Designation: D1574 – 04 (Reapproved 2013)

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

This standard is issued under the fixed designation D1574; 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 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 D2257 and D461. For the determination of alcohol-extractable matter in oven-dry scoured wool, refer to Test Methods D584 and D1334.

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]

D123 Terminology Relating to Textiles
D461 Test Methods for Felt (Withdrawn 2003)[3]
D584 Test Method for Wool Content of Raw Wool—Laboratory Scale
D1334 Test Method for Wool Content of Raw Wool—Commercial Scale
D1576 Test Method for Moisture in Wool by Oven-Drying
D2257 Test Method for Extractable Matter in Textiles
D2462 Test Method for Moisture in Wool by Distillation With Toluene
D4845 Terminology Relating to Wool

3. Terminology

3.1 *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 D4845.

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 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 D1574 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 D1574 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

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[1] This test method is under the jursidiction 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.

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,* [4] 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*—Ensolv.[5]

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

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

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

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

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, Ensolv, boils at 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 the boiling point (care should be taken to avoid any loss of solvent and extractable matter by overheating and sputtering); then dry to constant mass, by placing the flask in an oven at 105 ± 3°C. Cool in a desiccator and weigh to the nearest 1 mg.

9.5.1 Certain oils and finishing agents may contain volatile components that may be lost during evaporation of the solvent and oven-drying. If the identification of the oil or finishing agent is known, a weighed specimen of the material may be dissolved in a fresh portion of the solvent, the solvent then being evaporated as described in 9.5, and the residue weighed to determine the volatility of the oil. A correction factor specific for the known oil may be calculated using Eq 1 and 2 and subsequently using the correction factor to determine the mass of oven-dried matter corrected for the loss of volatile material for use in 10.1, Eq 5:

$$M = S - V \tag{1}$$

$$F = S/(S - V) = S/M \tag{2}$$

$$C = MF \tag{3}$$

where:

M = mass of oven-dried extracted matter,
V = mass of original material volatilized,
S = mass of original material dissolved in solvent,
F = correction factor, and
C = mass of oven-dried extracted matter corrected for loss of volatile material.

9.5.2 If the oil or finishing agent is not known but is thought to contain volatile components, and if it is desirable to determine the total quantity of extractables including the volatile fraction, the following procedure may be used:

9.5.2.1 Using the second test specimen intended for moisture testing (see 7.3.2), determine the true-moisture content using Test Method D2462. This procedure determines moisture independent of volatile matter, which would be lost in an oven-drying procedure.

(1) Test Method D2462 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.

9.5.2.2 Extract the first test specimen as described in Section 9, having determined the mass *T* of the test specimen before extraction (see 7.3.1), and the mass *W* of the oven-dried test specimen after extraction.

9.5.2.3 *Rationale*—Extractable matter, as calculated using Eq 4, consists of non-volatile matter only. When the percent of true-moisture, *X*, is determined as directed in 9.5.2.1, the corresponding mass of moisture, *U*, can be calculated. When the weighed test specimen has been extracted and oven-dried, its mass, *W*, is determined. But *W* is also equal to *T*, less the moisture *U*, less the non-volatile extractables *M*, less the volatile extractables *N* (Eq 7), from the available experimental data (*T, U, M,* and *W*), the mass of the volatile extractable matter *N* can be calculated (Eq 8) and subsequently, the percent volatile matter *Y*, can be obtained (Eq 9).

9.6 Air-dry the extracted specimen, then place the specimen in a suitable container and dry in an oven at 105 ± 3°C to constant mass, defined as the absence of any progressive decrease in mass in excess of 0.10 % of the average, as determined by three successive weighings using the procedure in either 9.6.1 or 9.6.2 to obtain the oven-dry mass of the specimen.

9.6.1 If the weighings of the dried specimen are to be obtained with the specimen inside the oven, perform the weighings with any forced-air circulation turned off. Continue readings of mass until the conditions specified in 9.6 are achieved.

9.6.2 If the weighings of the dried specimen are to be obtained outside the oven, dry the specimen in a container provided with a tight-fitting cover with this cover removed while in the oven. At the end of the drying period, cover the container and remove it from the oven. Place the covered container in a desiccator, loosen the cover, and cool the specimen and container to approximately room temperature. When cooling is completed, set the cover firmly on the container and weigh the container, cover, and specimen. Replace the container and specimen in the oven, remove the cover, and dry. Repeat the cooling and weighing procedures. Continue this procedure until the conditions specified in 9.6 are achieved.

NOTE 5—If matter insoluble in Ensolv is to be determined, use a polar solvent, such as ethyl alcohol, and proceed as directed in 10.3 of Test Method D2257.

10. Calculation

10.1 Calculate the percentage of extractable matter on the oven-dry specimen basis using Eq 4 or Eq 5:

$$\text{Extractable Matter, \%} = 100 \times M/(W+M) \tag{4}$$

$$\text{Extractable Matter, \%} = 100 \times C(W+C) \tag{5}$$

where:

M = mass of oven-dried extracted matter,
W = oven-dried mass of the extracted specimen, and

C = mass of oven-dried extracted matter corrected for loss of volatile material (see 9.5.1).

10.1.1 Calculate the percentage of volatile extractable matter on the oven-dry extracted specimen basis using Eq 6-9.

$$U = X \times T/100 \qquad (6)$$

$$W = T - U - M - N \qquad (7)$$

$$N = T - U - M - W \qquad (8)$$

$$Y = 100 \times N/(W+N) \qquad (9)$$

where:

T = mass of test specimen before extraction,
X = percentage of true moisture in T,
U = mass of true moisture in T,
N = mass of volatile extractable matter, and
Y = percentage of volatile extractable matter.

10.2 Calculate the average of the results of tests on all specimens to the nearest 0.01 percentage points.

11. Report

11.1 State that the specimens were tested as directed in ASTM Test Method D1574. Describe the material or product sampled and the method of sampling used.

11.2 Report the following information:

11.2.1 Average percentage of extracted matter to two significant figures.

11.2.2 Number of specimens tested and the range of extracted matter (difference between highest and lowest result).

11.2.3 State whether the calculated percentage of extractable matter was based on the use of the uncorrected or corrected mass of oven-dried extracted matter.

12. Precision and Bias

12.1 *Summary*—In comparing two averages of four observations, the difference should not exceed 0.06 percentage points in 95 out of 100 cases when all of the observations are taken by the same well-trained operator using the same piece of test equipment and specimens randomly drawn from the same sample of material. Much larger differences are likely to occur under all other circumstances. The procedure in Test Method D1574 for measuring extractable matter in wool has no bias because the value of extractable matter in wool can be defined only in terms of a test method. Sections 12.2-12.4 explain the basis for this summary and for evaluations made under other conditions.

12.2 *Interlaboratory Test Data*[6]—An interlaboratory test was run in 1979 in which randomly drawn specimens of two wool tops were tested in each of eleven laboratories. Each laboratory used one operator who tested two specimens of each wool top. The components of variance expressed as standard deviations were calculated to be:

Single-Material Components:	
Within-laboratory component	0.041 percentage points
Between-laboratory component	0.217 percentage points
Multi-Material Components:	
Within-laboratory component[A]	0.041 percentage points
Between-laboratory component	0.217 percentage points

[A] The within-laboratory component for multi-material comparisons are in addition to the within-laboratory component for single material comparisons and are not reduced by replication.

NOTE 6—The square roots of the components of variance are being reported so that variability is expressed in the appropriate units of measure rather than as the square of those units of measure.

12.3 *Precision*—For the components of variance reported in 12.2, two averages of observed values should be considered significantly different at the 95 % probability level if the difference equals or exceeds the critical differences of Table 2.

NOTE 7—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 amount of statistical bias, if any, between them must be established with each comparison being based on recent data obtained on specimens from a lot of material of the type being evaluated so as to be as homogeneous as possible and then randomly assigned in equal numbers to each of the laboratories.

12.4 *Bias*—The procedure in Test Method D1574 for measuring extractable matter in wool has no bias because the value of extractable matter in wool can be defined only in terms of a test method.

12.4.1 Different solvents (see Annex A1) remove varying proportions of specific extractable materials even when the procedure is otherwise unchanged.

13. Keywords

13.1 Extractable matter; wool

TABLE 2 Critical Differences

Number of Observations in Each Average	Critical Differences for the Conditions Noted[A] 95 % Probability Level Units: Percentage Points	
	Within-Laboratory Precision	Between-Laboratory Precision
	Single-material Comparisons	
1	0.11	0.61
4	0.06	0.60
8	0.04	0.60
	Multi-material Comparisons	
1	0.16	0.62
4	0.13	0.61
8	0.12	0.61

[A] The critical differences were calculated using $z = 1.960$.

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[6] A copy of the research report is available from ASTM Headquarters, 100 Barr Harbor Drive, Conshocken, PA 19428. Request RR: D-13-1069.

ANNEX

(Mandatory Information)

A1. COMPARISON OF PERCENT MATTER EXTRACTED USING VARIOUS SOLVENTS

A1.1 *Test Data*—An experiment was conducted in 1966 in which a number of commercially available oils or finishing agents were applied to different portions of a scoured, carded wool (grade 56's). Specimens of each treated wool portion were extracted with a variety of commonly used solvents. All extractions were made by one operator. The results, expressed as percent extractable matter, are stated in Table A1.1.

TABLE A1.1 Relative Percentages of Extracted Matter, Recovered from Treated Wool When Using the Solvents Listed

Oil or Finishing Agent Added	Solvents									
	3:2 Benzene Methanol	Methanol	Trichlorethylene	Benzene	Methylene Chloride	Chlorothene NU	Petroleum Ether, 60 to 100°C BR[A]	*n*-hexane	Genesolv D	Petroleum Ether, 30 to 60°C BR[A]
Sulfol 448	3.83	3.57	3.48	3.40	3.43	3.29	2.82	2.74	2.43	2.36
	3.61	3.41	3.38	3.15	2.94	2.89	2.53	2.48	2.33	2.29
Lanolubric #2	4.43	4.17	3.72	3.79	3.80	3.63	2.90	3.39	3.08	2.92
	4.22	4.32	3.53	3.49	3.28	3.31	2.94	2.58	2.57	2.44
Worsted Oil #12	3.28	2.84	1.78	2.38	1.56	1.67	1.27	1.39	1.22	1.22
	2.60	2.82	1.63	1.52	1.41	1.29	1.29	1.24	1.01	1.34
Nopcostat Lv-40	2.74	2.54	1.96	1.92	1.83	1.83	1.50	1.46	1.50	1.29
	2.57	2.64	2.05	2.09	1.96	1.96	1.62	1.41	1.36	1.50
Twitchell 7421	3.76	3.70	3.32	3.17	2.06	3.11	2.28	2.51	2.43	2.17
	3.91	3.82	3.38	3.20	2.98	2.96	2.61	2.47	2.34	2.43
Twitchell 7440	2.37	2.29	1.43	1.86	1.23	1.47	1.09	0.95	0.76	0.95
	2.08	2.23	1.45	1.41	1.08	1.23	1.33	0.71	0.61	1.14
Emerstat 7451	2.11	2.16	1.10	1.26	1.21	0.95	0.74	0.84	0.58	0.58
	2.25	2.19	1.04	1.31	1.04	0.83	1.09	0.83	0.52	0.89
Emerlube 7484	3.15	3.05	3.02	2.47	2.45	2.45	2.33	2.08	1.95	1.79
	3.11	2.94	2.85	2.58	2.44	2.60	2.15	2.15	1.91	1.84
Textiline 3645	2.56	2.28	0.92	1.01	0.73	0.78	0.60	0.46	0.41	0.46
	2.51	2.30	1.13	0.91	0.77	0.63	0.90	0.72	0.59	0.58
Maxitol #10	3.25	3.25	2.63	2.60	2.60	2.57	2.20	2.06	2.02	2.00
	3.28	3.20	2.60	2.40	2.47	2.36	2.24	1.99	1.98	1.93
Dexene CS	5.81	5.41	4.76	4.66	4.54	4.78	4.42	4.28	4.31	3.91
	5.56	5.26	4.78	4.69	4.37	4.65	4.43	4.32	4.32	3.87
Polymene F	2.57	2.18	1.29	1.14	0.86	1.00	0.95	0.67	0.52	0.71
	2.80	2.70	1.12	0.85	0.75	0.80	1.04	0.62	0.52	0.65
Polymene 602	2.23	2.17	0.84	1.08	0.80	0.64	0.52	0.32	0.24	0.36
	2.26	2.32	0.84	1.04	0.80	0.88	0.76	0.36	0.36	0.55
Polymene AF	8.63	8.53	8.01	7.99	7.62	7.54	7.58	7.21	5.96	4.16
	8.62	8.60	7.93	7.82	7.77	7.77	7.80	7.50	6.44	4.32
Polymene WR	2.36	2.39	1.82	1.70	1.85	1.58	1.42	1.36	1.24	1.27
	2.51	2.60	1.87	1.75	1.71	1.63	1.60	1.45	1.37	1.32
Prosotex 161	2.42	2.32	1.72	1.61	1.29	1.48	1.27	1.12	1.27	0.97
	2.54	2.39	1.80	1.87	1.39	1.52	1.51	1.27	1.51	1.14

[A] BR-boiling range.