SLOVENSKI STANDARD
oSIST prEN ISO 17751-2:2022

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Tekstilije - Kvantitativna analiza kašmirskih, volnenih, drugih specialnih živalskih vlaken in njihovih mešanic - 2. del: Metoda štetja z elektronskim mikroskopom (ISO/DIS 17751-2:2022)

Textiles - Quantitative analysis of cashmere, wool, other specialty animal fibres and their blends - Part 2: Scanning electron microscopy method (ISO/DIS 17751-2:2022)

Textilien - Quantitative Analyse von Kaschmir, Wolle, anderen speziellen tierischen Fasern und deren Mischungen - Teil2: Rasterelektronenmikroskopie-Verfahren (ISO/DIS 17751-2:2022)

Textiles - Analyse quantitative du cachemire, de la laine, d'autres fibres animales spéciales et de leurs mélanges - Partie 2: Méthode par microscopie électronique à balayage (ISO/DIS 17751-2:2022)

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59.060.10	Naravna vlakna	Natural fibres

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Textiles — Quantitative analysis of cashmere, wool, other specialty animal fibres and their blends —

Part 2:
Scanning electron microscopy method

Partie 2: Méthode par microscopie électronique à balayage

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Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 38, *Textiles*.

This second edition cancels and replaces the first edition (ISO 17751-2:2016), which has been technically revised.

The main changes are as follows:

— Clause 2 (Terms and definitions) has been added;

— in 3.1, a note to different types of specialty animal fibres has been added;

— the title of Subclause 7.2 has been changed from “Preparation method for test specimens of various types of samples” to “Preparation method for test specimens”;

— in 7.2.4.1, missing information on marking of masses of warp and weft yarns and laboratory sample have been supplemented;

— the title of Subclause 8.1 has been changed from “Test on each test specimen stub” to “Preparation and test on test specimen stub”;

— the title of Subclause 8.2 and has been changed from “Qualitative analysis(Purity analysis) and determination of fibre content” to “Qualitative analysis(Purity analysis)”;

— A new Subclause 8.3 and the title “Quantitative analysis” has been added;

— the title of Clause 9 has been changed from “Calculation of test result” to “Calculation and expression of test result”;

— Subclause 9.1 and the title “Calculation of the test result” has been added;

— Subclause 9.2 and the title “Expression of the test result” and its contents has been added;

— Clause 10 (Test report) and its contents has been added;

— the property of Annex A has been changed from informative to normative;

— in Annex C, density of some fibres has been modified and the density of coarse rabbit hair has been added;

— in Annex C, a footnote has been added to both fine rabbit and coarse rabbit;

— two references have been added in the bibliography;

A list of all parts in the ISO 17751 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Cashmere is a high value specialty animal fibre, but cashmere and other animal wool fibres such as sheep's wool, yak, camel etc., exhibit great similarities in their physical and chemical properties, so that their blends are difficult to distinguish from each other by both mechanical and chemical methods. In addition, these fibres show similar scale structures. It is very difficult to accurately determine the fibre content of such fibre blends by current testing means.

Research on the accurate identification of cashmere fibres has been a long undertaking. At present, the most widely used and reliable techniques include the light microscopy (LM) method and the scanning electron microscopy (SEM) method. The SEM method shows complementary characteristics to those of LM method.

— The advantage of LM method is that the internal medullation and pigmentation of fibres can be observed; the disadvantage is that some subtle surface structures cannot be clearly displayed. A decolouring process needs to be carried out on dark samples for testing, An improper decolouring process can affect the judgment of fibre analyst.

— The SEM method shows complementary characteristics to those of LM method, so some types of fibres need to be identified by scanning electron microscope.

The LM and SEM methods need be used together to identify some difficult-to-identify samples in order to utilize the advantages of both methods.

It has been proven in practice that the accuracy of a fibre analysis is highly related to the ample experience, full understanding, and extreme familiarity of the fibre analyst to the surface morphology of various types of animal fibres. So besides the textual descriptions, several micrographs of different types of animal fibres are given in Annex B.

Textiles — Quantitative analysis of cashmere, wool, other specialty animal fibres and their blends —

Part 2: Scanning electron microscopy method

1 Scope

This part of ISO 17751 specifies a method for the identification, qualitative, and quantitative analysis of cashmere, wool, other specialty animal fibres, and their blends using scanning electron microscopy (SEM).

This part of ISO 17751 is applicable to loose fibres, intermediate products, and final products of cashmere, wool, other specialty animal fibres, and their blends.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
specialty animal fibre
any type of keratin fibre taken from specialty animals(hairs) other than sheep

Note 1 to entry: Specialty animal fibres include cashmere, camel hair, yak, mohair, angora or rabbit, alpaca, etc.

3.2
scanning electron microscope
intermediate type of microscopic morphology observation instrument between transmitted electron microscope and light microscopewhich use a focused beam of high-energy electrons to generate a variety of physical information signals

Note 1 to entry: The principle consists of scanning a primary focused electron beam over a whole area of interest on the surface of solid test specimen, and the signal derived from which is then received, amplified and displayed in images for full observation of surface area topography of the test specimen.

Note 2 to entry: The signals obtained by a scanning electron microscope are, e.g., secondary electrons, Auger electrons, characteristic X-ray etc.

3.3
secondary electron
low-energy extra-nuclear electron released from and by ionization of a metal atom in the 5 nm to 10 nm scanned region of metal layer less than 10 nm thick nearest to the outermost meta-coated surface of a test specimen under impact of the focused primary electron beam of energy in units of tens of keV

Note 1 to entry: Being surface sensitive because of the small mean free path of the electron to escape from deep within the test specimen and, therefore the signal of which produces the highest-resolution morphological images of the coated surface.

3.4
scale
cuticle covering the surface of animal fibres

3.5
scale frequency
number of scales along the fibre axis per unit length

3.6
scale height
height of the cuticle at the scale's distal edge

3.7
fibre surface morphology
sum of the physical properties/attributes characterizing the fibre surface

EXAMPLE The fibre surface morphology includes scale frequency, scale height, patterns of scale edge, scale surface smoothness, fibre evenness along its axis, transparency under light microscope etc.

3.8
lot sample
portion representative of the same type and same lot of material drawn according to the requirements from which it is taken

3.9
laboratory sample
portion drawn from a lot sample according to the requirements to prepare test specimens

3.10
test specimen
portion taken from fibre snippets randomly cut from a laboratory sample for measurement purposes

4 Principle

A longitudinal view image of fibre snippets representative of a test specimen coated with a thin layer of gold and/or other metals is produced by a scanning electron microscope through scanning the side surface of the test specimen with a focused incident beam of high-energy electrons, detecting signals of secondary electrons emitted by the gold atoms excited when hit by the incident electron beam, and combining the beam position with the detected signals which contain information on surface topography of the test specimen.

All fibre types found in the test specimen are identified by comparing them with known fibre surface morphologies for different types of animal fibres.

For each fibre type, the number and diameter of fibre snippets are counted and measured. The mass fraction is calculated from the data for the number of fibre snippets counted, mean value, and standard deviation of the snippet diameter and the true density of each fibre type.

5 Apparatus

5.1 Scanning electron microscope, comprised of a vacuum system, electronic optical system, signal collecting and imaging system, display system, and measurement software.

5.2 Sputter coater with a gold and/or other metal cathode.

5.3 Microtome or double blades.

6 Reagents and materials

6.1 Glass tube, 10 mm to 15 mm in diameter.

6.2 Stainless-steel rod, approximately 1 mm in diameter.

6.3 Glass plate, measuring approximately 150 mm x 150 mm.

6.4 Double-sided adhesive tape.

6.5 Tweezers, scissors.

6.6 Test specimen stub, aluminium or brass, 13 mm in diameter.

6.7 Razor blade.

6.8 Acetone, analytical grade.

6.9 Ethyl acetone, analytical grade.

7 Sampling

Drawing the lot and laboratory samples in accordance with sampling method specified in Annex A.

8 Preparation of test specimen

8.1 Number of test specimen

Prepare a set of test specimen composed of 5 test specimen stubs. Fibre snippets on the test specimen stubs shall be sufficient to ensure at least 1 000 fibres snippets are examined.

8.2 Preparation method for test specimen

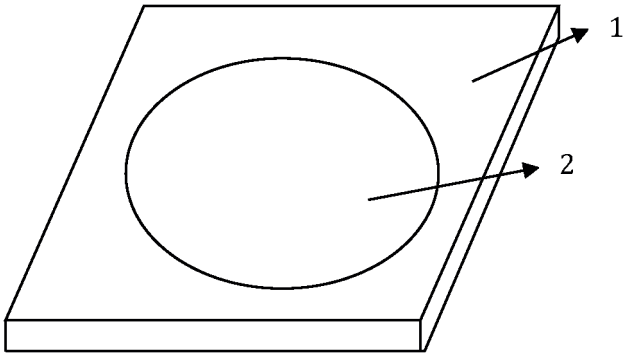
8.2.1 Loose fibre

8.2.1.1 Place the laboratory sample flat on the test table, pick up approximately 500 mg of fibres randomly on not less than 20 spots with tweezers from the top and bottom sides of the sample. Blend them homogeneously, and divide them into 3 equal portions. Sort those drawn fibres into basically parallel fibre bundles.

8.2.1.2 Cut each bundle in the middle with a microtome and razor blade or double blades to get approximately 0,4 mm long fibre snippets. Cut only once in each of the fibre bundles.

8.2.1.3 Collect all fibre snippets in the glass tube and suspend them in 1 ml to 2 ml acetone or ethyl acetate by stirring the mixture with a stainless-steel rod. Pour the suspension onto a glass plate to ensure that the fibre snippets are uniformly distributed on a spot of approximately 10 cm in diameter on the glass plate, as shown in Figure 1.

8.2.1.4 Press the double-sided adhesive tape on the mounting stubs and use a razor blade to trim the adhesive tape away from around the mounting stubs. After all the acetone or ethyl acetate in the fibre snippets suspension has evaporated, press the mounting stubs with the adhesive tape end onto the glass plate at the positions shown in Figure 2. Transfer the uniformly mixed fibre snippets to the adhesive tape on the test specimen stub.

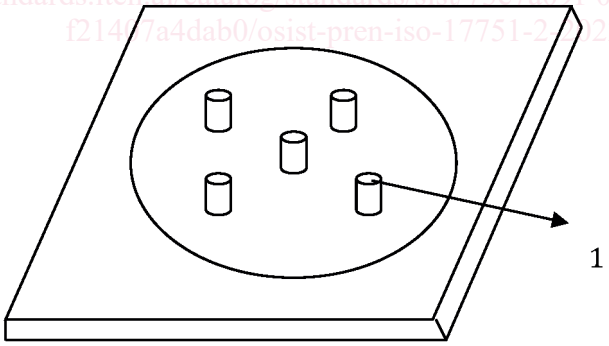


Key

1 glass plate

2 fibre snippets

Figure 1 — Fibre suspension on glass plate



Key

1 test specimen stub

Figure 2 — Positions of test specimen stubs

8.2.1.5 If the fibre snippets have aggregated after the evaporation of the acetone or ethyl acetate, they shall be recollected by scraping them off the glass plate with a razor blade and repeat operation procedures 8.2.1.3 and 8.2.1.4.

8.2.2 Sliver

8.2.2.1 Cut the laboratory sliver sample into three sections. Take out an appropriate amount of the fibre bundle in the longitudinal direction from each sliver section.

8.2.2.2 Cut in the middle of each fibre bundle to obtain approximately 0,4 mm long fibre snippets with a microtome and razor blade. Cut only once in each fibre bundle.

8.2.2.3 Other operation procedures are the same as described in 8.2.1.3 to 8.2.1.5.

8.2.3 Yarn

8.2.3.1 Divide the laboratory sample into three equal portions.

8.2.3.2 Cut each portion in the middle with a microtome and razor blade to obtain approximately 0,4 mm long fibre snippets. Cut only once in each yarn portion.

8.2.3.3 Other operation procedures are the same as described in 8.2.1.3 to 8.2.1.5.

8.2.4 Woven fabrics

8.2.4.1 If the warp and weft yarn share the same composition, all the yarn segments unravelled from a square sample of a complete pattern may be cut to obtain an appropriate test specimen. For those fabric samples composed of different compositions of warp and weft yarns, unravel the warp and weft yarns separately, weigh them and record their masses as M_T and Mw respectively. If the fabrics have a definite repetition in the pattern, unravel at least the integral multiple of a complete pattern. The unravelled warp and weft yarn bundles are kept as warp and weft yarn samples respectively as the laboratory sample.

8.2.4.2 Cut once from the parallel yarn portion in the middle with a microtome or razor blade to obtain approximately 0,4 mm long fibre snippets. Cut only once in each yarn segments.

8.2.4.3 Other operation procedures are the same as described in 8.2.1.3 to 8.2.1.5.

8.2.5 Knitted fabrics

8.2.5.1 Unravel at least 25 yarn segments from the laboratory sample for woollen knitted fabrics. Unravel at least 50 yarn segments for worsted knitted fabrics. Cut each yarn portion in the middle to obtain approximately 0,4 mm long fibre snippets. Cut only once in each yarn portion.

8.2.5.2 Other operation procedures are the same as described in 8.2.1.3 to 8.2.1.5.

8.3 Coating of the test specimen

Use the sputter coater to apply a thin layer of gold and/or other metals to the test specimen on test specimen stub.

9 Test Procedure

9.1 General

When possible, it is recommended to carry out the analysis of the two test specimens independently by two operators.

9.2 Preparation and test on test specimen stub

9.2.1 Place a stub with the test specimen into the test chamber of the SEM. First, view the selected stub at a lower magnification (for example, at ×10). Then, selecting from an area near the upper left

edge of the stub on the monitor, set the magnification to ×1000, scan the stub and observe the fibres. Identify the fibre types according to characteristics of the fibre morphologies (see details in Annex B) of cashmere, sheep's wool and other animal fibres.

9.2.2 Return to the lower magnification after identifying all fibres in the selected area. Choose another observation area along vertical or horizontal direction, repeat the above operations until finished, scanning the entire stub before continuing on to analyse fibre snippets on another stub.

9.3 Qualitative analysis (purity analysis)

Examine 150 fibres on the first test specimen stub. The following three conditions may happen.

— Case 1: If only one fibre type is found, examine another 300 fibre snippets on a second stub. If no fibre of a second type is found, the sample is declared as pure.

— Case 2: If two fibre types are found and the amount of one type is lower than 3% by number (less than 5 fibres of the second type), it is considered as a minor component. Examine 300 further snippets from the second stub and calculate the percentage by number of the two types of fibres.

— Case 3: if two or more fibre types are found andthe fibre mixture is considered to be a blend, perform a quantitative analysis according to 8.3.

9.4 Quantitative analysis

If the sample is found to be a blend, examine 220 further fibres and measure the diameters of the first 25 fibres of each component identified, (or all fibres of that component, if less than 25) on each of the remaining four stubs. At least a total of 1 030 fibres shall be identified for a sample and 100 measurements of fibre diameter are made for each component. The mean fibre diameter of each component is calculated according to diameters measured for the 100 fibres. If the total amount of each component is less than 100, calculate the mean fibre diameter according to the actual number of that fibre component.

This diameter is measured in vacuum condition and is not comparable to diameter measured by other instruments. So the value shall only be used for calculation of fibre content of each component in Clause 10.

10 Calculation and expression of test result

10.1 Calculation of test result

10.1.1 Calculate the mass fraction of each component with Formula (1). Density of various types of animal fibres is specified in Annex C.

$$w_i = \frac{N_i\left(D_i^2 + S_i^2\right)\rho_i}{\sum\left[N_i\left(\left(D_i^2 + S_i^2\right)\rho_i\right)\right]} \times 100 \quad (1)$$

where

w_i is the mass fraction of the component, %;

N_i is the number of fibres counted for the component;

S_i is the standard deviation of mean fibre diameter of the component, in micrometres (μm);

D_i is the mean fibre diameter of the component, in micrometres (μm);

ρ_i is the density of the component, in grams per millilitre (g/cm[3]).

10.1.2 Calculate the mass fraction of a fibre component in woven fabric samples composed of different warp and weft yarn compositions with Formula (2).

$$w_i = \frac{w_{iT} \times m_T + w_{iW} \times m_W}{m_T + m_W} \times 100 \qquad (2)$$

where

w_i is the mass fraction of the component in woven fabric sample, %;

w_{iT} is the mass fraction of the component in the warp yarns of the woven fabric sample, %;

m_T is the mass of the warp yarns in the woven fabric sample, in grams(g);

w_{iW} is the mass fraction of the component in the weft yarns of the woven fabric sample, %;

m_W is the mass of the weft yarns in the woven fabric sample, in grams(g).

10.2 Expression of the test result

Take the mean value of calculations of the two tests as the test result. If the difference between two tests is larger than 3.0 %, the third test specimen shall be tested. In such a case, the mean value of the three tests is taken as the test result. Fibre content percentage of angora or rabbit is the sum of percentages of both coarse and normal angora or rabbit hairs.

Test result of fibre content is rounded to one decimal.

10.3 Test report

Test result shall at least include the following information:

a) reference to this document;

b) sample description;

c) test results;

d) any deviations from the procedure;

e) the date of the test;

f) any unusual features observed.

