



Standard Test Method for Moisture in Wool by Distillation With Toluene¹

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1. Scope

1.1 This test method covers the determination of the amount of moisture present in grease wool, scoured wool, carded wool, garnetted wool, wool top and intermediate wool products, and rovings, by distillation with toluene.

1.2 Equations are given for calculating the amount of water present as moisture content (as-received basis) and moisture regain (dry fiber) basis. The term that corresponds to the basis used in the calculation and report must always be stated.

1.3 This test method is not applicable to material known to contain any steam-distillable, water-soluble matter. If it is suspected that such matter is present, the method should be used with caution.

1.4 Xylene or other solvents should not be substituted for toluene as no other solvents have been evaluated for use in this standard.

NOTE 1—The determination of moisture in wool by oven-drying is covered in Test Method D 1576 and for textile materials in general in Test Methods D 2654. A method for sampling wool for the determination of moisture in wool is covered in Practice D 2525.

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 safety hazard statements, see Section 8.

2. Referenced Documents

2.1 ASTM Standards:

D 123 Terminology Relating to Textiles²

D 1060 Practice for Core Sampling of Raw Wool in Packages for Determination of Percentage of Clean Wool Fiber Present²

D 1576 Test Method for Moisture in Wool by Oven-Drying²

D 1776 Practice for Conditioning Textiles for Testing²

D 2258 Practice for Sampling Yarn for Testing²

D 2495 Test Method for Moisture in Cotton by Oven-Drying²

D 2525 Practice for Sampling Wool for Moisture²

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.

D 2654 Test Methods for Moisture in Textiles²

D 3333 Practice for Sampling Man-Made Staple Fibers, Sliver, or Tow for Testing³

E 123 Specification for Apparatus for Determination of Water by Distillation⁴

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. 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 Discussion under 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

³ *Annual Book of ASTM Standards*, Vol 07.02.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

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.

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 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 definitions of other textile terms used in this test method, refer to Terminology D 123.

4. Summary of Test Method

4.1 A sample or specimen of wool or material made of wool is weighed, then stabilized in the laboratory atmosphere in which the specimen is prepared, and reweighed. Any resulting change in mass is used to calculate the original moisture content of the sample or specimen from the results observed on the stabilized specimen.

4.2 The specimen is immersed in water-saturated toluene which is then heated, the distilled water vapor and solvent vapor are condensed and collected in a graduated trap, wherein the water separates and settles to the bottom. After cooling in a water bath to achieve a specified temperature, the volume of water collected in the trap is read from the graduated trap and converted to its equivalent weight.

4.3 Results are calculated as percent moisture content or percent moisture regain using the appropriate equation.

5. Significance and Use

5.1 Test Method D 2462 for testing for moisture in wool is considered satisfactory for acceptance testing of commercial shipments since current estimates of between-laboratory precision are acceptable.

5.1.1 In case of a dispute arising from differences in reported test results when using Test Method D 2462 for acceptance testing of commercial shipments, the purchaser and the supplier should conduct comparative testing to determine if there is a statistical bias between their laboratories. Competent statistical assistance is recommended for the investigation of the bias. As a minimum, the two parties should take a group of test specimens that are as homogenous as possible and that are from a lot of the type material in question. The test specimens should be 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 acceptance probability level chosen by the two parties before the test 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 light of the known bias.

5.2 This test method is the preferred method for all suitable samples of wool where it is important to obtain a result free from the possible biases, introduced by the conditions discussed in 5.3 and 5.4.

5.3 This test method is free from the interferences caused by different conditions of ambient atmosphere such as might affect the results of oven-drying. A slight amount of residual moisture may be retained in a specimen subjected to oven-drying because of the relative humidity of the ambient air; however, the amount of moisture retained may be estimated from published data.⁵

5.4 This test method is free from the interference caused by nonaqueous volatile material. Such material, when present, is erroneously measured as moisture by oven-drying methods, the extent of the error depending upon the amount and characteristics of any added oils or finishes.

5.5 This test method is relatively cumbersome, time consuming, and costly compared to oven-drying, and is not recommended for routine process control, in-plant evaluations, or for other purposes where a high degree of accuracy is not necessary. The cost of operation can be reduced somewhat by redistilling the used toluene, which is then suitable for reuse without further treatment.

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. 7–18.

5.6 Unlike an oven-drying method, any moisture gained or lost by a specimen after its mass has been determined will appear as a direct error in the final result. Since one of the principal uses of the method is to determine the average moisture present in large lots of wool or wool products exposed to variable atmospheric conditions, numerous laboratory samples and test specimens are common. To avoid errors of the type mentioned above, this procedure includes provisions for stabilizing the sample(s) in the laboratory atmosphere so that, during the time necessary for selecting, weighing, and transferring the specimens to flasks, gain or loss of moisture which cannot be accounted for will be minimized. A further advantage of the stabilizing process is realized in cases where the interest is solely in the average moisture content of the sample, and the actual moisture content within the sample is highly variable. By stabilizing the sample before selecting the specimens, equal precision can be achieved with fewer specimens.

6. Apparatus

6.1 *Flask, Erlenmeyer*, wide-mouth, 1000 cm³ (mL) capacity (takes a No. 11 stopper).⁶

6.2 *Distilling Receiver, Dean & Stark*, 10 cm³ (mL) capacity, graduated in 0.1 cm³ (mL).⁷

NOTE 2—The tolerance on the accuracy of the graduations specified in Specification E 123 for this apparatus is $\pm 0.1 \text{ cm}^3$. If greater accuracy is required for a test result, the graduated trap(s) used should be calibrated.

NOTE 3—Illustrations of acceptable forms of the glass apparatus required by this method appear in Specification E 123.

6.3 *Condenser, Liebig*, sealed, with 500-mm jacket.

6.4 *Balance*, capacity of at least 500 g with a sensitivity of 0.05 g.

6.5 *Heater*, for distillation apparatus, electrical with variable heat control, and arranged so that the surface of the flask above the lowest solution level is not heated by direct radiation.

6.6 *Water Bath*, with thermostatic controls, maintained at a temperature of $21 \pm 2^\circ\text{C}$.

6.7 *Sample Containers*—Moisture-tight mason jars have been found to be satisfactory where the sample size is not too great (up to 200 g for example). For larger samples, bags of various plastic materials are suitable if the wall thickness is sufficient to provide a good moisture vapor barrier. For example, for polyethylene, a wall thickness of at least 4 mils (approximately 0.1 mm) has been found to be adequate.

7. Reagent

7.1 *Toluene, Water-Saturated*, prepared from toluene having a boiling range such that all distills within a range of 2°C including 110.6°C . Prepare a sufficient quantity of water-saturated solution of toluene for the testing program immediately at hand as follows: To each 1000 cm³ (mL) of toluene, add 50 to 100 cm³ (mL) of distilled water. Shake for about 5 min and allow to settle. Decant the toluene to a flask and attach a reflux condenser with a graduated water trap. Reflux for 1 h or until water no longer accumulates in the trap. Assume the

toluene in the flask to be water-saturated and store in glass-stoppered bottles until used.

7.2 *Potassium Dichromate Cleaning Solution*—Prepare this solution by mixing 35 cm³ (mL) of a saturated (at room temperature) potassium dichromate solution with 1000 cm³ (mL) of concentrated sulfuric acid.

8. Hazards

8.1 Toluene is flammable and slightly toxic. It should be used in a well-ventilated area, for example, under a hood, to prevent accumulation of vapors.

9. Sampling

9.1 *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 4—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.

9.2 Use extreme care to prevent gain or loss of moisture during the sampling operation and in 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 .

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

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

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

10. Number of Specimens

10.1 Take a number of specimens per laboratory sampling unit such 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:

10.1.1 *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:

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

⁶ Corning No. 5100 or equivalent is suitable.

⁷ Corning No. 3600 or equivalent is suitable.

TABLE 1 Values of Student's *t* for One-Sided and Two-Sided Limits and the 95 % Probability^A

df	One-sided	Two-Sided	df	One-Sided	Two-Sided	df	One-Sided	Two-Sided
1	6.314	12.706	11	1.796	2.201	22	1.717	2.074
2	2.920	4.303	12	1.782	2.179	24	1.711	2.064
3	2.353	3.182	13	1.771	2.160	26	1.706	2.056
4	2.132	2.776	14	1.761	2.145	28	1.701	2.048
5	2.015	2.571	15	1.753	2.131	30	1.697	2.042
6	1.943	2.447	16	1.746	2.120	40	1.684	2.021
7	1.895	2.365	17	1.740	2.110	50	1.676	2.009
8	1.860	2.306	18	1.734	2.101	60	1.671	2.000
9	1.833	2.262	19	1.729	2.093	120	1.658	1.980
10	1.812	2.228	20	1.725	2.086	∞	1.645	1.960

^A Values in this table were calculated using Hewlett Packard HP 67/97 Users' Library Programs 03848D, "One-Sided and Two-Sided Critical Values of Student's *t*" and 00350D, "Improved Normal and Inverse Distribution." For values at other than the 95 % probability level, see published tables of critical values of Student's *t* in any standard statistical text.

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* (Table 1), and

E = 0.5 percentage points, the allowable variation.

10.1.2 *No Reliable Estimate of s*—When there is no reliable estimate of *s* for the user's laboratory, Eq 1 should not be used 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.

11. Conditioning

11.1 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 is achieved.

NOTE 5—Preconditioning and conditioning as directed in Practice D 1776 is acceptable. Such conditions, however, are not necessary since the object of the conditioning for the purpose of this test is merely to stabilize the sample in order that no changes in moisture that cannot be detected will occur while the specimens are being prepared and weighed.

11.2 Weigh the conditioned sample(s) to the nearest 0.05 g and record the net mass, *S*. This mass, and the net mass at time of sampling, *M*, will be used to convert the observed moisture content to the moisture content at the time of sampling.

12. Procedure

12.1 Prior to using the equipment for this test, clean the receiver and condenser with the potassium dichromate cleaning solution, rinse thoroughly with tap water, then with methyl alcohol, and dry.

NOTE 6—This cleaning operation is not needed between successive determinations in the same equipment.

12.2 Select a specimen, determine its mass to the nearest 0.05 g, and designate this mass as *W*.

12.3 Transfer the specimen immediately after weighing, or after removal from the sealed container, to a distilling flask and at once add 700 cm³ (mL) of freshly prepared water-saturated toluene (7.1). Connect the flasks, receivers, and condensers and place the flasks on electric heaters. Start the tap water flowing through the condensers. Add additional water-saturated toluene through the top of the condensers until the receiver traps are full of toluene and begin to run over into the flasks.

12.4 Heat and bring the toluene to the boiling point and adjust the rate of distillation to 2 drops per second.

12.5 When the water is accumulating at a rate of less than 0.1 cm³ (mL) per 15 min, increase the distilling rate to about 4 drops per second. Wash down the condensers by pouring water-saturated toluene in at the top of the condensers. Brush the condensers down with a nylon brush wet out with water-saturated toluene, or dislodge any visible drops of water with a copper wire.

12.6 Continue the distillation until no discernible change is observed in the location of the meniscus over a 15-min period.

12.7 Separate the receivers containing water and toluene from the flasks and condensers. Place the receivers with their contents in the water bath which is maintained at 21 ± 2°C. After approximately 30 min, read the volume of water in the traps, *V*, to the nearest 0.05 cm³ (mL).

13. Calculation

13.1 If the specimen mass *W* is the same as the stabilized sample mass *S*, calculate the amount of moisture as a percentage of the as-sampled mass (moisture content) or of the dry fiber mass (moisture regain) by Eq 2 or Eq 3, to the nearest 0.01 percentage point.

Moisture content, percentage points

$$= [1 - (W - 0.997V)/M] \times 100 \quad (2)$$

Moisture regain, percentage points

$$= [M/(W - 0.997V) - 1] \times 100 \quad (3)$$

where:

V = volume of the water collected in the trap, mL (mL),

M = original mass of the sample (at time of sampling), g, and

W = mass of the specimen tested, g, and
 0.997 = factor for converting the observed volume of water, V , in mL, to the corresponding mass of water in g.

13.2 If the specimen mass W represents only a portion of the stabilized sample mass S , calculate the amount of moisture as a percentage of the as-sampled mass (moisture content) or of the dry fiber mass (moisture regain) by either Eq 4 or Eq 5:

Moisture content, percentage points

$$= \frac{[(S/M)(0.997V/W - 1) + 1]}{\times 100} \quad (4)$$

Moisture regain, percentage points

$$= \frac{[M/S(1 - 0.997V/W) - 1] \times 100}{(5)}$$

where:

S = stabilized mass of the sample from which the specimen was taken, g, and other terms are as described in 13.1.

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

13.4 The following equations may be used to convert between moisture regain and moisture content, and vice versa.

$$R = [C/(100 - C)] \times 100 \quad (6)$$

$$C = [R/(100 + R)] \times 100 \quad (7)$$

where:

R = moisture regain, percentage points, and
 C = moisture content, percentage points.

14. Report

14.1 State that the specimens were tested as directed in Test Method D 2462. Describe the material or product sampled, and state the method of sampling used.

14.2 Report the following information:

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

14.2.2 The number of specimens tested.

14.2.3 The range of the moisture results (difference between the largest and smallest observed results).

15. Precision and Bias

15.1 *Interlaboratory Test Data*⁸—An interlaboratory test was carried out in 1963 in which each of six laboratories tested at least 12 specimens of each of three different wool materials at two different levels of moisture content. The components of variance for moisture content expressed as standard deviations were calculated to be:

Within-laboratory component	0.429 percentage point
Between-laboratory component	0.126 percentage point

NOTE 7—The within-laboratory component includes the single-operator component which was not determined separately.

15.1.1 The components listed above do not include any lot or laboratory sampling error. This error must be considered in any application of the method.

15.2 *Precision*—For the components of variance reported in 15.1, 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 neglecting the errors contributed by lot sampling and laboratory sampling.

Number of Observations in Each Average	Critical Differences, Percentage Points, for the Conditions Noted ^{A,B}	
	Within-Laboratory Precision	Between-Laboratory Precision
2	0.84	0.91
3	0.69	0.77
5	0.53	0.64
10	0.38	0.51

^A The critical values listed in this table were calculated using $t = 1.960$ which is based on infinite degrees of freedom.

^B The values of the critical difference constitute 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.

15.3 *Bias*—The procedure in Test Method D 2462 for determination of the amount of moisture present in wool by toluene extraction has no bias because the value of that property can be defined only in terms of a test method.

16. Keywords

16.1 wool; moisture content

⁸ ASTM Research Report File, RR D-13-1016. A copy of the report is available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

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