Designation: D2462 – 90 (Reapproved2008)

Standard Test Method for Moisture in Wool by Distillation With Toluene[1]

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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 D1576 and for textile materials in general in Test Methods D2654. A method for sampling wool for the determination of moisture in wool is covered in Practice D2525.

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:*[2]

D123 Terminology Relating to Textiles
D1060 Practice for Core Sampling of Raw Wool in Packages for Determination of Percentage of Clean Wool Fiber Present
D1576 Test Method for Moisture in Wool by Oven-Drying
D1776 Practice for Conditioning and Testing Textiles
D2258 Practice for Sampling Yarn for Testing
D2525 Practice for Sampling Wool for Moisture
D2654 Test Method for Moisture in Textiles (Withdrawn 1998)[3]
D3333 Practice for Sampling Manufactured Staple Fibers, Sliver, or Tow for Testing
E123 Specification for Apparatus for Determination of Water by Distillation

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).

[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 Felt.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

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 moisutre 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 D123.

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 D2462 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 D2462 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.[4]

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

[4] Toner, R. K., Bowen, C. F., and Whitwell, J. C., "Equilibrium Moisture Relations for Textile Fibers," *Textile Research Journal*, 17, January 1947, 17—18.

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.

5.6 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 cm^3 (mL) capacity (takes a No. 11 stopper).[5]

6.2 *Distilling Receiver, Dean & Stark,* 10 cm^3 (mL) capacity, graduated in 0.1 cm^3 (mL).[6]

NOTE 2—The tolerance on the accuracy of the graduations specified in Specification E123 for this apparatus is ± 0.1 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 E123.

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 ± 2°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.

[5] Corning No. 5100 or equivalent is suitable.
[6] Corning No. 3600 or equivalent is suitable.

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 cm^3 (mL) of toluene, add 50 to 100 cm^3 (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 cm^3 (mL) of a saturated (at room temperature) potassium dichromate solution with 1000 cm^3 (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 D2525 for bales of fiber and containers of top or sliver or to use Practice D2258 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 D1060 for cored samples or Practice D3333 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