SLOVENSKI STANDARD
SIST EN ISO 17751-2:2016

01-junij-2016

Tekstilije - Kvantitativna analiza kašmirskih, volnenih, drugih specialnih živalskih vlaken in njihovih mešanic - 2. del: Metoda štetja z elektronskim mikroskopom (ISO 17751-2:2016)

Textiles - Quantitative analysis of cashmere, wool, other specialty animal fibers and their blends - Part 2: Scanning Electron Microscopy method (ISO 17751-2:2016)

Textilien - Quantitative Analyse von Kaschmir, Wolle, anderen speziellen tierischen Fasern und deren Mischungen - Teil 2: Rasterelektronenmikroskopie-Verfahren (ISO 17751-2:2016)

Textiles - Analyse quantitative du cachemire, de la laine, d'autres fibres animales spéciales et leurs mélanges - Partie 2: Méthode par microscopie électronique à balayage (ISO 17751-2:2016)

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59.060.10	Naravna vlakna	Natural fibres

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English Version

Textiles - Quantitative analysis of cashmere, wool, other specialty animal fibers and their blends - Part 2: Scanning Electron Microscopy method (ISO 17751-2:2016)

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European foreword

This document (EN ISO 17751-2:2016) has been prepared by Technical Committee ISO/TC 38 "Textiles" in collaboration with Technical Committee CEN/TC 248 “Textiles and textile products” the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2016, and conflicting national standards shall be withdrawn at the latest by October 2016.

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INTERNATIONAL STANDARD

ISO 17751-2

First edition
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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 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 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).