Designation: D 2257 – 9~~6~~8

Standard Test Method for Extractable Matter in Textiles[1]

This standard is issued under the fixed designation D 2257; 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 procedure for determining the extractable material on most fibers, yarns, and ~~fabrics.~~

~~1.1.1 The preferred solvent for use in this method is HH solvent (1,1,2-trichloro-1,2,2-trifluoroethane). By mutual agreement other solvents may be used.~~

~~1.1.2 This test method does not extract cross-linked finishes~~ fabrics. Three options are included. Option 1 uses heat and Soxhlet extraction apparatus. Option 2 uses room temperature and extraction funnels. Option 3 uses either Option 1 or ~~resins which may be on the textile.~~

~~1.2 *This standard does not purport to address all*~~ *Option 2 extraction but provides for calculation of extractable matter from the* ~~*safety concerns, if any, associated with its use. It is the responsibility*~~ *loss in mass of the* ~~*user of this standard*~~ *material due to* ~~*establish appropriate safety and health practices and determine*~~ *the* ~~*applicability of regulatory limitations prior to use.* See Note 2, 5.4.1, and Section 7.~~ *extraction rather than the extractable matter residue.*

NOTE 1—Other standards for the determination of extractable matter in textiles made of specific fibers ~~have been published by ASTM:~~ include: Specification D 541, Specification D 681, and Test Method D 1574.

1.1.1 The solvents for use in this method are any solvents that the party or parties concerned agreed on. Such as, Halogenated Hydrocarbon (HH) chloroform, tetrachloroethane, alcohol.

1.1.2 This test method may not extract cross-linked finishes or resins which may be on the textile.

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.* See Note 3, and Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

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[1] This test method is under the jurisdiction of ASTM Committee D-13 on Textiles and is the direct responsibility of Subcommittee D13.51 on Chemical Conditioning and Performance.

Current edition approved ~~May 10, 1996.~~ Sept. 10. Published ~~August 1996.~~ February 1999. Originally published as D 2257 – 89. Last previous edition D 2257 – 96.

D 123 Terminology Relating to Textiles[2]
D 541 Specification for Single Jute Yarn[2]
D 681 Specification for Jute Rove and Plied Yarn for Electrical and Packing Purposes[2]
D 1574 Test Method for Extractable Matter in Wool and Other Animal Fibers[2]
D 1909 Table of Commercial Moisture Regains for Textile Fibers[2]
D 2258 Practice for Sampling Yarn for Testing[2]
D 4290 Terminology for Moisture in Textiles[3]

2.2 *Other Documents:*

IWTO 10-62(E) Method for Determination of the Dichloromethane Soluble Matter in Combed Wool Sliver[4]

3. Terminology

3.1 *Definitions:*

3.1.1 ~~*commercial moisture regain*, *n*—an arbitrary value formally adopted as the regain to be used with the moisture-free mass when calculating the commercial mass of a shipment or delivery.~~

~~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~~1.1 *Discussion*—Nonfibrous material is usually oily, waxy, resinous, or polymeric in nature, but may also include other material, such as protein, particularly if ethyl alcohol is used, or in, the extracting solvent.

~~3.1.3 *wool*, *n*—as defined in the Wool Products Labeling Act~~

3.1.2 For definitions of ~~1939,[4] the fiber from the fleece of sheep or lamb, or hair of the angora goat or cashmere goat (and may~~

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[2] *Annual Book of ASTM Standards*, Vol 07.01.

~~[3] Available from International Wool Secretariat, Carlton Gardens, London S.W.1, England.~~

[3] *Annual Book of ASTM Standards*, Vol 07.02.

~~[4] Act of Congress, "Wool Products Labeling Act of 1939,'' 76th Congress, 3rd Session, approved October 14, 1940, and amended July, 1980.~~

[4] Available from International Wool Secretariat, Carlton Gardens, London S.W. 1, England.

~~include the so-called specialty fiber from the hair of the camel, alpaca, llama, and vicuna) which has never been reclaimed from any woven or felted wood product.~~

~~3.1.4 For~~ other moisture terms related to textiles, refer to Terminology D 4290[5]. 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 either in Soxhlet apparatus (Option 1), or extraction funnel (Option 2)first with~~ a halogenated hydrocarbon~~ an agreed solvent (Note~~ 2) and then with alcohol.~~ 2). The solvents are evaporated and the residues and the specimens are dried and weighed separately. The amounts of extracted matter are reported as percentages of either the oven-dried mass or of the oven-dried mass plus commercial moisture regain for the textile.

4.2 Alternatively, in Option 3, the specimen is dried and weighed before and after extraction using Option 1 or Option 2. Extractable matter is calculated as the loss in mass reported as percentages of the extracted oven-dried mass or this mass plus commercial moisture regain for the textile.

5. Significance and Use

5.1 This test method may be used for acceptance testing of commercial shipments.

5.1.1 In case of a dispute arising from differences in reported test results using this test method for acceptance testing of commercial shipments, the purchaser and the supplier should conduct comparative tests to determine if there is a statistical bias between the laboratories. Competent statistical assistance is recommended for the investigation of bias. As a minimum, the two parties should take a group of test specimens which are as homogeneous as possible and which 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~~ the Student's *t*-test~~ appropriate statistical analysis and~~ an acceptable~~ a probability level chosen by the two parties before the testing begins. If a bias is found, either its cause must be found and corrected, or the purchaser and supplier must agree to interpret future test results with consideration to the known bias.

5.2 This test method is used for the determination of the amounts of naturally present oily or waxy impurities that have not been completely removed from textiles made from animal fibers, and for the determination of the amounts of oily or waxy finishing materials applied to raw materials or textiles during manufacture. See~~ 3.1.2. The percentage of extracted~~ 3.1.1, *extractable matter* ~~*is usually calculated on the basis of the dry mass of the clean fiber, or on the basis of the mass of clean fiber plus commercial moisture regain.*~~ .

5.3 The test method may be used as a step in the determination of the commercial weight of fiber, yarn, and textile shipments.

5.4 The International Wool Textile Organization specifies the use of a halogenated hydrocarbon, dichloromethane, also called methylene chloride (CH_2Cl_2) instead of the solvent specified in this test method~~ (see Method~~ (IWTO-10-62(E)).

5.5 Extractables by mass loss is frequently used for~~ Determination~~ textiles which have a relatively large amount of~~ the Dichloromethane Soluble Matter in Combed Wool Sliver, IWTO-10-62(E)).~~

~~5.4.1 **Warning**—Dichloromethane is toxic, and its use is recommended only when necessary~~ extractable material to~~ conform to international specifications, and then with adequate ventilation under~~ effect a~~ hood.~~ significant mass change.

6. Apparatus and Reagents

6.1 *~~Soxhlet Extraction~~Extraction Apparatus*~~, equipped with fat-free cellulose~~

6.1.1 Soxhlet Extraction Apparatus for Option 1 extraction

6.1.2 Extraction Funnels, wide-mouth, 125 or~~ Alundum thimbles.~~ 150-mL capacity for Option 2 extraction.

6.2 *~~Ventilated Forced-Draft Drying Oven~~Thimbles*~~, capable of maintaining a temperature of 105 ± 3°C.~~ fat-free cellulose or Alundum, for Option 1.

6.3 *~~Analytical Balance~~Specimen Compressor*,~~ sensitive to 0.0001 g.~~ pestle or long forceps, for handling specimens in Option 2.

6.4 *~~Desiccator.~~*

~~6.5 *Halogenated Hydrocarbon Solvent*—1,1,2-trichloro1,2,2-trifluoroethane,[5] hereinafter called "HH solvent."~~ Containers,

6.4.1 To hold extractables and that will seal to prevent moisture changes, for example wighing bottles, for Option1.

6.4.2 To collect solvent from extractions, for Option 2.

NOTE~~ 2—**Precaution:** Various solvents have been used in the past, and~~ 2—If metal containers are~~ still used~~ used, check to~~ some extent. Many of these solvents are~~ ensure that the extracatable matter does not~~ recommended, however, because of flammability, toxic or anesthetic effects, or unpleasant odors. HH solvent~~ react with the metal if the reidue is~~ nonflammable, has a very low level~~ to be weighed.

6.5 *Oven*

6.5.1 *Ventilated Forced-Draft Drying Oven*, capable of~~ toxicity and~~ maintaining a~~ high degree~~ temperature of~~ stability. It can, however, exhibit dangerous anesthetic effects~~ 105 ± 3°C.

6.5.2 *Vacuum Type*, maintained at~~ high concentrations. As with all volatile solvents, the~~ 65 ± 2°C for use~~ of adequate ventilation under a hood is recommended~~ in Option 3 when~~ using this solvent.~~ low-boiling ingredients are present.

6.6 ~~*Ethyl Alcohol, Neutral* (95 %)—Either pure ethyl alcohol (C~~Tray, to contain desiccant, with a screen to prevent specimen or container contact with desiccant, for use in the vacuum oven. If Phosphorus pentoxide (P$_2$~~H~~0$_5$~~OH~~)) or~~ specially denatured~~

~~alcohol conforming~~ sulfuric acid (H$_{s04}$) is used, the screen must be acid reistant.

6.7 *Dessicator*

6.8 *Analytical Balance*—sensative to ~~Formula 40C~~ 0.0001 g.

6.9 *Nitrogen,* to supply the vacuum oven.

6.10 *Solvent*—as agreed (see 1.1.1).

NOTE 3—**Precaution:** Various solvents have been used in the past, and are still used to some extent. Many of these solvents are flammable, toxic or have anesthetic effects, or unpleasant odors. As with all volatile solvents, the ~~U.S. Bureau~~ use of ~~Internal Revenue may be used.~~ adequate ventilation under a hood is recommended when using this solvent.

7. Hazards

7.1 Refer to the manufacturer~~’~~’s material safety data sheets for specific information on chemicals used in this test.

7.2 After extraction with alcohol or other flammable or toxic solvents, the specimens must be air-dried under a hood until nearly all of the solvent has evaporated before they are dried in the oven~~, o~~. Otherwise there is a danger of building up a dangerous concentration of explosive vapor in the oven. Oven doors have been blown across the room by the force of an explosion.

7.3 **Warning**—Dichloromethane is toxic, and its use is recommended only when necessary to conform to international specifications, and then with adequate ventilation under a hood.

8. Sampling

8.1 *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 primary sampling unit.

NOTE ~~3~~4—An adequate specification or other agreement between the purchaser and the supplier requires taking into account the variability between shipping containers, within a shipping container, and between specimens taken from a single unit within a shipping container, so as to provide a sampling plan with a meaningful producer’s risk, consumer’s risk, acceptable quality level, and limiting quality level.

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

8.2.1 *Yarn on Packages*—Take at random from each shipping container in the lot sample the number of packages directed in an applicable material specification or other agreement between the purchaser and the supplier, such as an agreement to use Practice D 2258. Preferably, the same number of packages should be taken from each shipping case in the lot sample. If differing numbers of packages are to be taken from shipping cases in the lot sample, determine at random which shipping cases are to have each number of packages drawn.

8.2.2 *Yarn on Beams*—Take a laboratory sample from each of the beams in the lot sample as agreed between the purchaser and the supplier.

8.2.3 *Fabrics*—Take a full width swatch 1 m long from the end of each roll of fabric in the lot sample, after first discarding a minimum of 1 m of fabric from the very outside of the roll.

8.2.4 *Staple Fiber*—Systematically take five laboratory sample units from each bale in the lot sample as directed in Practice D 3333.

8.2.5 *Tow, Sliver, or Top*—Take from the lead end of a strand from each shipping container in the lot sample the first metre of material that has a clean, uniform appearance. If the shipping containers in the lot sample contain multiple packages, take a laboratory sample from one package drawn at random from each container.

8.3 *Test Specimens*—Take a 10 ± 1-g specimen from each unit in the laboratory sample. If necessary, take an additional specimen from a random unit in the laboratory sample until there is a total of ten specimens.

9. Conditioning

9.1 Neither preconditioning nor conditioning is necessary.

10. Procedure

10.1 *Presence of boiling ingredients* (extractable volatile at low temperatures).

10.1.1 Use the following procedure to determine the presence of low boiling ingredients, that is, extractables that are volatile at low temperatures.

10.1.~~1~~2 Extract one or more specimens of the material as directed in 10.2~~-~~ or 10.~~7~~3.

10.1.~~2~~3 Transfer the solvent ~~from the flask of the Soxhlet apparatus~~ quantitatively to a tared weighing bottle and evaporate the solvent by blowing with a stream of air at room temperature until there is a mass change of less than 0.001 g in 10 min.

10.1.~~3~~4 Heat the residue in an oven at 105 ± 3°C for 30 min, cool, and weigh.

10.1.~~4~~5 If there is a mass loss greater than 0.001 g in the residue (extractables) due to heating in the oven, the material contains extractable matter which volatilizes at low temperatures, and the specimen solvent extract should not be heated in the oven.

10.1.~~5~~6 If there is no detectable loss in residue mass (less than 0.001 g) due to heating in the oven, the material does not contain low boiling ingredients and the extracted residue is dried in the oven before final weighing.

10.2 *Option 1, Soxlet Extraction,*

10.2.1 Extract fat-free cellulose or Alundum thimbles for each specimen in the solvents to be used, in the Soxhlet chamber. A suitably folded qualitative grade filter paper may be used in place of the thimble.Thimbles are not needed for fabric yarn or thread specimens.

10.~~3~~2.2 Weigh 10 ± 1 g of each specimen to the nearest 0.010 g.

10.~~4~~2.3 Place the specimens in the thimbles, if used, and transfer to the Soxhlet apparatus. Thimbles must be long enough to cover the opening of the siphon tube.

10.~~5~~2.4 Add sufficient ~~HH~~ solvent to overflow plus about 25 % more to prevent the solvent from boiling dry.

10.~~6~~2.5 Adjust the heat for 3 to 5 siphonings per hour and extract the specimens for at least 20 siphonings. Maintain the temperature of the solvent in the Soxhlet apparatus at a level low enough to avoid interference with siphoning and in any event, below 50°C. Be certain that the solvent is going through the thimble or filter paper during the extraction, and not overflowing. In case there is evidence that 20 siphonings are not sufficient, the purchaser and supplier should agree upon a suitable number of siphonings.

10.~~7~~2.6 Remove from the source of heat when the upper chamber of the apparatus is nearly full and ready to siphon again. Pour off the nearly pure solvent from the extraction tube and save for reuse after suitable distillation.

10.~~8~~2.7 Determine the mass of a weighing bottle to 0.001 g. Transfer the contents of the flask to the tared weighing bottle, rinse with a small quantity of solvent, and add this to the contents of the weighing bottle.

10.~~9~~2.8 If low boiling ingredients were found (10.1.~~4~~5) evaporate the solvent as directed in 10.1.~~2~~3 without oven drying the residue.

10.~~10~~2.9 If low - boiling ingredients were not found, evaporate the solvent ~~as directed~~ and then dry the residue in ~~10.1.3. Repeat~~ an oven at 105 ± 3° C for 30 min. Cool in a desiccator and weigh. Repeat the drying, cooling, and weighing cycle until the difference ~~between successive weighings at~~ in intervals of 30 min results in a mass change of less than 0.001 g.

10.2.1~~1~~0 Record the final mass of the residue to the nearest 0.001 g.

NOTE 5—Solvent in weighing bottles may be recovered for subsequent reuse by connecting the effluent stream of solvent and air to a suitable condenser.

10.2.11 Remove the remaining solvent from the Soxhlet apparatus, reinsert the thimble and specimen, and fill with the proper amount of alcohol. Extract through at least 12 siphonings.

10.2.12 Determine the mass of a weighing bottle to 0.001 g. Transfer the alcohol from the flask to the weighing bottle, evaporate the alcohol, dry, and weigh the residue to the nearest 0.001 g.

10.2.13 Remove the specimen from the apparatus and air dry it. Continue as directed in 10.5.

10.3 *Option 2, room termperature extraction*:

10.3.1 Place the specimen in an extraction funnel.

10.3.2 Cover the specimen with solvent and let stand for about 10 min with occasional stirring to facilitate specimen surface and solvent contact.

10.3.3 Open the funnel stopcock and drain off the solvent into a weighed container. Compress the specimen to force out the excess solvent from the specimen.

10.3.4 Repeat 10.3.2 and 10.3.3 at least once more (Note 6).

NOTE 6—Two extractions will normally remove more than 90 % of solvent extractable matter. The number of solvent rinses will depend on how readily the surface material is removed and how much is present.

10.3.5 If low boiling ingredients were found (10.1.5), evaporate the solvent as directed in 10.1.3 without oven drying the residue.

10.3.6 If low-boiling ingredients were not found, evaporate the solvent and then dry the residue in an oven at 105 ± 3°C for 30 min. Cool in a desiccator and weigh. Repeat the drying, cooling and weighing cycle until the difference in intervals of 30 min results in a mass change of less than 0.001g.

10.3.7 Repeat 10.3.1 through 10.3.7 using alcohol.

10.3.8 Remove the specimen from the funnel and air dry it. Continue as directed in 10.5.

10.4 *Option 3, extractable matter by loss in mass:*

10.4.1 Dry the specimen as directed in 10.5.1 or 10.5.2 depending on the presence of low-boiling ingredients.

10.4.2 Extract the specimen with solvent as directed in 10.2 or 10.3, except the solvent extract is not saved for the extractable matter residue.

10.4.3 Air dry the specimen and oven dry it by the same procedure as used in 10.5.1.

10.5 *Specimen drying*:

10.5.1 For Options 1 and 2, and Option 3, when no low-boiling ingredients are present:

10.5.1.1 Dry the air-dried specimen in a tared weighing container in the oven at 105 ± 3°C for 1 h. Cool it in a desiccator and weigh it. Repeat the drying, cooling and weighing cycle until the change in specimen mass in two successive weighings at interals of 30 min is within 0.01%. Record the mass of the dry specimen to the nearest 0.01g.

10.5.2 For Option 3, when low-boiling ingredients are present:

10.5.2.1 Set the temperature of the vacuum oven for 65 ± 2°C and turn on the nitrogen supply.

10.5.2.2 Place the tray of desiccant, with screen cover, in the oven.

~~10.12 Remove~~

NOTE 7—If silica gell or $CaCO_3$ is used, avoid stirring up the~~ remaining HH solvent from~~ dust.

NOTE 8—**Precaution:** If P_2O_5 or H_2SO_4 is used, excercize extreme care in handling and avoid spills. See Section 7.

10.5.2.3 Place the~~ Soxhlet apparatus, reinsert the thimble~~ specimen loosely over a tared wighing container and~~ specimen, and fill with~~ the~~ proper amount of alcohol. Extract through at least 12 siphonings.~~

~~10.13 Determine~~ container cover on the~~ mass of~~ screen.

10.5.3 Dry the specimen for 4-6 h under low pressure with a~~ weighing bottle to 0.001 g. Transfer~~ nitrogen sweep. Tuck the ~~alcohol from~~ specimen in the~~ flask~~ container and transfer with the cover to cool in the~~ weighing bottle, evaporate~~ desiccator.

NOTE 9—It may be necessary to close the~~ alcohol, dry,~~ containers during transfer depending on the distance between the oven and~~ weigh~~ the desiccator. A partial vacuum develops while closed~~u~~ hot containers are cooling, especially if the containers are glass. This partial vacuum will impart a buoyancy to the~~ nearest 0.001 g.~~

~~10.14 Air-dry~~ container, decreasing its apparent mass if not released. Therefore, leave the~~ extracted specimen. Then heat in an oven at 105 ± 3°C until~~ covers off while the~~ change~~ containers are in~~ mass is no more than 0.010 g in 15 min when weighed in~~ the~~ oven or when placed~~ desiccator.

10.5.4 Weigh the specimen in~~ a tared weighing bottle and then cooled in an efficient desiccator and weighed outside~~ the~~ oven.~~

~~10.15 Record~~ container with the~~ mass of~~ cover on.

10.5.5 Continue the~~ oven-dry specimen~~ drying, cooling and~~ the~~ weighing~~ bottle, if used,~~ cycle until the change in specimen mass in two successive weighings is within 0.01%. Record the mass to the nearest 0.010 g for Options 1 and 2, and 0.0001 g for Option 3.

11. Calculation

11.1 From extractable material residues:

11.1.1 Calculate the~~ percentages~~ ammount of~~ the HH~~ solvent~~ and alcohol extracts~~ extractable material for each specimen to the nearest 0.1 % using Eq~~ 1-4:~~ 1 through Eq 3:

$$S_d = 100F/W \quad (1)$$

~~$A_d = 100\,L/W$ (2)~~

$$S_c = 100S_d/(100 + R)$$ ~~(3)~~

~~(4) $Ac = 100Ad/(100 + R)$~~

Sc F W

where:

S_d = ~~HH~~solvent extractable, dry basis, %,

~~A_d = alcohol extractable, dry basis, %,~~

S_c = ~~HH~~solvent extractable, commercial~~ mass~~ moisture regain basis, %,

~~A_c = alcohol extractable, commercial mass basis, %,~~

F = mass of~~ HH~~ solvent residue,

~~L = mass of alcohol residue,~~

W = oven-dry mass of test specimen~~ after~~ before extraction, and

R = commercial moisture regain (see Table D 1909).

If the textile is a blend of two or more types of fibers, calculate R according to Eq~~ 5:~~ 3:

$$R = 0.01 \times (A \times R_a + B \times R_b + \;....) \quad (3)$$

where:

A = fiber A in the blend, %,

B = fiber B in the blend, %,

R_a = commercial moisture regain for fiber A~~, and~~ (from Table D 1909), % ,

R_b = commercial moisture regain for fiber B (from Table D 1909), %.

11.2 From loss in mass:

11.2.1 Calculate the ammount of solvent extractable material for each specimen to the nearest 0.1%, using Eq 4 and 2:

$$S_d = (W_1 - W_2)/W_1 \quad (4)$$

where:

W_1 = oven-dried mass of specimen before solvent extraction, g,

W_2 = oven-dried mass of specimen after solvent extraction, *g*.

11.~~2~~3 Averages:

11.3.1 Calculate the~~ percentage of extracted matter for each~~ average solvent~~ separately.~~ extractable material for the lot.

12. Report

12.1 State that the specimens were tested as directed in~~ ASTM~~ Test Method D 2257. Describe the material(s) or product(s) sampled and the method of sampling used.

12.2 Report the following information:

12.2.1 ~~Id~~The~~n~~ option used, and, if~~y~~ Option 3, which extraction was used,

12.2.2 the exact name of the solvents used,

12.2.3 the solvent, the individual values obtained for the solvent~~ used~~ and the~~ average percentage of extracted~~ percent extractable matter~~ separately.~~

~~12.2.2 State whether~~ for the lot, and

12.2.4 whether results are reported on the oven-dr~~y~~ied of commerical moisture regain basis or~~ commercial mass basis.~~ oven-dried weight.

13. Precision and Bias

13.1 *Within-Laboratory Test Data*—~~T~~For Option 1, the estimate of the standard deviation of duplicate extractions from 160 lots performed in one laboratory was 0.16 percentage points.

13.2 *Within-Laboratory (Multi-Operator) Precision*—For the component of variance reported in 13.1, two averages of observed values should be considered significantly different at the 95 % probability level if they differ by the following critical differences or~~ more:~~

more:

Observations in Each Average	Critical Differences Percentage Point
1	0.44
2	0.31
3	0.26
5	0.20
10	0.14

13.3 *Bias*—The ~~procedure~~ options in this test method ~~has~~ have no bias because the value of the extractable matter in textiles can be defined only in terms of a test method.

14. Keywords

14.1 extractable matter; fabric; textile fabric; textile strand

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