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

Part 1: Light microscopy method

Textiles — Analyse quantitative du cachemire, de la laine, d'autres fibres animales spéciales et leurs mélanges —
Partie 1: Méthode de microscopie optique

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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 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 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 C.

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

Part 1: Light 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 light microscopy (LM).

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 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 139, *Textiles — Standard atmospheres for conditioning and testing*

3 Terms and definitions

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

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

3.2
light microscope
optical instrument used to produce magnified images utilizing visible light source

Note 1 to entry: Types of microscopes suitable for fibre identification include projection microscopes and visual microscopic image analysers. Transmitted–light type microscopes with direct graduated scale equipped on optical lens are also applicable.

3.3
scale
cuticle covering the surface of animal fibres

3.4
scale frequency
number of *scales* (3.3) along the fibre axis per unit length

3.5
scale height
height of the cuticle at the *scale's* (3.3) distal edge

3.6
fibre surface morphology
sum of the physical properties/attributes characterizing the fibre surface

EXAMPLE The fibre surface morphology includes *scale frequency* (3.4), *scale height* (3.5), patterns of scale edge, scale surface smoothness, fibre evenness along its axis, transparency under *light microscope* (3.2), etc.

3.7
lot sample
portion representative of the same type and same lot of material drawn according to the requirements from which it is taken

3.8
laboratory sample
portion drawn from a *lot sample* (3.7) according to the requirements for preparing specimens

3.9
test specimen
portion taken from fibre snippets randomly cut from a *laboratory sample* (3.8) for measurement purposes

4 Principle

A longitudinal view image of fibre snippets representative of a test specimen is magnified to an appropriate scale/size under an optical microscope. All the 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 diameters of the 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, materials, and reagents

5.1 Apparatus

5.1.1 Projection microscope, comprised of a light source, a light condenser, a stage, an objective, an ocular, and a circular transparent viewing screen or non-transparent projection table with a graduated scale in millimetres. The objective and ocular shall be capable of providing at least a magnification of ×500 at the screen.

5.1.2 Visual microscopic image analyser, comprised of a microscope, a camera, a computer, a data acquisition card, exclusive analysing software, and a display. The objective and ocular of the microscope shall be capable of providing at least a magnification of ×500.

5.1.3 Transmitted-light type microscope, comprised of a light source, a light condenser, a stage, an objective, and an ocular with a graduated scale. The objective and ocular of this type of microscope shall be capable of providing a magnification of ×400 to ×500.

5.2 Materials

5.2.1 Microtome.

5.2.2 Scissors, tweezers, cleaning fabric, watch-glass, etc.

5.2.3 Slides and cover glasses.

5.2.4 Wedge scale, with divisions of ×500 magnification. A moveable linear rule-type scale finely graduated in millimetres may also be used.

5.3 Reagents

5.3.1 Liquid paraffin with a refractive index between 1,43 and 1,53.

6 Drawing of laboratory sample and conditioning

6.1 Drawing methods for lot samples and laboratory samples are given in Annex A.

6.2 The laboratory sample shall be conditioned for at least 4 h under the standard atmospheres stipulated in ISO 139.

7 Preparation of the test specimens

7.1 Number of test specimens

Prepare one or more slides so that at least 1 000 fibres shall be identified.

7.2 Preparation of the test specimens

7.2.1 Loose fibre

7.2.1.1 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 (5.2.2) from the top and bottom sides of the sample. Blend them homogeneously and divide them into three equal portions. Sort these drawn fibres into basically parallel fibre bundles.

7.2.1.2 Cut each fibre bundle in the middle with a microtome (5.2.1) to get approximately 0,6 mm long fibre snippets. Cut only once in each of the fibre bundles.

7.2.1.3 Place all the fibre snippets on the watch glass, drop an appropriate amount of liquid paraffin (5.3.1), stir with tweezers (5.2.2) to make the suspended snippet liquid distribute uniformly on the watch glass, then take an appropriate amount of this specimen blend and put it on the slide. Cover with a cover glass.

7.2.2 Sliver

7.2.2.1 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.

7.2.2.2 Cut in the middle of each fibre bundle to obtain approximately 0,6 mm long fibre snippets with a microtome (5.2.1). Cut only once in each fibre bundle.

7.2.2.3 Other operating procedures are the same as those stipulated in 7.2.1.3.

7.2.3 Yarn

7.2.3.1 Divide the laboratory sample into three equal portions.

7.2.3.2 Cut each portion in the middle with a microtome (5.2.1) to obtain approximately 0,6 mm long fibre snippets. Cut only once in each yarn portion.

7.2.3.3 Other operating procedures are the same as those stipulated in 7.2.1.3.

7.2.4 Woven fabrics

7.2.4.1 If the warp and weft yarn share the same composition, all the yarns 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.)

7.2.4.2 Cut from the parallel yarn portion in the middle with a microtome to obtain approximately 0,6 mm long fibre snippets. Cut only once in each yarn portion.

7.2.4.3 Other operating procedures are the same as those stipulated in 7.2.1.3.

7.2.5 Knitted fabrics

7.2.5.1 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 0,6 mm long fibre snippets. Cut only once in each yarn portion.

7.2.5.2 Other operating procedures are the same as those stipulated in 7.2.1.3.

If, prior to analysis, Soxhlet extraction in light petroleum (boiling point 40 °C to 60 °C) is carried out to remove excess surface greases or oils, it shall be reported.

7.3 Decolouring of the laboratory sample

If a decolouring process is carried out on those dark laboratory samples for which it is difficult to see the fibre morphology, prepare the test specimens according to the requirements in 7.2. The decolouring process application shall be reported.

The recommended decolouring methods are given in Annex B.

NOTE The decolouring process can lead to different fibre diameters measured from the decoloured fibres than from those diameters measured from the original fibres taken from fabric or yarns prior to decolouring.

8 Test procedure

8.1 Settings of magnification with micrometer scale

Put the micrometer with a 0,01 mm scale on the stage. The 20 scales from the micrometer (0,20 mm) projected on the screen shall be precisely magnified to 100 mm which means the magnification is ×500.

8.2 Fibre identification and fibre diameter measurement

8.2.1 Projection microscope with a graduated scale in millimetres on the screen (5.1.1).

8.2.1.1 The slide should be scanned in a raster pattern. This ensures that all parts of the slide are covered and avoids the possibility of any fibre being measured twice.

8.2.1.2 Observe and measure the diameters of the various types of fibres in the view. Measure the diameters of at least 100 fibres for cashmere and wool and at least 150 fibres for other speciality animal fibres. At the same time, identify the fibre types according to various fibre morphologies (reference details are given in Annex C). Record the number of different types of fibres, and identify more than 1 000 fibre snippets from each test specimen.

If the number of fibres identified reaches 1 000 while the measurement is still being carried out in the middle of the slide, keep moving and counting until the end of the slide. For fibre types in which only a minor proportion is blended into and the number of fibres measured fail to meet the requirement of number for fibre diameter measurement, measure all fibres of the type found in the specimen slide.

8.2.1.3 For those fibres observed with diameters exceeding 30 µm for cashmere, 35 µm for yak wool, 40 µm for camel, and 30 µm for Angora rabbit hair, record them as cashmere coarse hair, yak hair, camel coarse hair, and coarse rabbit hair respectively. Measure their fibre diameters and record the number of such fibres. If any of the above mentioned fibres accounts for less than 0,3 % of the total amount counted in the specimen, the component can be neglected.

8.2.1.4 If a measurement falls between two divisions, take the lower of the two values.

8.2.1.5 Calculate the mean fibre diameter and standard deviation for a given component according to Formulae (1) and (2), respectively.

$$\bar{d} = \frac{\sum (d \times F)}{\sum F} \tag{1}$$

$$S = \sqrt{\frac{\sum F(d - \bar{d})^2}{\sum F}} \tag{2}$$

where

$\bar{d}$ is the mean fibre diameter of the component, in micrometres (µm);

d is the group diameter, d = (recorded group value + 0,5) × 2, in micrometres (µm);

F is the number of fibres measured with the same diameter;

S is the standard deviation, in micrometres (µm).

8.2.2 Projection microscope used to measure the fibre diameter with a wedge scale or a transparent moveable linear-rule-type scale.

8.2.2.1 Measurement is made by moving the wedge scale (5.2.4) with its length at right angles to the fibre image until a division coincides with one edge of the focused fibre image. The width of the fibre image is read off on the other edge of the wedge scale. When measuring an image whose edges are not in focus together, adjust the focusing so that one edge is in focus when a fine line appears and the other edge shows a white line. Measure the width from the edge that is in focus to the inside of the white line.

8.2.2.2 If the width of a fibre image coincides with wedge scale division and lies exactly on a millimetre division of N, the width of the measured fibre image may be assigned to either data group N-1 or N+1 depending on actual conditions. If such cases reoccur, alternately assign them to data group N-1 and to data group N+1.

8.2.2.3 Other operating procedures are the same as those stipulated in 8.2.1.1 to 8.2.1.3.

8.2.2.4 The mean fibre diameter and standard deviation of a given component is calculated using Formulae (3) and (4), respectively.

$$\bar{d} = \frac{\sum(A \times F)}{\sum F} \tag{3}$$

$$S = \sqrt{\frac{\sum F\left(A - \bar{d}\right)^2}{\sum F}} \tag{4}$$

where

$\bar{d}$ is the mean fibre diameter of the component, in micrometres (μm);

A is the median, in micrometres (μm);

F is the number of fibres measured;

S is the standard deviation, in micrometres (μm).

8.2.2.5 Fibre diameter measurement operation with rule-type scale and calculation are the same as stipulated in 8.2.1.

8.2.3 Visual microscopic image analyser (5.1.2).

8.2.3.1 Observe various type of fibres in the screen view. Measure the fibre diameter when edges of fibre in focus shows clear fine lines. Move the cursor to one side of the focused fibre, click on the left mouse button, then move the cursor to the other side of the focused fibre. Click on the left mouse button again, the fibre diameter value will be automatically recorded after measurement. Test result will be automatically calculated and recorded in the report sheet.

8.2.3.2 Other procedures are the same as those stipulated in 8.2.1.1 to 8.2.1.3.

8.2.4 Transmitted-light type microscope (5.1.3).

Proceed as described in 8.2.1, but with measuring using the graduated scale of the ocular.

9 Calculation of test result

9.1 Calculate the mass fraction of each component using Formula (5).

$$w_i = \frac{N_i\left(D_i^2 + S_i^2\right)\rho_i}{\sum\left[N_i\left(D_i^2 + S_i^2\right)\rho_i\right]} \times 100 \tag{5}$$

where

w_i is the mass fraction of the component, in %;

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/ml).

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

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 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 rabbit hair is the sum of percentages of both fine and coarse rabbit hairs).

Test result of fibre content is rounded to one decimal.

9.2 Calculate the mass fraction of a fibre component in woven fabric samples through Formula (6).

$$w_i = \frac{w_{iT} \times m_T + w_{iW} \times m_W}{m_T + m_W} \times 100 \quad (6)$$

where

w_i is the mass fraction of the component in the woven fabric sample, in %;
w_{iT} is the mass fraction of the component in the warp yarns of the woven fabric sample, in %;
m_T is the mass of the warp yarns in the woven fabric sample;
w_{iW} is the mass fraction of the component in the weft yarns of the woven fabric sample, in %;
m_W is the mass of the weft yarns in the woven fabric sample.