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

Part 2:
Scanning Electron Microscopy method

Textiles — Analyse quantitative du cachemire, de la laine, d'autres fibres animales spéciales et leurs mélanges —

Partie 2: Méthode par microscopie électronique à balayage

Please see the administrative notes on page iii

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ISO/CEN PARALLEL PROCESSING

This final draft has been developed within the International Organization for Standardization (ISO), and processed under the **ISO-lead** mode of collaboration as defined in the Vienna Agreement. The final draft was established on the basis of comments received during a parallel enquiry on the draft.

This final draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel two-month approval vote in ISO and formal vote in CEN.

Positive votes shall not be accompanied by comments.

Negative votes shall be accompanied by the relevant technical reasons.

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Foreword

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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).

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The committee responsible for this document is ISO/TC 38, *Textiles*.

ISO 17751 consists of the following parts, under the general title *Textiles — Quantitative analysis of cashmere, wool, other speciality animal fibres and their blends*:

— *Part 1: Light Microscopy method*

— *Part 2: Scanning Electron Microscopy method*

Introduction

Cashmere is a high value speciality 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 works on accurate identification of cashmere fibre has been a long undertaking. At present, the most widely used and reliable ones include Light Microscopy (LM) method and Scanning Electron Microscopy (SEM) method. The advantage of LM method is that the internal medullation and pigmentation of fibres can be observed, but some subtle surface structures are not able to be clearly displayed. A decolouration process needs to be carried out on dark samples for testing while improper decolouration process will affect the judgment of fibre analyst. The Scanning Electron Microscopy (SEM) method shows complementary characteristics to those of LM method so some types of fibres need to be identified by scanning electron microscope. Both Light Microscopy method and Scanning Electron Microscopy method need be used together to identify some difficult-to-be-identified samples in order to utilize the advantages of both methods.

It is proven in practice that accuracy of fibre analysis is highly related to the ample experience, fully understanding, and extreme familiarity of the fibre analyst to the surface morphology of various types of animal fibres so besides text description, a large amount of micrographs of different types of animal fibres are given in the Annex of this part of ISO 17751.

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

2 Terms and definitions

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

2.1
specialty animal fibre
any type of keratin fibre taken from animal (hairs) other than sheep

2.2
scanning electron microscope
intermediate type of microscopic morphology observation instrument between transmitted electron microscope and light microscope which 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 specimen and the signal derived from which is then received, amplified, and displayed in images for full observation of surface area topography of the specimen.

Note 2 to entry: The signals obtained by a scanning electron microscope are, e.g. *secondary electrons* (2.3), Auger electrons, characteristic X-ray, etc.

2.3
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 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 specimen and, therefore, the signal of which produces the highest-resolution morphological images of the coated surface.

2.4
scale
cuticle covering the surface of animal fibres

2.5
scale frequency
number of *scales* (2.4) along fibre axis per unit length

2.6
scale height
height of the cuticle at the *scale's* (2.4) distal edge

2.7
fibre surface morphology
sum of physical properties/attributes characterizing the fibre surface

EXAMPLE The fiber surface morphology includes *scale frequency* (2.5), *scale height* (2.6), patterns of scale edge, scale surface, smoothness, fibre evenness along its axis, transparency under light microscope, etc.

2.8
lot sample
portion representative of the same type and same lot of material drawn according to requirements from which it is taken

2.9
laboratory sample
portion drawn from *lot sample* (2.8) according to requirements to prepare specimens

2.10
test specimen
portion taken from fibre snippets randomly cut from *laboratory sample* (2.9) for measurement purposes

3 Principle

A longitudinal view image of fibre snippets representative of a test specimen coated with a thin layer of gold 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 the difference in known fibre surface morphology among different types of animal fibres.

Number and mean diameter of fibre snippets are counted and measured respectively for each fibre type. Mass percentage is calculated from the data for the number of fibre snippets counted, mean value, and standard deviation of snippet diameter and true density to each fibre type.

4 Apparatus, tools, and reagents

4.1 Apparatus

4.1.1 Scanning electron microscope.

The scanning electron microscope proper shall comprise the following components: vacuum system, electronic optical system, signal collecting and imaging system, display system, and measurement software.

4.1.2 Sputter coater with a gold cathode.

4.2 Accessories

4.2.1 Microtome.

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

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

4.2.4 Glass plate, measuring approximately 150 mm × 150 mm.

4.2.5 Double-sided adhesive tape.

4.2.6 Tweezers, scissors.

4.2.7 Specimen stub, aluminium or brass, 13 mm in diameter.

4.3 Reagents

Acetone (analytical grade) or ethyl acetate (analytical grade).

5 Sample drawing

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

6 Preparation of test specimens

6.1 Number of test specimens

Prepare five specimen stubs. Fibre snippets on specimen stubs shall be sufficient to ensure at least 1 000 fibres to be examined.

6.2 Preparation method for test specimen of various type of samples

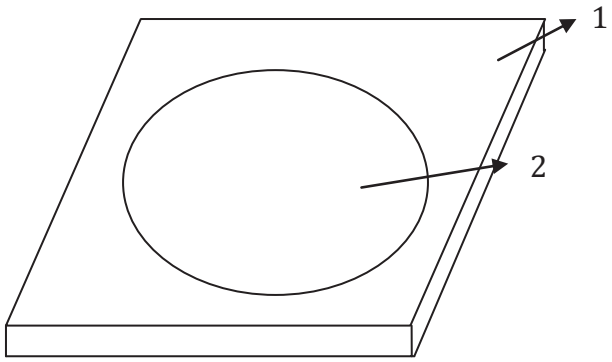
6.2.1 Loose fibre

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

6.2.1.2 Cut the fibre bundle in the middle with microtome to get approximately 0,4 mm long fibre snippets. Cut only once in each of the fibre bundles.

6.2.1.3 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.

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

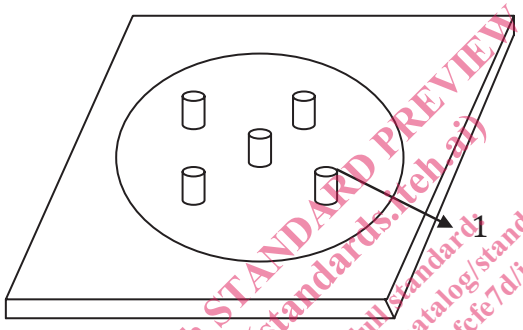


Key

1 glass plate

2 fibre snippets

Figure 1 — Fibre suspension on glass plate



Key

1 specimen stub

Figure 2 — Positions of specimen stubs

NOTE 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 procedures 6.2.1.3 and 6.2.1.4.

6.2.2 Sliver

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

6.2.2.2 Cut in the middle of each fibre bundle to obtain approximately 0,4 mm long fibre snippets with microtome. Cut only once in each fibre bundle.

6.2.2.3 Other operation procedures are the same as stipulated in 6.2.1.3 and 6.2.1.4.

6.2.3 Yarn

6.2.3.1 Divide laboratory sample into three equal portions.

6.2.3.2 Cut each portion in the middle with microtome to obtain approximately 0,4 mm long fibre snippets. Cut only once in each yarn portion.

6.2.3.3 Other operation procedures are the same as stipulated in 6.2.1.3 and 6.2.1.4.

6.2.4 Woven fabrics

6.2.4.1 If the warp and weft yarn share the same composition, all 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 warp and weft yarns respectively and weigh them respectively (unravel at least the integral multiple of a complete pattern in the case of fabrics where there is a definite repetition in the pattern).

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

6.2.4.3 Other operation procedures are the as stipulated in 6.2.1.3 and 6.2.1.4.

6.2.5 Knitted fabrics

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

6.2.5.2 Other operation procedures are the same as stipulated in 6.2.1.3 and 6.2.1.4.

6.3 Coating the specimens

Use the sputter coater to apply a thin layer of gold to the specimens on specimen stub.

7 Test procedure

7.1 Test on each specimen stub

7.1.1 Place a stub with specimen into the test chamber of SEM. Firstly, view the selected stub at a lower magnification (for example, at ×10). Select from an area near the upper left edge of the stub on the monitor, set the magnification to ×1 000, scan the stub and observe the fibres, identify fibre types according to characteristics of fibre morphologies (see details in Annex B) of cashmere, sheep's wool, and other animal fibres.

7.1.2 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 operation until finished scanning the whole stub before continuing to analyse fibre snippets on another stub.

7.2 Qualitative analysis (purity analysis) and determination of fibre content

7.2.1 Examine 150 fibres on the first 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 five 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 fibre types are found and the content of each type is higher than 3 % by number, the fibre mixture is considered to be a blend. Perform a quantitative analysis according to 7.2.2.

7.2.2 Quantitative analysis of fibre blends.

If the sample is found to be a blend one, 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 20) on each of the remaining 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 to 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.

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 8.

8 Calculation of test result

8.1 Calculate percentage by mass of each component through Formula (1).

$$P_{\mathrm{i}} = \frac{N_{\mathrm{i}}\left(D_{\mathrm{i}}^2 + S_{\mathrm{i}}^2\right)\rho_i}{\sum\left[N_{\mathrm{i}}\left(D_{\mathrm{i}}^2 + S_{\mathrm{i}}^2\right)\rho_{\mathrm{i}}\right]} \times 100 \qquad (1)$$

where

P_{i} is the percentage by mass of some component, %;

N_{i} is the number of fibres counted for some component;

S_{i} is the standard deviation for mean diameter of some component, in micron (μm);

D_{i} is the mean diameter of some component, in micron (μm);

ρ_{i} is the density of some component, in gram per cubic centimetre (g/cm^3).

NOTE Density of various types of animal fibres is given in Annex C.

8.2 Percentage by mass of some fibre component in woven fabric samples may be calculated through Formula (2).

$$P_{\mathrm{i}} = \frac{P_{\mathrm{iT}} \times W_{\mathrm{T}} + P_{\mathrm{iW}} \times W_{\mathrm{W}}}{W_{\mathrm{T}} + W_{\mathrm{W}}} \times 100 \qquad (2)$$

where

P_{i} is the percentage by mass of some component in woven fabric sample, %;

P_{iT} is the percentage by mass of some component in warp yarns of woven fabric sample, %;

W_{T} is the mass of warp yarn in woven fabric sample;

P_{iW} is the percentage by mass of some component in weft yarns of woven fabric sample, %;

W_{w} is the mass of weft yarn in woven fabric sample.

Annex A
(informative)

Drawing of lot sample and laboratory sample

A.1 Loose fibre

50 % of the total number of packages should be sampled. Take out a bundle of fibres from at least three parts of each package. After blending them homogeneously, divide the sample into two equal portions, one portion randomly selected is retained and the other is rejected. After mixing the retained portion to ensure it is homogenized, it is divided again into two equal portions in the same way and one portion (selected at random) is rejected. Continue the subdivision procedure until about 20 g fibres remain as lot sample. Divide the 20 g of fibre sample into two portions, one portion is used as the laboratory sample and the other is retained as spare sample.

A.2 Silver

Take one sliver of 30 cm long from a ball top or a sliver can. Randomly take four of such slivers altogether. Strip from each of the four slivers in its longitude direction to form another sliver which is the laboratory sample. Keep the remaining portions as spare sample.

A.3 Yarn

Take 20 times of 20 cm long woollen yarn segment from each of five different cones or skeins (10 different cones or skeins for worsted yarn) to obtain 100 yarn segments for woollen and 200 yarn segments for worsted yarns. Cut in the middle of each portion, one portion is used as laboratory sample and the other is retained as spare sample.

A.4 Woven fabrics

Take three trapezoidal samples each measuring 5 cm × 10 cm (warp × weft) from places which are 10 cm from the edges of the fabric and mark its warp and weft directions respectively (cut at least the integral multiple of a complete pattern in the case of fabrics where there is a definite repetition of the pattern). Cut along weft direction from the middle of each fabric sample and divide it into two portions, one is used as laboratory sample and the other is retained as spare sample.

A.5 Knitted fabrics

Take three samples each measuring 5 cm × 10 cm (transverse × longitudinal). Avoid rib sections such as cuff or bottom parts. Cut each sample from the middle along longitudinal direction into two portions, one is used as the laboratory sample and the other is retained as spare sample.