Designation: D584 – 10

Standard Test Method for Wool Content of Raw Wool—Laboratory Scale[1]

This standard is issued under the fixed designation D584; 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 a laboratory procedure for the determination of the wool base content and the clean wool fiber present in samples of raw wool. This test method is also applicable to other animal fibers such as mohair, cashmere, alpaca, and camel hair.

NOTE 1—Sampling of lots of raw wool in packages is covered in Practice D1060; the determination of vegetable matter and other alkali-insoluble impurities in scoured wool is covered in Test Method D1113; the determination of wool content on a commercial scale is covered in Test Method D1334. For factors for the conversion of woolbase content to its equivalent in terms of scoured wool, top, or noil of various commercially specified compositions (formerly covered in the appendix of this test method), refer to Practice D2720.

NOTE 2—Because of the trade practice the term weight is used in this test method instead of the technically correct term mass.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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:*[2]

D123 Terminology Relating to Textiles

D584 Test Method for Wool Content of Raw Wool—Laboratory Scale

D1060 Practice for Core Sampling of Raw Wool in Packages for Determination of Percentage of Clean Wool Fiber Present

D1113 Test Method for Vegetable Matter and Other Alkali-Insoluble Impurities in Scoured Wool

D1334 Test Method for Wool Content of Raw Wool—Commercial Scale

D2525 Practice for Sampling Wool for Moisture

D2720 Practice for Calculation of Commercial Weight and Yield of Scoured Wool, Top, and Noil for Various Commercial Compositions

D4845 Terminology Relating to Wool

E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)

2.2 *Other Standard:*

IWTO-19-85 (E) Method for the Determination of Wool Base, Vegetable Matter Base; IWTO Clean Wool Content; IWTO Scoured Yield in Raw Wool[3]

3. Terminology

3.1 For all terminology relating to D13.13, Wool and Wool Felt, refer to Terminology D4845.

3.1.1 The following terms are relevant to this standard: *clean wool fiber present, other alkali-insoluble impurities, oven-dried, raw wool, vegetable matter base, vegetable matter present, wool base, yield.*

3.2 For definitions of other textile terms used in this test method, refer to Terminology D123.

4. Summary of Test Method

4.1 The entire sample, or each test specimen drawn therefrom in a specified manner, is weighed, scoured, dried, and reweighed. The oven-dry scoured wool is tested to determine its content of alcohol-extractable matter, mineral matter (ash), vegetable matter base, and other alkali-insoluble impurities. The wool-base content, laboratory scoured yield, clean wool fiber present, and vegetable matter present are calculated as percentages of the mass of the raw wool sample.

5. Significance and Use

5.1 This test method is considered satisfactory for acceptance testing of commercial shipments since this test method has been used extensively in the trade for acceptance testing.

[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] Specifications of Test Methods, International Wool Textile Organization, International Wool Secretariat, Ilkley, West Yorkshire, U.K. LS298PB.

5.1.1 In case of dispute arising from differences in reported test results when using Test Method D584 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 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 then 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 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 known bias.

5.2 The wool-base content of wool in any condition or form is a basic quantity. From it may be calculated commercial masses or yields in any of the various recognized defined systems used in international commerce (Note 1).

5.2.1 The procedures for determining the wool base content of greasy wool provided in this test method and in IWTO Method 19-85(E) are in essential agreement.

NOTE 3—This is not true for scoured wool, as IWTO Method 19-85(E) does not require rescouring of scoured wool containing less than 5% residual grease.

5.3 Not all of the wool base present in a lot of raw wool can be recovered in useful form by commercial cleaning operations. The amount of wool loss varies, depending on factors such as the character of the wool, the nature and percentage of the impurities present, the cleaning process and equipment used, and so forth.

5.4 No ASTM standard specifies or recommends any specific procedure or practice for estimating anticipated loss of wool during commercial cleaning (or other) operations. The following statutory practice is described solely for information:

5.4.1 For the purpose of duty assessment on importations of raw wool into the United States, the Tariff Schedules of the United States[4] provides a statutory formula for calculating the allowance to be made for wool "that would ordinarily be lost during commercial cleaning operations." The formula is based on the clean wool fiber present (called "absolute clean content" in the Tariff Schedules) and on the vegetable matter present. The allowance, in terms of clean wool fiber present, is equal to 0.5 % of the clean wool fiber present plus 60 % of the vegetable matter present, the total allowance not to exceed 15 % of the clean wool fiber present. The dutiable quantity (called "clean yield" in the Tariff Schedules) is the difference between the clean wool fiber present and the allowance so calculated.

6. Apparatus

6.1 *Subsampling Equipment*—A cylindrical or rectangular chamber having a sliding cover plate by means of which wool in the chamber may be compressed, and openings in the bottom plate through which cores may be bored with a sampling tube approximately 12 mm in inside diameter. The openings shall be about 18 mm in diameter and spaced uniformly on 40 to 50-mm centers over the entire plate. The volume of the chamber must be sufficient to contain the sample, but the relative dimensions are optional. For greasy wool samples weighing 10 kg, a chamber 300 by 300 by 700 mm is satisfactory. A replaceable inner lining of soft wood or similar material for the sliding cover plate is recommended to avoid damage to the cutting edge of the sampling tube.

6.1.1 *Sampling Tube*—Similar to that used to obtain core samples, as described in Practice D1060.

6.2 *Scouring Equipment*—A scouring bowl with accessories, and a flotation jar.

6.2.1 *Scouring Bowl*—A rectangular or cylindrical vessel of 30 to 50-L capacity, with an attached drain board. The lower portion of the bowl is in the shape of an inverted pyramid or cone that is connected to a sliding-disk valve and a short length of drain pipe. At the bottom of the bowl, above the valve and drain pipe, is a close-fitting, removable perforated plate (6.2.1.1). The drain pipe is centered over a No. 200 (75-µm) sieve, 120 to 200 mm in diameter, supported in a catch-basin.

6.2.1.1 *Two Plates,* one with 1 to 2-mm openings, the other similar plate covered on its upper surface with No. 100 (150-µm) woven wire cloth.

6.2.1.2 *Thermostatic Device,* capable of delivering water to the scouring bowl at a desired temperature with a tolerance of ±3°C.

6.2.1.3 *Paddle* or other *Stirring Device.*

6.2.1.4 *Spray or Shower Head* with a flexible connection for use in rinsing.

6.2.2 *Flotation Jar*—A glass or transparent plastic vessel of 1 to 2-L capacity, approximately 200 mm tall, for separating by flotation the short wool fibers retained by the No. 200 (75-µm) sieve from associated sand and other heavy impurities.

6.3 *Wringer or Basket Centrifuge,* for the removal of excess water from the scoured sample before drying in the oven.

6.3.1 *Net Bag,* having openings of 60 mesh (250 µm) or finer. Bags are used with a squeeze roll type of wringer or with a centrifuge.

6.3.2 *Metal Can,* with bottom formed from 100-mesh (150-µm) wire screen supported by a perforated metal plate may be used with basket centrifuges. The dimensions of the can must be such that the can is capable of containing the scoured sample, fitting into the centrifuge, and adaptable to the dryer.

6.4 *Dryer*—A forced-draft oven or, preferably, a heated air flow-through type of dryer capable of supplying clean air at a desired temperature with a tolerance of ±2°C.

6.5 *Muffle Furnace,* thermostatically controlled in the range of 700 ± 25°C.

6.6 *Soxhlet Extraction Apparatus,* medium size.

7. Reagents

7.1 *Scouring Solution A*—A solution containing approximately 0.3 % of soda ash (Na_2CO_3) and 0.1 % of soap having

[4] Tariff Schedules of the United States, Schedule 3. Part 1, Subpart C, Headnote 1 (c).

a titer of not over 25°C. Addition to the solution of approximately 0.3 % of a lime-sequestering agent of the polyphosphate type is recommended.

7.2 *Scouring Solution B*—A solution containing approximately 0.15 % of Na_2CO_3 and 0.05 % of soap having a titer of not over 25°C. Addition to the solution of approximately 0.3 % of a lime-sequestering agent of the polyphosphate type is recommended.

NOTE 4—For nonreferee tests, various scouring solutions containing nonionic detergents, with or without soda ash or builders, at various temperatures, are sometimes used instead of Scouring Solutions A and B.

7.3 *Washing Solution*—A solution containing approximately 0.02 % of a nonionic detergent of the polyoxyethylene type.

7.4 *Alcohol*—Either pure ethyl alcohol (C_2H_5OH) or specially denatured alcohol conforming to Formula 3A or 30 of the U.S. Bureau of Internal Revenue.

8. Preparation of Sample and Test Specimens

8.1 For the sampling of commercial shipments take a lot sample as directed in an applicable material specification, or as agreed upon between the purchaser and the seller. In absence of a material specification or other agreement, take a lot sample from a lot of packaged raw wool as directed in Practice D1060, and take a lot sample from a lot of raw wool in bulk form as directed in Practice D2525.

8.2 *Weighing*—Determine the net mass, in grams, of the laboratory sample as received to four significant figures, taking care to avoid any change in moisture content during weighing.

8.2.1 Pieces of outer bale wrappers (burlap or plastic) are occasionally present in core samples. If such material is present, remove and weigh it before discarding. Deduct the mass of this material from the net mass of the sample as received (8.2) to obtain the adjusted net mass, *M*.

8.2.2 Remove and discard, without weighing, strings and other extraneous material not containing wool or vegetable matter that are present in substantial amount.

8.3 *Small Samples*—If the mass of the sample is not more than three times the scouring capacity of the scouring bowl, test the entire sample, in one, two, or three portions as may be required. Consider the maximum scouring capacity of the bowl to be the mass of raw wool in grams equal to 12 times the volume of scouring solution in litres.

8.3.1 If the sample is a core sample, no further preparation is required. If it is not a core sample and consists of fibers exceeding 50 mm in length, cut the fibers with scissors or a paper cutter to less than 50-mm lengths.

8.4 *Large Samples*—If the mass of the sample exceeds three times the scouring capacity of the bowl (8.3), prepare test specimens by subsampling as follows:

8.4.1 Place the sample in the chamber of the subsampling device (6.1), compress to a density of 0.2 to 0.3 g/cm^3, and take a full-length core through each opening of the plate.

8.4.2 Open the chamber, redistribute the wool, compress and take a second test specimen as in 8.4.1. Repeat 8.4.2 until the desired mass of core samples are obtained.

8.4.3 Remove, weigh, and retain the remainder of the sample in an airtight container for use in drawing additional test specimens, if necessary or desired.

8.4.4 Weigh the blended core samples into individual test portions in preparation for scouring (10.1).

9. Conditioning

9.1 Neither preconditioning nor conditioning in the standard atmosphere for testing is required.

10. Procedure

10.1 *Scouring*—Scour each portion of the sample (8.3) or each test specimen (8.4.1, 8.4.2) as directed in 10.1.1 or 10.1.2, whichever is applicable.

10.1.1 *Greasy or Pulled Wool:*

10.1.1.1 With the coarse perforated plate in place in the scouring bowl, immerse the wool in Scouring Solution A at a temperature of 52 ± 3°C (not less than 1 L for each 12 g of wool) and stir for 3 min. Drain the solution through the No. 200 (75-µm) sieve. Spray the wool with a strong stream of warm water (35 to 45°C) so as to flush out as much as possible of sand and other soil, then remove the wool from the bowl and place it on the drain board. Raise the plate, and remove and discard any impurities other than vegetable matter (Note 5) lodged thereon.

NOTE 5—The total amount of vegetable matter present in the sample is generally one factor used in estimating wool yield (see 5.4.1). If such an estimate is to be made, loss of vegetable matter must be avoided.

10.1.1.2 Spray the material on the No. 200 (75-µm) sieve with warm water, then transfer to the flotation jar. Fill the jar with warm water, using the spray to cause agitation and aeration. After the sediment has settled, decant the floating wool and vegetable matter into the bowl. Refill the jar with the spray, allow to settle, and again decant. If the sediment still contains wool or vegetable matter estimated to exceed 0.05 % of the specimen mass, repeat once more before discarding the sediment.

10.1.1.3 With the coarse perforated plate in place in the scouring bowl, immerse the wool in Scouring Solution B at a temperature of 52 ± 3°C, and repeat the stirring, draining, spraying, and flotation operations described in 10.1.1.1 and 10.1.1.2.

10.1.1.4 With the No. 100 (150-µm) screen in place in the scouring bowl, repeat the preceding operations twice more but with warm water (35 to 45°C) instead of scouring solution. During the rinsing, remove by hand and discard as much as possible of the remaining strings, skin pieces, and other extraneous material free from wool fiber or vegetable matter (8.2.1).

10.1.1.5 Thoroughly mix the wool fibers and vegetable matter recovered by flotation after the last rinse with the main body of scoured wool, place in the net bag or other container (6.3.1, 6.3.2), and centrifuge or pass through the wringer to remove excess water.

10.1.2 *Scoured Wool*—Proceed as directed in 10.1.1.3-10.1.1.5.

10.2 *Drying*—Determine the oven-dry mass of the scoured wool as directed in 10.2.1 or 10.2.2.

10.2.1 Using air drawn from an atmosphere having a relative humidity of 65 ± 2 % at a temperature of 20 ± 2°C, dry each scoured wool portion or test specimen in the forced draft oven (6.4) at 105 ± 2°C to constant mass. Weigh to four significant figures. Consider the mass as constant when two successive weighings 10 min apart differ by 0.1 % or less.

10.2.2 If the air used is at other than the atmospheric conditions described in 10.2.1, determine the temperature and relative humidity of the air as directed in Test Method E337. Correct the constant oven-dry mass by applying to it the appropriate percentage correction obtained from Table 1.

10.3 *Dusting and Washing*—Examine the dried scoured wool. If lime or dung impurities are present, estimated to exceed 0.5 % of the specimen mass, carefully manipulate the wool by hand or in a suitable mechanical device to break up and dust out these impurities without loss of fiber, then wash once with the washing solution at a temperature between 35 and 45°C, rinse with warm water, and dry to constant mass (10.2.1).

10.4 *Alcohol Extraction*—Determine the content of alcohol-extractable matter in each oven-dry scoured portion or test specimen as follows:

10.4.1 Place a representative specimen weighing about 10 g of the dried scoured wool in a tared moisture dish, redry for 30 min in an oven at 105 to 110°C, cool in a desiccator, and weigh to the nearest 0.01 g. Place a thin layer of cotton gauze, previously extracted with alcohol, in the bottom of a Soxhlet tube as a filter covering the opening of the siphon tube. Transfer the specimen to the Soxhlet tube and extract for 20 siphoning cycles (2 to 3 h) with neutral 95 % ethyl alcohol. Evaporate the alcohol extract in a tared beaker on a steam bath with forced draft ventilation, such as a hood (**Warning—** Flammable vapors may be evolved), dry the residue for 10 min in an oven at 100 to 105°C, cool in a desiccator, weigh to the nearest 1 mg, and calculate the mass of extract as a percentage of the specimen mass.

10.5 *Ash*—Determine the ash content of each oven-dry scoured portion or test specimen as follows: Place two representative 5-g specimens of the dried scoured wool in tared, tall-form porcelain or silica crucibles of approximately 80-mL capacity, redry for 30 min in an oven at 105 to 110°C, cool in a desiccator, and weigh to the nearest 0.01 g. Char the wool, until no more volatile matter is evolved (Note 6). Ignite the charred specimens in a muffle furnace at 700 ± 25°C until the carbon has been burned off, cool in a desiccator, weigh to the nearest 1 mg, and calculate the mass of ash as a percentage of the specimen mass.

NOTE 6—Wool tends to swell during charring, with possibility of loss. A satisfactory procedure is to wet out the weighed dry specimen with 5 mL of water, then burn the wool with flames from above and below until charring is complete.

10.6 *Vegetable Matter and Total Alkali-Insoluble Impurities*—Determine the percentages of vegetable matter base and of total oven-dry, ash-free, alcohol-extractives-free, alkali-insoluble impurities in each oven-dry scoured portion or test specimen (10.2.1) as directed in Test Method D1113.

11. Calculation

11.1 *Small Samples*—When the entire sample has been scoured (8.3.1), calculate the percentage wool base present using Eq 1, the percentage clean wool fiber present using Eq 2, the percentage vegetable matter present using Eq 3, and the percentage laboratory scoured yield using Eq 4.

$$B = \left[\sum P_i\,(100 - A_i - E_i - T_i)\right]/M \quad (1)$$

$$F = B/0.86 \quad (2)$$

$$VM = \left(\sum P_i V_i\right)/(0.86\,M) \quad (3)$$

$$Y = F + VM \quad (4)$$

where:

B = wool base present, as a percentage of the sample mass (8.2.1),
F = clean wool fiber present, as a percentage of the sample mass (8.2.1),
VM = vegetable matter present, as a percentage of the sample mass (8.2.1),
Y = laboratory scoured yield, as a percentage of the sample mass (8.2.1),
P_i = mass, g, of the oven-dry scoured wool from the ith portion or test specimen (10.2.1),
A_i = average percentage of ash in the oven-dry scoured wool from the ith portion or test specimen (10.5),

TABLE 1 Correction Table for Moisture Content of Drying Air[A,B]

Drying Air Temperature, °C	Drying Air Relative Humidity, %							
	20	30	40	50	60	70	80	90
5	0.44	0.41	0.39	0.36	0.33	0.30	0.28	0.24
10	0.42	0.38	0.34	0.30	0.26	0.22	0.18	0.14
15	0.39	0.33	0.28	0.22	0.16	0.11	0.05	–0.01
20	0.35	0.27	0.19	0.12	0.04	–0.04	–0.12	–0.20
25	0.29	0.19	0.09	–0.02	–0.13	–0.24	–0.34	–0.45
30	0.22	0.07	–0.06	–0.22	–0.35	–0.50	–0.64	–0.79
35	0.12	–0.06	–0.25	–0.46	–0.64	–0.80	–0.98	–1.20

[A] Values in this table are percentage corrections to be added to the observed oven-dry mass. They were calculated using the equation:

$$\text{Percentage correction} = 0.053\left[9.470 - (622\,e \times r)/(760 - e \times r)\right]$$

where:

r = relative humidity percent/100, and
e = saturation pressure of water vapor, mm Hg. Values of e are given in hygrometric tables.

[B] The table and equation are those shown in IWTO Method 19 – 74 (E).

E_i = percentage of alcohol-extractable matter in the oven-dry scoured wool from the ith portion or test specimen (10.4.1),

T_i = percentage of total oven-dry, ash-free, alcohol-extractives-free, alkali-insoluble impurities in the oven-dry scoured wool from the ith portion or test specimen (10.6),

V_i = percentage of vegetable matter base in the oven-dry scoured wool from the ith portion or test specimen (10.6),

M = mass, g, of the sample (8.2.1), and

0.86 = factor to convert mass of wool base present to mass of clean wool fiber present and mass of vegetable matter base to mass of vegetable matter present.

NOTE 7—For the factor to convert wool base (B) to IWTO Clean Wool Content (2.2) refer to Practice D2720, Table 1.

11.2 *Large Samples*—When only test specimens (8.4) have been tested, calculate the percentage wool base present using Eq 5, the percentage clean wool fiber present using Eq 2, the percentage vegetable matter present using Eq 6, and the percentage laboratory scoured yield using Eq 4.

$$B = \left[M_B \sum P_i (100 - A_i - E_i - T_i)\right] / M \sum M_i \quad (5)$$

$$VM = \left(M_B \sum P_i V_i\right) / \left(0.86\, M \sum M_i\right) \quad (6)$$

where:

M_B = combined mass, g, of all test specimens and remainder (8.4), and

M_i = mass, g, of the ith test specimen, and other symbols have the meanings assigned in 11.1.

12. Report

12.1 State that the specimens were tested as directed in Test Method D584. Describe the material or product sampled and the method of sampling used.

12.2 Report the following information:

12.2.1 Percentages of wool base present, laboratory scoured yield, vegetable matter present, and clean wool fiber present, each to the nearest 0.1 percentage point, and

12.2.2 Net mass of the sample.

TABLE 2 Critical Differences for Clean Wool Fiber Present in Raw Wool

Number of Observations in Each Average	Critical Differences, Percentage Points[A]	
	Single-Operator Precision	Between-Laboratory Precision
1	1.1	1.4
3	0.6	1.0
5	0.5	1.0

[A] The critical differences were calculated using $t = 1.960$ which is based on infinite degrees of freedom.

13. Precision and Bias

13.1 *Interlaboratory Test Data*[5]—An interlaboratory test was run in 1970 in which four laboratories each tested three random specimens from each of four samples from different greasy, low-burr wools. The average components of variance (for clean wool fiber present) expressed as standard deviations were calculated to be as follows:

Single-operator component	0.39 percentage point
Between-laboratory component	0.30 percentage point

13.2 *Precision*—For the components of variance stated in 13.1, two averages of observed percentages of clean wool fiber present should be considered significantly different at the 95 % probability level if the difference equals or exceeds the critical differences listed in Table 2.

NOTE 8—The tabulated values of the critical differences should be considered to be a general statement particularly with respect to between-laboratory precision. Before a meaningful statement can be made about two specific laboratories the statistical bias, if any, between them must be established with each comparison being based on recent data obtained on specimens taken at random from one sample of the material to be tested.

13.3 *Bias*—No justifiable statement on the accuracy of Test Method D584 for the laboratory scale measurement of wool content of raw material can be made since the true percentage cannot be established by an independent method.

14. Keywords

14.1 clean wool content; raw wool; yield

[5] A copy is available from ASTM Headquarters. Request RR:D-13-1010.