INTERNATIONAL STANDARD

**ISO 17751-2**

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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*



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# Foreword

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

— The advantage of the 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 the fibre analyst.

—The SEM method shows opposite 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 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 the 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 the physical properties/attributes characterizing the fibre surface

EXAMPLE The fibre 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 a *lot sample* (2.8) according to requirements to prepare specimens

**2.10**
**test specimen**
portion taken from fibre snippets randomly cut from a *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 comparing them with known fibre surface morphologies for different types of animal fibres.

For each fibre type, the number and mean 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.

## 4 Apparatus, materials, and reagents

### 4.1 Apparatus

**4.1.1 Scanning electron microscope**, comprised of a 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 Materials

**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.

**4.2.8 Razor blade**.

### 4.3 Reagents

**4.3.1 Acetone (analytical grade)**

**4.3.2 Ethyl acetate (analytical grade)**.

## 5 Sample drawing

Draw the 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. The fibre snippets on the specimen stubs shall be sufficient to ensure that at least 1 000 fibres are examined.

### 6.2 Preparation method for test specimens of various types of samples

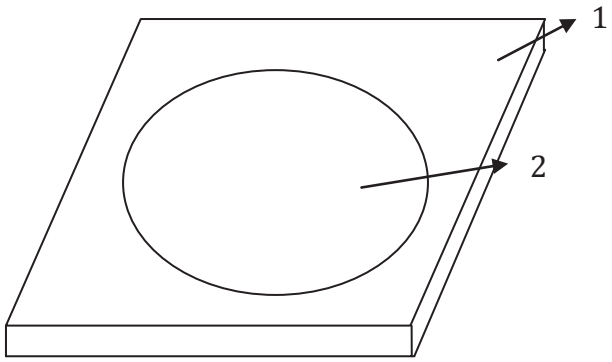
#### 6.2.1 Loose fibre

**6.2.1.1** 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 (4.2.6) from the top and bottom sides of the sample. Blend them homogeneously, and divide them 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 a microtome (4.2.1) 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 (4.2.2) and suspend them in 1 ml to 2 ml acetone (4.3.1) or ethyl acetate (4.3.2) by stirring the mixture with a stainless steel rod (4.2.3). Pour the suspension onto a glass plate (4.2.4) 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 (4.2.5) on the mounting stubs and use a razor blade (4.2.8) to trim the tape away from around the mounting stubs. After all the acetone (4.3.1) or ethyl acetate (4.3.2) in the fibre snippets suspension has evaporated, press the mounting stubs with the adhesive tape end onto the glass plate (4.2.4) at the positions shown in Figure 2. Transfer the uniformly mixed fibre snippets to the adhesive tape (4.2.5) on the specimen stub (4.2.7).

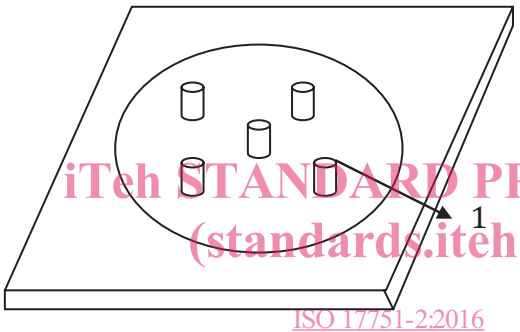



**Key**

1 glass plate

2 fibre snippets

**Figure 1 — Fibre suspension on glass plate**






**Key**

1 specimen stub

**Figure 2 — Positions of specimen stubs**

If the fibre snippets have aggregated after the evaporation of the acetone (4.3.1) or ethyl acetate (4.3.2), they shall be recollected by scraping them off the glass plate (4.2.4) with a razor blade (4.2.8) 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 an appropriate amount of the fibre bundle in the longitudinal 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 a microtome (4.2.1). Cut only once in each fibre bundle.

**6.2.2.3** Other operating procedures are the same as those stipulated in 6.2.1.3 and 6.2.1.4.

### 6.2.3 Yarn

**6.2.3.1** Divide the laboratory sample into three equal portions.

**6.2.3.2** Cut each portion in the middle with a microtome (4.2.1) to obtain approximately 0,4 mm long fibre snippets. Cut only once in each yarn portion.

**6.2.3.3** Other operating procedures are the same as those 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 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 and weigh them separately. (If the fabrics have a definite repetition in the pattern, unravel at least the integral multiple of a complete pattern.)

**6.2.4.2** Cut once from the parallel yarn portion in the middle with a microtome (4.2.1) to obtain approximately 0,4 mm long fibre snippets. Cut only once in each yarn segments.

**6.2.4.3** Other operating procedures are the same as those 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 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.

**6.2.5.2** Other operating procedures are the same as those stipulated in 6.2.1.3 and 6.2.1.4.



## 6.3 Coating the specimens



Use the sputter coater (4.1.2) to apply a thin layer of gold to the specimens on specimen stub (4.2.7).



# 7 Test procedure

## 7.1 Test on each specimen stub

**7.1.1** Place a stub with the specimen into the test chamber of the SEM. First, view the selected stub at a lower magnification (for example, at ×10). Then, selecting 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 the fibre types according to the characteristics of the 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 the vertical or horizontal direction. Repeat the above operation until finished, scanning the entire stub before continuing on 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 (4.2.7). 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, 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 1 030 fibres shall be identified for a sample and 100 measurements of fibre diameter made for each component. The mean fibre diameter of each component is calculated according to the 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 a vacuum condition and is not comparable to a 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 the mass fraction of each component using Formula (1).

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

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 for mean diameter of the component, in micrometres (μm);

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

$\rho_i$ is the density of the component, in grams per millilitre (g/ml).



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

**8.2** The mass fraction of a given fibre component in woven fabric samples may be calculated through Formula (2).

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

where

$w_i$ is the mass fraction of the component in the 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 yarn in the woven fabric sample;

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

$m_W$ is the mass of the weft yarn in the woven fabric sample.

# Annex A
(informative)

# Drawing of lot sample and laboratory sample

## A.1 Loose fibre

Fifty percent 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, divide it again into two equal portions in the same way. Reject one portion (selected at random).

Continue the subdivision procedure until about 20 g of fibres remain; this is the lot sample.

Divide the 20 g fibre lot sample into two portions — use one portion as the laboratory sample and retain the other as a spare sample.

## A.2 Sliver



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



## A.3 Yarn

Take twenty 20 cm long woollen yarn segments from each of five different cones or skeins to obtain 100 woollen yarn segments.

Take twenty 20 cm long worsted yarn segments from each of ten different cones or skeins to obtain 200 worsted yarn segments.

Cut the yarn bundle in the middle to get two portions — use one portion as a laboratory sample and retain the other as a 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. For each sample, 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 the weft direction from the middle of each fabric sample and divide it into two portions — use one as the laboratory sample and retain the other as a 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 the longitudinal direction into two portions — use one as the laboratory sample and retain the other as a spare sample.

# Annex B
(informative)

# Surface morphology of common animal fibres

## B.1 Cashmere from China

### B.1.1 Typical ring-shaped morphology

See Figures B.1 to B.10.

This cashmere has a high fibre diameter evenness in its axial direction and good lustre. The fibre scales are regular, most are ring-shaped and a few irregular ring-shaped; few variations can be seen. The scale envelops the fibre shaft flatly and evenly; scales are thin with smooth surfaces. The distance between adjacent scales is large. The mean scale height is less than 0,4 µm. The mean scale frequency is between 54 scales/mm and 64 scales/mm.



**Figure B.1 — Scales encircle fibre shaft flatly and regularly with regular patterns and smooth surface**

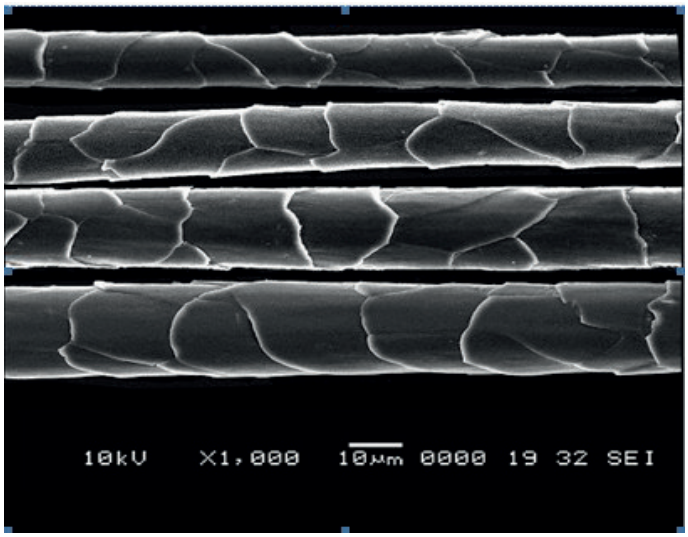



**Figure B.2 — Some scale patterns are slightly irregular**

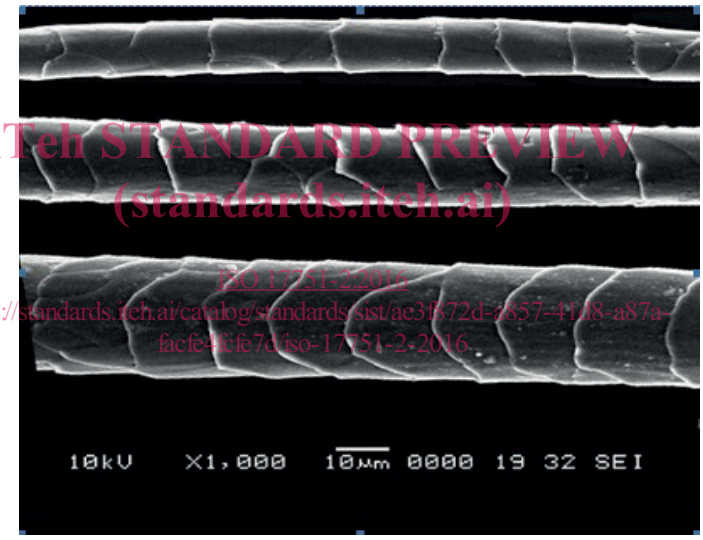





**Figure B.3 — Scale frequencies from low to high**