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Standard Test Method for Extractable Matter in Wool and Other Animal Fibers[1]

This standard is issued under the fixed designation D 1574; 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 amount of extractable matter in samples of all forms of wool, except grease wool, that is extractable with ~~trichlorotrifluoroethane (CCl_2-$FCClF_2$).~~ a non-flammable vapor degreasing and cleaning solvent.

1.2 This test method does not cover the determination of the amounts of different components in the extracted matter nor their identification.

1.3 This test method is suitable for use with other animal fibers.

NOTE 1—The determination of extractable matter in yarns and in felts is covered in Test Methods D 2257 and D 461. For the determination of alcohol-extractable matter in oven-dry scoured wool, refer to Test Methods D 584 and D 1334.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *This standard does not purport to address 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.* See 5.4 and Note 3.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 123 Terminology Relating to Textiles
D 461 Test Methods for Felt
D 584 Test Method for Wool Content of Raw Wool—Laboratory Scale
D 1334 Test Method for Wool Content of Raw Wool—Commercial Scale
D 1576 Test Method for Moisture in Wool by Oven-Drying
D 2257 Test Method for Extractable Matter in Textiles
D 2462~~Test Method for Moisture in Wool by Distillation with Toluene~~[2] Test Method for Moisture in Wool by Distillation With Toluene
D 4845 Terminology Relating to Wool

3. Terminology

3.1 ~~*Definitions:*~~

~~3.1.1*animal fiber*, *n*—any natural protein-base fiber.~~

~~3.1.1.1*Discussion*—As used in this standard, "animal fiber" refers in particular to those fibers covered by the Wool Products Labeling Act of 1939 and "recycled wool" as defined in the Act as amended in 1980. It also includes, but is not limited to, those fibers listed in Table 1 of Terminology D123~~Definitions:

3.1.1 For definitions of textile terms used in this test method: animal fiber, extractable matter, grease wool, recycled wool, wool and wool, refer to Terminology D 4845.

~~3.1.2*extractable matter*, *n*—nonfibrous material in or on a textile, not including water, which is removable by a specified solvent or solvents as directed in a specified procedure.~~

~~3.1.2.1*Discussion*—For the purposes of this method, extractable matter does not include moisture but (*1*) is non-fibrous material, (*2*) is usually oily, waxy, or resinous in nature, and (*3*) may include protein, particularly if the extracting solvent is ethyl alcohol or contains ethyl alcohol.~~

~~3.1.3*grease wool*, *n*—wool taken from the living sheep and which has not been commercially scoured.~~

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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* ~~, Vol 07.01.~~volume information, refer to the standard's Document Summary page on the ASTM website.

~~3.1.4 *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."~~

~~3.1.4.1 *Discussion*—In the amended Act of 1980, the term "recycled wool" replaced the terms "reprocessed wool" and "reused wool."~~

~~3.1.5 *wool*, *n*—the fibrous covering of sheep, *Ovis* species.~~

~~3.1.5.1 *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.~~

~~3.1.6 *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."~~

~~3.1.7 For definitions of other textile terms used in this test method, refer to Terminology D123~~

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

4. Summary of Test Method

4.1 The specimen is extracted in a Soxhlet apparatus with a specified halogenated hydrocarbon solvent. The extract is filtered, the solvent is evaporated, and both the residue and the extracted specimen are dried and weighed. The amount of extractable matter is calculated and reported as a percentage of the oven-dry mass of the wool specimen, that is, the mass of the oven-dried extracted specimen plus the mass of the oven-dried extracted material.

4.2 Special procedures are provided to correct for errors in determining the amount of extractable matter as summarized in 4.1 if it is known or thought to contain volatile components.

5. Significance and Use

5.1 Test Method D 1574 is considered satisfactory for acceptance testing since the method has been used extensively in the trade for acceptance testing.

5.1.1 In case of a dispute arising from differences in reported test results when using Test Method D 1574 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 Student's *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 the light of the known bias.

5.2 This test method may be used to estimate the quantity of oil, grease, and waxy materials remaining on or in wool fibers after scouring, or the quantity of lubricant added before carding or remaining after carding, or the quantity of such materials added or removed in subsequent processing operations.

5.3 The residues obtained in this test may be subjected to chemical analysis for identification and assay of the component materials, if desired.

5.4 The specified solvent in this test method does not remove some materials, such as soaps, that may be present in wool and hence in some cases may reflect more closely the added content of some extractables in wool such as oils. When such materials are present and an estimate of their quantity is desired, some other solvent or combination of solvents should be used as specified in a material specification or by agreement. The information in the Annex of this test method may be useful for this purpose. Various solvents have been used in the past, and are still used to some extent. Many of these solvents are undesirable, however, because of flammability, toxic or anesthetic effects, or unpleasant odors. Trichlorotrifluoroethane is nonflammable, has a very low level of toxicity, and a high degree of stability and is a good solvent for most fatty or mineral oils, greases, and waxes. The specified solvent, under conditions of the test, was found in interlaboratory test not to extract wool protein.

NOTE 2—Since the specified solvent is primarily a solvent for oils rather than soap, the extractable matter obtained in this procedure may be considered a measure of commercial oil content.

NOTE 3—Due to the hazardous nature of some of the solvents listed in Table A1.1, the user should refer to the manufacturer's recommendations for use before using under the conditions of this test method.

6. Apparatus and Reagents

6.1 *Soxhlet Extraction Apparatus* , including an extraction tube, condenser, and flask. A large extraction tube having a standard-taper joints T55/50 and 24/40 and a 300-mL flask are recommended, but any size capable of holding at least a 10-g specimen is satisfactory.

6.2 *Heater for Extraction Apparatus* , preferably electric, thermostatically controlled, and of a design that avoids direct heating of the flask above the lowest solution level.

6.3 *Drying Oven*, ventilated either by natural convection or forced draft, thermostatically controlled and maintained at 105 ± 3°C throughout the oven chamber.

6.4 *Filter Paper*, [3] medium speed, retentive, in large sheets, 580 by 580 mm (23 by 23 in.), or in circles large enough to form a thimble of a size to fit the apparatus, or as an alternative, a cellulose extraction thimble.

6.5 *Weighing Containers*, of perforated metal if weighing of the specimen is to be performed in the drying enclosure; or containers that can be hermetically sealed (such as glass weighing bottles) if the specimen is to be cooled in a desiccator before weighing in the ambient atmosphere.

6.6 *Balance*, having a capacity adequate for weighing specimens, flasks, and containers and having a sensitivity of 0.001 g.

6.7 *Solvent*~~— Extraction grade trichlorotrifluoroethane~~— Ensolv.[4] ~~(CCl_2FCClF_2).~~

7. Sampling

7.1 *Lot Sample*— As a lot sample for acceptance testing, unless otherwise agreed upon between purchaser and supplier, take at random the number of packages of wool from a lot as directed in Table 1. Consider packages of wool as the primary sampling unit.

7.2 *Laboratory Sample*—Consider the package of wool in the lot sample as the laboratory sample.

7.3 *Test Specimen*—From each package in the laboratory sample take at random two specimens each weighing 10 ± 1 g.

7.3.1 If the determination of volatile matter in extractables, as described in 9.5.2, is to be undertaken, weigh the test specimen to the nearest 0.001 g and record this mass as *T*. This specimen is for extraction.

7.3.2 For each test specimen taken for extraction, take a second test specimen for moisture determination, weighing between 50 and 70 g. Weigh to the nearest 0.05 g the same time the extraction test specimen is weighed so that the moisture content of the two specimens at the time of weighing will be identical.

8. Conditioning

8.1 Neither preconditioning nor conditioning is necessary.

9. Procedure

9.1 Wrap the specimen in a single sheet of fresh filter paper (or place in an extraction thimble free of matter extractable by the specified solvent) and insert the assemblage in the extraction tube. Make certain that all open edges of the filter paper or thimble extend above the siphon tube.

9.2 Attach to the extraction tube a 300-mL flask that has previously been dried at 105 ± 3°C, cooled in a desiccator, and weighed to the nearest 1 mg.

9.3 Slowly pour sufficient solvent over the specimen to start the siphoning action. (If a thimble is used to hold the specimen, pour the solvent onto the specimen within the thimble). When the siphoning has stopped add another 20 mL of solvent. Connect the extraction tube to the condenser, place the flask on the heater, and extract for 20 siphon cycles. Adjust the heat so that the total time taken for 20 siphon cycles is 100 ± 10 min.

NOTE 4—The specified solvent, ~~trichlorotrifluoroethane,~~Ensolv, boils at ~~47.2°C~~69 ° C at sea level. However, at higher elevations the boiling point is reduced and care must be taken to ensure the specified rate of siphoning.

9.4 Following the twentieth cycle of siphoning, remove the flask and the extraction tube from the heater. Pull the specimen and filter paper (thimble) up to the mouth of the extraction tube and allow to drain until a state of dripping only is observed. Remove the specimen and filter paper (thimble) from the extraction tube and treat as described in 9.6.

9.5 Pour the solvent thus contained in the extraction tube into the flask. Reconnect the flask, extraction tube, and condenser and place back the flask on the heater to slowly boil off the solvent (for recovery if desired) into the extraction tube until the flask contains about 20 to 30 mL of solvent. Remove the flask from the assemblage and slowly evaporate the solvent, in the flask, below

[3] Whatman Filter Paper No. 2, or its equivalent, has been found satisfactory for this test method.

[4] ~~This solvent is available from various suppliers under various trade names. DuPont offers it as Freon TF and Allied Chemical Corp. offers it as Genesol D. In both these solvents, nonvolatile residue is claimed to be less than 2 ppm.~~

[4] This solvent is available from Enviro Tech International Inc., West LeMoyne, Melrose Park, IL 60160 (www.ensolv.com).

TABLE 1 Sampling Schedule for Wool in Package

Number of Packages in Lot	Number of Packages in Lot Sample
1 to 3	all
4 to 24	4
25 to 50	5
more than 50	10% of the packages with a maximum of 8