Designation: D1113 – 13

Standard Test Method for Vegetable Matter and Other Alkali-Insoluble Impurities in Scoured Wool[1]

This standard is issued under the fixed designation D1113; 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 content of oven-dried, ash-free, alcohol extractive-free vegetable matter and other alkali-insoluble impurities present in scoured wool. It is also applicable to "related fibers" such as the hair from the goat, camel, alpaca, and other animals.

NOTE 1—The determination of clean wool fiber present on a laboratory scale is covered in Test Method D584, the determination of clean wool fiber present on a commercial scale is covered in Test Method D1334, and the calculation of commercial weight and yield of various commercial compositions (formerly covered in Appendix to Test Method D584) is covered in Practice D2720.

1.2 *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
D584 Test Method for Wool Content of Raw Wool—Laboratory Scale
D1334 Test Method for Wool Content of Raw Wool—Commercial Scale
D2720 Practice for Calculation of Commercial Weight and Yield of Scoured Wool, Top, and Noil for Various Commercial Compositions
D4845 Terminology Relating to Wool

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: other alkali-insoluble impurities, oven-dried, vegetable matter base.

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

4. Summary of Test Method

4.1 The wool, or other animal fiber, is dissolved in a boiling 3% sodium hydroxide solution or a hot 10% sodium hydroxide solution under specified controlled conditions. The weights of the ash-free, oven-dried components of the undissolved residue are converted by means of tabulated factors to the corresponding weights of vegetable matter base and other alkali-insoluble impurities.

5. Significance and Use

5.1 Test Method D1113 is considered satisfactory for acceptance testing of commercial shipments, and the procedure has been used extensively in the trade for this purpose, particularly in connection with the determination of clean wool fiber present by Test Method D584. The procedure in Test Method D1113 is used by the U.S. Customs Service for the determination of the vegetable matter in importations of raw wool on which the allowance for loss of wool during commercial cleaning is based in part.[3]

5.1.1 In case of a dispute arising from differences in reported test results when using Test Method D1113 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 that are as homogeneous as possible and that 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 Students *t*-test for unpaired data and an acceptable probability level chosen by the two parties before the testing is begun. If a bias is found, either

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

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.

6. Apparatus

6.1 *Filter,* 40-mesh sieve (U. S. Sieve Series, opening 0.0165 in. (0.42 mm)) or metal screen, or cheese cloth having comparable openings.

6.2 *Oven*—A forced-draft oven designed to supply clean air at a desired temperature with a tolerance of ±2°C.

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

6.4 *Beakers*—Heat resistant glass or stainless steel, of 2-litre capacity.

7. Reagents and Materials

7.1 *Sodium Hydroxide Solution* (NaOH), 3 % by weight.

7.2 *Sodium Hydroxide Solution,* 10 % by weight.

7.3 *Sodium Hypochlorite Solution* (NaOCl), 5 % by weight.

8. Hazards

8.1 Sodium hydroxide is extremely corrosive, and care must be exercised to avoid contact with the eyes, skin, or clothing.

8.2 Operators should wear eye protection while handling caustic solutions.

9. Sampling

9.1 *Lot Sample*—As a lot sample for acceptance testing, take at random the number of shipping containers directed in an applicable material specification or other agreement between the purchaser and the supplier. Consider shipping containers to be the lot sampling unit.

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

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

9.2.1 For tests to be made in connection with Test Methods D584 and D1334, prepare a portion of scoured and dried wool from each lot sampling unit of the lot sample described in 9.1. Make sure that each portion is approximately 100 g, which will become a laboratory sampling unit. Note that these portions are to be obtained from random locations in the wool mass. If the scoured wool is judged to contain over 5 % of vegetable matter and sufficient scoured wool is available, prepare another replicate, that is, a second laboratory sample unit.

9.2.2 For tests on samples of scoured wool not obtained in connection with Test Methods D584 and D1334 take replicate sample units as directed in 9.2.1.

9.3 *Test Specimens*—As test specimens, prepare two test specimens from each laboratory sampling unit by combining ten or more pinches of fiber into a bundle having a mass of 40 ± 1 g.

10. Conditioning

10.1 Dry specimens taken as directed in 9.1 under the conditions for oven drying prescribed in Test Method D584.

10.2 Weigh specimens taken as directed in 9.2 in the condition as received, in the oven-dry condition, or after exposure in the standard atmosphere for testing textiles, depending upon the directions or requirements of the interested parties.

10.3 Weigh specimens (10.1 or 10.2) to the nearest 0.01 g. (W_2).

11. Procedure

11.1 *Preferred Method*—In a 2-L heat-resistant glass or stainless steel beaker, bring 1 L of 3 % NaOH solution to a boil. With the solution at a boil, carefully add the entire weighed specimen. Quickly immerse the wool in the NaOH solution with the aid of a stirring rod, and adjust the heat to resume boiling of the solution. Boil the solution gently with continuous stirring for 90 ± 2 s. Remove the beaker from the heat and add 500 ml of tap water, stir, then allow to settle.

NOTE 3—The preferred method uses 3 % sodium hydroxide solution, in which most wools are soluble when treated as directed. However, certain coarse, dry carpet wools do not dissolve completely in the 3 % solution. For such wools the alternative method (11.1.1), in which 10 % sodium hydroxide solution is used, is necessary.

NOTE 4—Keep depilatory in pulled wool specimens to a minimum by treating the sample as directed in 10.3 of Test Method D584.

11.1.1 *Alternative Method* (*Note 2*)—In a 2-L heat-resistant glass or stainless steel beaker, bring 600 cm^2 (mL) of 10 % NaOH solution to a boil. Remove the beaker from the heat, place on a dry wooden or asbestos mat, and immediately add the weighed specimen. Stir continuously for 3 min ± 5 s, add 1000 mL of tap water, stir, and allow to settle.

11.2 *Filtration*—Decant the solution through the 40-mesh filter, using a stirring rod or a jet of water to assist filtration by agitation of the solution on the filter. Wash all the remaining vegetable matter and other alkali-insoluble impurities in the beaker onto the filter. Discard any sand or depilatory (Note 3) in the beaker and on the filter. Sprinkle about 15 mL of 5 % sodium hypochlorite solution over the residue on the filter, then rinse with a moderate spray of water at 35 to 40°C for 3 to 4 min or until the residue is neutral as indicated by litmus paper.

11.3 *Components of Alkali-Insoluble Impurities*—Observe the rinsed material on the filter. Estimate the fraction of the total dry weight corresponding to each type of burr and other vegetable matter, as well as non-wool fibers, skin, paint, and tag material present (Note 5).

11.3.1 *More Accurate Determination of Components*—Separate and treat each of the component types from the residue as directed in 11.4 and use the appropriate calculations.

NOTE 5—For clean wool fiber determination (Test Method D584), take care to exclude or correct for components emanating from the original or sample package wrapper or container.

11.4 *Drying and Ashing*—Transfer the rinsed material from the filter into a tared porcelain crucible or dish. Dry at 105± 2°C to constant weight (W_1). Ignite in a muffle furnace at 700 ± 25°C to a white ash, cool in a desiccator, and weigh (A).

12. Calculation of Results

12.1 *Vegetable Matter Base*—Using Eq 1 and the appropriate values in Table 1, calculate the correction factor for converting the weight of recovered oven-dried, ash-free, alkali-insoluble material to the weight of vegetable matter base in the specimen. Calculate the percentage of vegetable matter base using Eq 2.

$$F_v = f_1 v_1 + f_2 v_2 + \ldots \tag{1}$$

$$V = 100 F_v (W_1 - A)/W_2 \tag{2}$$

where:

F_v = correction factor for vegetable matter only,

V = vegetable matter base expressed as a percent of the weight of the original specimen,

$f_1\ f_2$ = weight (mass) fractions of the individual types of vegetable matter in the recovered alkali-insoluble impurities,

$v_1 v_2$ = factors for the individual types of vegetable matter (from Table 1),

W_1 = weight (mass) of oven-dried recovered alkali-insoluble impurities,

W_2 = weight (mass) of specimen used for the test, and

A = weight (mass) of ash from recovered alkali-insoluble impurities.

12.2 *Total Alkali-Insoluble Impurities*— Using Eq 3 and the appropriate values in Table 1, calculate the correction factor for converting the weight (mass) of recovered oven-dried, ash-free, alkali-insoluble material to the original weight of total oven-dried, ash-free, extractives-free alkali-insoluble impurities in the specimen. Calculate the percentage of such total impurities according to Eq 4.

$$F_T = F_v + f_c c + f_p p + f_s s + f_t t + f_x x \tag{3}$$

$$T = 100 F_T (W_1 - A)/W_2 \tag{4}$$

TABLE 1 Correction Factors for Alkali-Insoluble Impurities

NOTE 1—Average value of the ratio of scoured, oven-dry ash-free, extractive-free weight of vegetable matter and other alkali-insoluble matter to the corresponding weight of oven-dry, ash-free material recovered by this method.

Type of Alkali-Insoluble Impurities	Correction Factor for Preferred Method (3 % NaOH Solution)	Correction Factor for Alternative Method (10 % NaOH Solution)
Cockle burr[A]	1.1	1.1
Sand burr[A]	1.1	1.2
Spiral burr[A]	1.2	1.3
Bean burr[A]	1.0	1.1
Shives[A] and similar vegetable matter	1.3	1.6
Cotton and other fibers	1.1	1.1
Skin	2.0	2.0
String (paper)	1.1	1.1
Hard tag pieces	1.4	1.4
Paint pieces[B]	...	...

[A] See Fig. 1.

[B] The correction factor for paint pieces was previously listed as 2.0. This factor may still be used in instances where the quantity of paint is not greater than 0.5 percent. However, a wide variation can exist in this correction factor due to the many types of paint currently being used. It is suggested that samples free of paint be used where possible.

where:

F_v, W_1, W_2 and A = have the meanings given in 12.1, and

F_T = average correction factor for total alkali-insoluble impurities,

T = percentage oven-dried, ash-free, extractives-free total alkali-insoluble impurities,

f_c, f_p, f_s, f_t, f_x = weight fractions of cotton and other fibers, paint, skin, tag material, and paper string, respectively, in the recovered alkali-insoluble impurities, and

c, p, s, t, x = factors for cotton and other fibers, paint, skin, tag material, and paper string, respectively (from Table 1).

13. Report

13.1 State that the tests were made as directed in ASTM Test Method D1113. Describe the material or product sampled and the method of sampling used.

13.2 Report the following information:

13.2.1 Percentages of vegetable matter base and total alkali-insoluble impurities to the nearest 0.1 percentage point.

13.2.2 The basis for the calculated percentages: oven-dried weight, as-received weight, or conditioned weight of the sample.

13.2.3 State whether 3 % or 10 % NaOH was used.

14. Precision and Bias

14.1 *Interlaboratory Test Data*[4]—The first of a planned series of interlaboratory tests was run in 1970, in which four laboratories each scoured and dried three subsamples randomly drawn from the same core sample of a grease wool, using the procedures prescribed in Test Method D584. In each laboratory two 40-g specimens were taken from each oven-dried scoured subsample and tested by one operator for total alkali-insoluble impurities, using 3 % NaOH solution and visually estimating the fractions of the impurity components. This procedure was repeated on core samples from two additional greasy wools. The vegetable matter base content for all three scoured wools was less than 1 percentage point. Under these conditions the components of variance for the percentage of total alkali-insoluble impurities expressed as standard deviations were found to be:

Single-operator component	0.151 percentage point
Between-laboratory component	0.042 percentage point

14.2 *Precision*—For the components of variance listed in 14.1, two averages of observed percentages of alkali-insoluble impurities in scoured wool should be considered significantly different at the 95 % probability level if the difference equals or exceeds the following critical differences (Note 6):

	Critical Difference, Percentage Points, for the Conditions Noted[A]	
Number of Observations in Each Average	Single-Oprator Precision	Between Laboratory Precision
1	0.42	0.43
2	0.30	0.32
3	0.24	0.27

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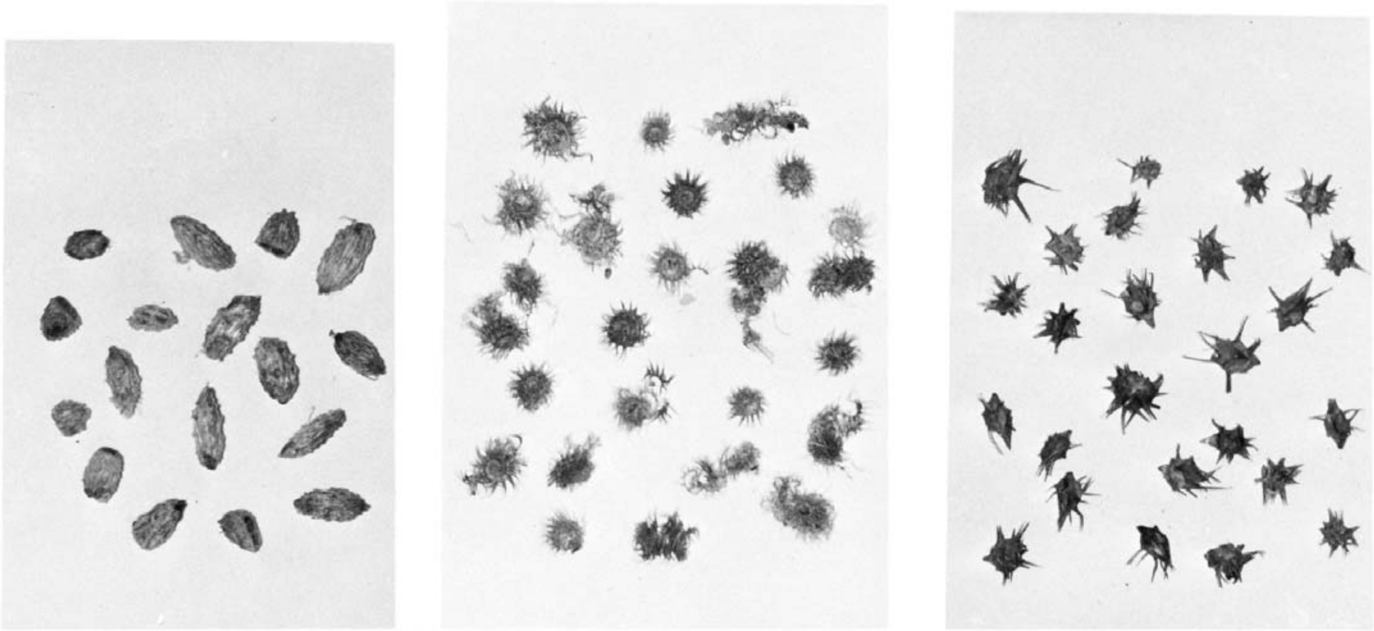
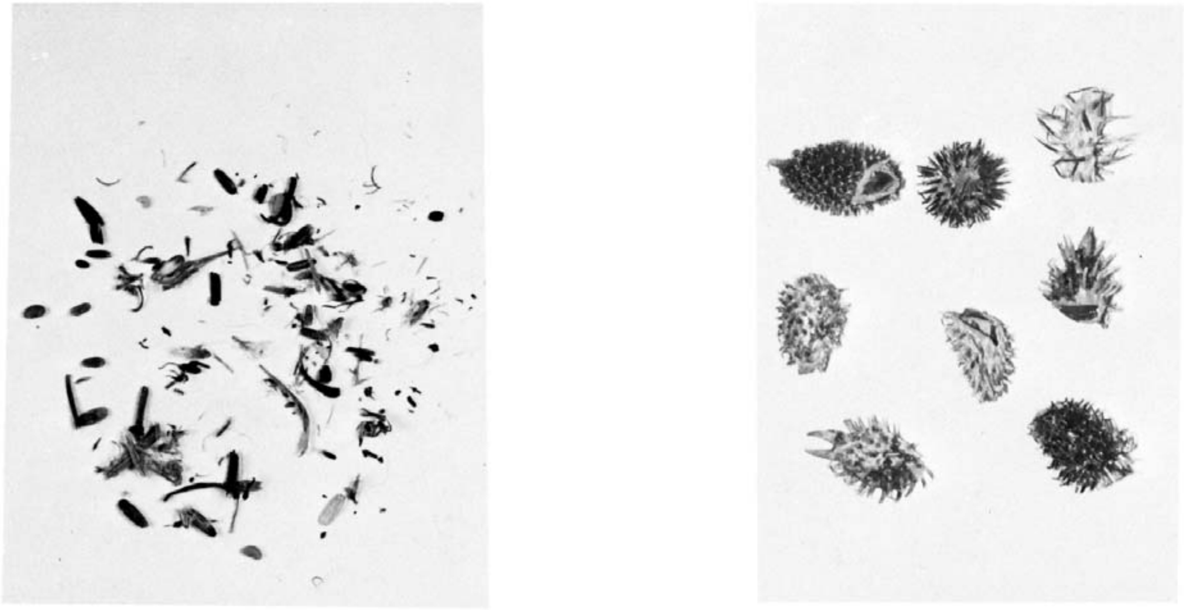
[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D13-1012.



Bean Burrs
(*Genus Xanthium Spinosum*)

Spiral Burrs
(Genus *Medicago*)

Sand Burrs
(Genus *Madhuca (Bassia) or Cenchrus*)



Shives (Composite of small plant fragments and slivers)

Cockle Burrs
(Genus *Xanthium Chinense*)

FIG. 1 Types of Vegetable Matter

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

NOTE 6—The tabulated values of the critical differences constitute a general statement applicable only to scoured wools containing less than 1 % total alkali-insoluble impurities, when the tests are made under the conditions described in 14.1. 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 specimens randomly drawn from a sample of scoured wool containing less than 1 % total alkali-insoluble impurities.

14.3 *Bias*—The procedure in Test Method D1113 for measuring the value of the vegetable matter and other alkali-insoluble impurities in scoured wool has no bias because the value of that property can be defined only in terms of a test method.

15. Keywords

15.1 alkali solubility; impurity; vegetable matter; wool

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