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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

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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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

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 superb fine fibre with low production and high price, 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 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, but some subtle surface structures are not able to be clearly displayed. A decoloration process needs to be carried out on dark samples for testing while improper decoloration process will affect the judgment of fibre analyst. The Scanning Electron Microscopy (SEM) method shows opposite 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, full 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 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 137, *Wool — Determination of fibre diameter — Projection microscope method*

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 microscope suitable for fibre identification include projection microscope and visual microscopic image analyser. Transmitted–light type with direct graduated scale equipped on optical lens is also applicable.

3.3
scale
cuticle covering the surface of animal fibres

3.4
scale frequency
number of *scales* (3.3) along 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 physical properties/attributes characterizing the fibre surface

EXAMPLE 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 *lot sample* (3.7) according to the requirements to prepare specimens

3.9
test specimen
portion taken from fibre snippets randomly cut from *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 optical microscope, and 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 for each fibre type.

5 Apparatus, accessories, and reagents

5.1 Apparatus

5.1.1 Projection microscope

The projection microscope proper shall comprise 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

The proper visual microscopic image analyser shall comprise 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

The transmitted-light type microscope shall comprise 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 Accessories

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

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

6 Drawing of laboratory sample and conditioning

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

6.2 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 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.

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

7.2.1.3 Put all fibre snippets on the watch glass, drop appropriate amount of liquid paraffin, stir with tweezers to make the suspended snippet liquid distribute uniformly on the watch glass, then take appropriate amount of specimen blend and put on the slide. Cover with a cover glass.

7.2.2 Sliver

7.2.2.1 Cut the laboratory sliver sample into three sections and take out appropriate amount of 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 microtome. Cut only once in each fibre bundle.

7.2.2.3 Other operation procedures are the same as stipulated in 7.2.1.3.

7.2.3 Yarn

7.2.3.1 Divide laboratory sample into three equal portions.

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

7.2.3.3 Other operation procedures are the same as 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 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 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).

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

7.2.4.3 Other operation procedures are the same as stipulated in 7.2.1.3.

7.2.5 Knitted fabrics

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

7.2.5.2 Other operation procedures are the same as stipulated in 7.2.1.3.

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

7.3 Decolouring of laboratory sample

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

Recommended decolouring method is given in Annex B.

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

8 Test procedure

8.1 Settings of magnification with micrometer scale

Put micrometer with 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 graduated scale in millimetre on the screen (5.1.1).

8.2.1.1 The slide should be scanned in a raster pattern as described in ISO 137.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 diameter of various type of fibres into the view. Measure the diameter of at least 100 fibres for cashmere and wool and at least 150 fibres for other speciality animal fibres. At the same time, identify fibre types according to various fibre morphologies (reference details are given in Annex C), record the number of different types of fibres respectively, 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 just carried out in the middle of the slide, keep moving and counting till the end of the slide. For fibre types which only a minor proportion is blended into and the number of fibres measured failed to meet the requirement of number for fibre diameter measurement, measure all fibres of such type found in the specimen slide.

Conditions of fibres excluded from measurement during diameter measurement operation process are subject to stipulation in ISO 137.

8.2.1.3 For those fibres observed with diameter exceeding 30 μm for cashmere, 35 μm for yak wool, 40 μm for camel, and 30 μm for Angora rabbit hair, record as cashmere coarse hair, yak hair, camel coarse hair, and coarse rabbit hair respectively. Measure their fibre diameter and record number of such fibres. If any of the above mentioned fibres accounts to 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 some 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 some component, in micron (μm);

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

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

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

8.2.2 Projection microscope which fibre diameter is measured with wedge scale or a transparent moveable linear rule-type scale.

8.2.2.1 Measurement is made by moving the wedge scale 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 In the event when the width of a fibre image coincides with wedge scale division and lie exactly on a millimetre division of N, the width of the measured fibre image may either be assigned to data group N-1 or N+1 depending on actual conditions. If such cases happen again, alternately assign it to data group N-1 and N+1.

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

8.2.2.4 Mean fibre diameter and standard deviation of some component is calculated through 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 some component, in micron (µm);

A is the median, in micron (µm);

F is the number of fibres measured;

S is the standard deviation, in micron (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 into the screen view. Measurement of fibre diameter when edges of fibre in focus shows clear fine lines. Move the cursor to one side of the focused fibre, click 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 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 percentage by mass of each component through Formula (5).

