of the normal cycle. Repeat the cooling and weighing procedures. Continue this procedure until the conditions specified in 10.1 are achieved.

## 11. Calculation

11.1 Calculate to the nearest 0.01 percentage point the percent moisture present in the sample as taken, using Eq 2 for moisture content or Eq 3 for moisture regain.

$$\text{Moisture content, percentage points} = [1 - ((W \times D)/(M \times B))] \times 100 \quad (2)$$

$$\text{Moisture regain, percentage points} = [((M \times B)/(W \times D)) - 1] \times 100 \quad (3)$$

where:
$M$ = net mass of subsample at time of sampling,
$W$ = net mass of subsample at time of measurement,
$B$ = net mass of specimen before drying, and
$D$ = oven-dry mass of specimen.

11.2 Calculate the average moisture content (or moisture regain) of all specimens tested for one lot to the nearest 0.1 percentage point.

11.3 The following equations may be used to convert moisture regain in percentage points to moisture content in percentage points and vice versa:

$$R = [C/(100 - C)] \times 100 \quad (4)$$

$$C = [R/(100 + R)] \times 100 \quad (5)$$

where:
$R$ = moisture regain, percentage points, and
$C$ = moisture content, percentage points.

## 12. Report

12.1 State that the specimens were tested as directed in Test Method D 1576. Describe the material or product sampled and state the method of sampling used.

12.2 Report the following information:

12.2.1 The average value of the results calculated for a particular lot to the nearest 0.1 percentage point, stating whether the reported value is the moisture content or the moisture regain.

12.2.2 The number of specimens tested.

12.2.3 The range of the moisture contents or moisture regains (difference between the largest and smallest observed results).

## 13. Precision and Bias

13.1 *Interlaboratory Test Data*[6]—An interlaboratory test was carried out in 1963 in which 4 laboratories tested 12 specimens each of a nominally uniform wool material for moisture content. The components of variance expressed as standard deviations were calculated to be:

| | |
|---|---|
| Within-laboratory component | 0.236 percentage point |
| Between-laboratory component | 0.469 percentage point |

13.1.1 The within-laboratory component includes the single-operator component which was not determined separately. The components listed above do not include any sampling error. This error must be added in any application of the method.

13.2 *Precision*—For the components of variance reported in 13.1, two averages of observed values should be considered significantly different at the 95 % probability level if the difference equals or exceeds the critical differences listed below:

| | Critical Differences, Percentage Points, for the Conditions Noted[A,B] | |
|---|---|---|
| Number of Observations in Each Average | Within-Laboratory Precision | Between-Laboratory Precision |
| 2 | 0.46 | 1.38 |
| 3 | 0.38 | 1.35 |
| 5 | 0.29 | 1.33 |
| 10 | 0.21 | 1.32 |

[A] The values for the critical differences were calculated using $t = 1.960$ which is based on infinitedegrees of freedom.

[B] The values of the critical differences listed constitutes a general statement particularly with respect to between-laboratory precision. Before a meaningful statement can be made about two specific laboratories, the amount of statistical bias, if any, between them must be established, with each comparison being based on recent data obtained on randomized specimens from one sample of the type of material to be tested.

13.3 *Bias*—The procedure in Test Method D 1576 for determination of the amount of moisture present in wool by oven-drying has no bias because the value of that property can be defined only in terms of a test method.

## 14. Keywords

14.1 moisture content and wool

[6] Supporting data are available on loan from ASTM Headquarters, 100 Barr Harbor Drive, Conshohocken, PA 19428. Request RR:D13-1016.