Designation: D 1113 – 90a (Reapproved 1995)

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

This standard is issued under the fixed designation D 1113; 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 D 584, the determination of clean wool fiber present on a commercial scale is covered in Test Method D 1334, and the calculation of commercial weight and yield of various commercial compositions (formerly covered in Appendix to Test Method D 584) is covered in Practice D 2720.

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:*

D 123 Terminology Relating to Textiles[2]

D 584 Test Method for Wool Content of Raw Wool—Laboratory Scale[2]

D 1334 Test Method for Wool Content of Raw Wool—Commercial Scale[2]

D 2720 Practice for Calculation of Commercial Weight and Yield of Scoured Wool, Top, and Noil for Various Commercial Compositions[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *other alkali-insoluble impurities, n*— in scoured wool, the oven-dried, ash-free, alcohol-extractives-free, alkali-insoluble substances other than vegetable matter base, such as skin, cotton or other fibers, paper string, tag (dung) pieces, and paint pieces, etc.

3.1.2 *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.

3.1.2.1 *Discussion*—An oven-dried material will retain a small amount of moisture that 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.

3.1.2.2 *Discussion*—The term "mass" in the above definition is the correct designation for what is commonly designated "weight".

3.1.3 *vegetable matter base, n— in raw wool*, oven-dried scoured burrs, seeds, twigs, leaves, and grasses, free of mineral matter and alcohol-extractable matter.

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

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 D 1113 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 D 584. The procedure in Test Method D 1113 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 D 1113 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

[1] This test method is under the jurisdiction of ASTM Committee D-13 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] *Tariff Schedules of the United States*, Schedule 3, Part 1, Subpart C, Headnote 1 (*c*).

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 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 D 584 and D 1334, 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 D 584 and D 1334 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 D 584.

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 D 584.

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 D 584), take