SLOVENSKI STANDARD
kSIST-TP FprCEN ISO/TR 11827:2016

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Tekstilije - Preskušanje sestave - Identifikacija vlaken (ISO/TR 11827:2012)

Textiles - Composition testing - Identification of fibres (ISO/TR 11827:2012)

Textiles - Essai de composition - Identification des fibres (ISO/TR 11827:2012)

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TECHNICAL REPORT

ISO/TR 11827

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Textiles — Composition testing — Identification of fibres

Textiles — Essai de composition — Identification des fibres

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard (“state of the art”, for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TR 11827 was prepared by Technical Committee ISO/TC 38, *Textiles.*

Introduction

The correct identification of fibres in textiles and the accurate determination of the composition of each fibre present is a legal requirement in many countries throughout the world for imported textile goods and at the point of sale to the public. Fibre identification can be carried out by a number of different techniques, e.g. microscopy, solubility, spectroscopy, melting point, pyrolysis, density, refractive index, etc.

Textiles — Composition testing — Identification of fibres

IMPORTANT — The electronic file of this document contains colours which are considered to be useful for the correct understanding of the document. Users should therefore consider printing this document using a colour printer.

1 Scope

This Technical Report describes procedures for the identification of natural and man-made fibres, and may be used, when necessary, to coordinate with methods for the quantitative analysis of fibre blends.

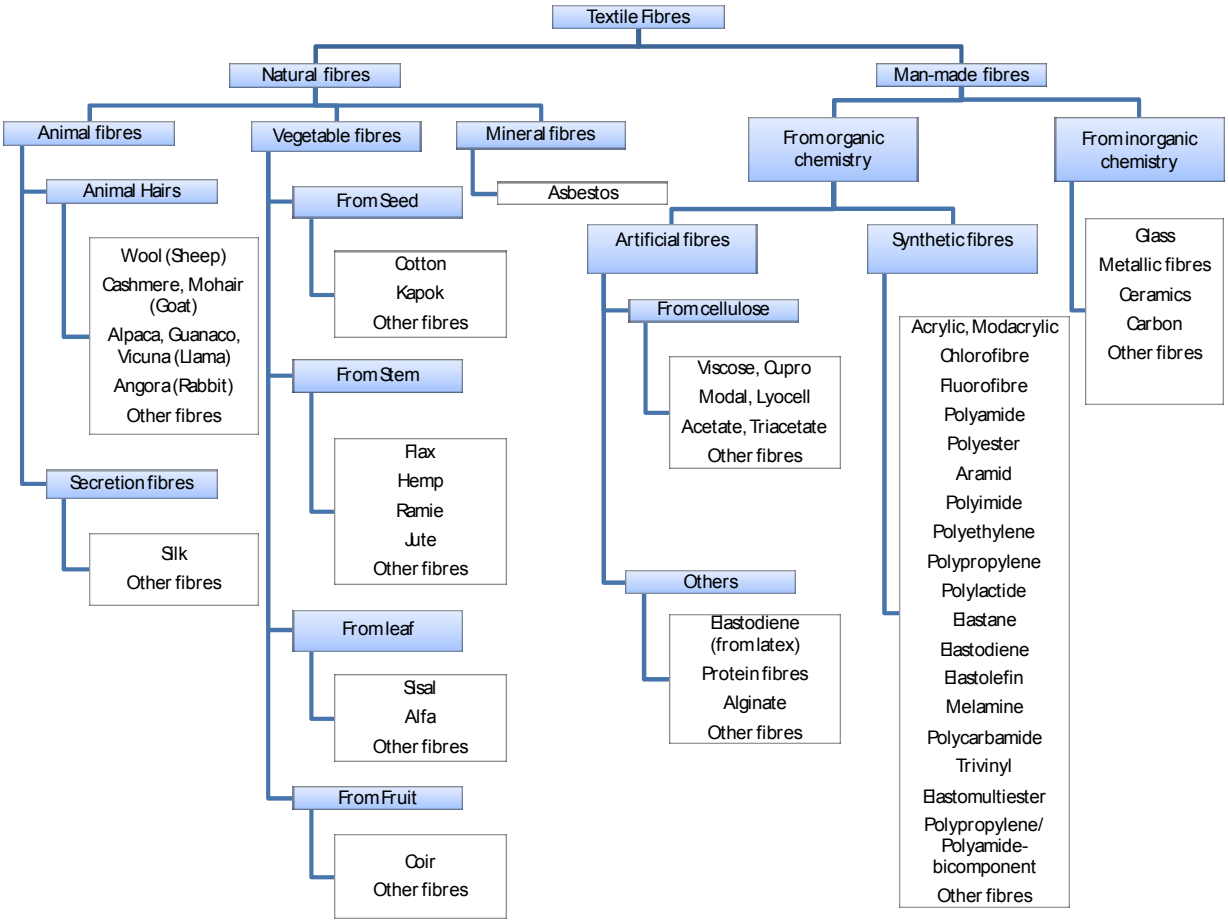

Figure 1 — Classification of the textile fibres in relation to their origin

2 Safety note

This Technical Report calls for the use of substances/procedures that may be injurious to the health/ environment if appropriate conditions are not observed. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety/environment at any stage.

3 Normative references

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

ISO 1833-4, *Textiles — Quantitative chemical analysis — Part 4: Mixtures of certain protein and certain other fibres (method using hypochlorite)*

ISO 2076, *Textiles — Man-made fibres — Generic names*

ISO 6938, *Textiles — Natural fibres — Generic names and definitions*

4 Terms and definitions

For the purposes of this document, the following terms and definitions given in ISO 2076 and ISO 6938 and the following apply.

4.1
natural fibre
fibre which occurs in nature: it can be categorized according to its origin into animal, vegetable and mineral fibre

4.2
man-made fibre
manufactured fibre
fibre obtained by a manufacturing process

4.2.1
artificial fibre
manufactured fibre made by transformation of natural polymers (macromolecular material existing in nature)

4.2.2
synthetic fibre
manufactured fibre made from synthetic polymers (macromolecular material which has been chemically synthesised)

4.2.3
bicomponent fibre
fibre composed of two fibres forming polymer components, which are chemically or physically different or both

5 Principle

Objective: identify the fibres

Means: based on fibre properties (single or combination)

Properties for example:

- Morphology
- Solubility
- Light absorption or transmission by IR
- Burning behaviour
- Thermal behaviour

- Colouration
- Optical behaviour
- Elemental composition

6 Apparatus and preparation of solutions

6.1 Apparatus

6.1.1 **Light Microscope,** using transmitted light

6.1.2 Scanning Electron Microscope

6.1.3 Bunsen Burner or other flame source

6.1.4 Infrared Spectrometer

6.1.4.1 Attenuated Total Reflection (ATR) spectroscopy device

6.1.4.2 Fourier Transform Infrared (FT-IR) spectrometer

6.1.5 Melting Point device (heated block)

6.1.6 Differential Scanning Calorimeter (DSC)

6.1.7 Thermal Gravimetric Analysis (TGA) device (thermobalance)

6.1.8 Gravimetric device (density gradient column)

6.1.9 Energy Dispersive X-ray (EDX) device

6.2 Preparation of solutions

Use only reagents of recognized analytical grade.

6.2.1 Sodium hydroxide and calcium oxide

Prepare a mixture of sodium hydroxide and calcium oxide (mass ratio of 1:1,4)

6.2.2 Iodine/potassium iodine solution

Dissolve 20 g of potassium iodide in 20 ml to 50 ml of distilled water. In this solution dissolve 2,5 g of iodine and dilute to 100 ml

6.2.3 Zinc chloride/iodine solution

Dissolve 66 g of zinc chloride, anhydrous, and 6 g of potassium iodide in 34 ml of water.

Add a small amount of iodine crystal so that the solution is saturated.

6.2.4 Chlorine bleaching solution

Prepare the solution according to ISO 1833-4.

6.2.5 Zinc chloride/formic acid solution

Dissolve 100 g of zinc chloride, anhydrous in 100 ml of water.

Set the density of this solution to 1,566 g/ml.

Add 6 ml of concentrated formic acid to 100 ml of this solution.

6.2.6 Sodium carbonate 0,25 % solution

Add 0,25 g of sodium carbonate to 100 ml of distilled water and dissolve.

6.2.7 Sodium hydroxide 5 % solution

Dissolve 5 g of sodium hydroxide in distilled water and dilute to 100 ml.

6.2.8 Sulphuric acid 75 % solution

Add carefully, while cooling, 700 ml of concentrated sulphuric acid (ρ 1,84 g/ml) to 350 ml of distilled water.

After the solution has cooled to room temperature, dilute to 1 l with water.

6.2.9 Chloroform/trichloroacetic acid solution

Dissolve 50 g of trichloroacetic acid in 50 g of chloroform.

6.2.10 Ethanol / potassium hydroxide solution

Dissolve 15 g of potassium hydroxide in 100 ml of ethanol.

7 Techniques

7.1 Microscopy

7.1.1 Light Microscopy

Examine the longitudinal view and/or the cross section of a fibre sample under a light microscope (6.1.1) using transmitted light and magnification.

Compare with photomicrographs in Annex B.

7.1.2 Scanning Electron Microscopy

Examine the longitudinal view and/or the cross section of the surface of a fibre sample under a scanning electron microscope (6.1.2) using magnification.

Compare with photomicrographs in Annex C.

7.1.3 Refractive Index

7.1.3.1 General

Refractive index governs the visibility of all colourless and transparent objects.

When a fibre is examined in air (n=1,0), the relatively large difference in refractive index between the fibre and air causes about 5 % of the incident light to be reflected and the transmitted light to be markedly refracted. These effects produce heavy shadows that obscure fine details of the fibre structure and can introduce misleading identification. To reduce the degree of contrast in the shadow regions the fibres are mounted in a medium of suitable refractive index for microscopic evaluation.

7.1.3.2 Mounting media

If fibres are mounted in a medium of similar refractive index, surface characteristics are practically invisible but internal structure and the presence of voids, or inclusions such as pigmentation, are clearly revealed. When it is desired to examine the surface details of a fibre a mounting medium of significantly different refractive index should be selected, preferably one with a much higher refractive index than that of the fibre, e.g. 1-bromonapthalene or di-iodo-methane.

Mountants should be relatively stable, and should not be volatile or react with the polymer fibre. The most commonly used mountant is liquid paraffin which gives an image of satisfactory contrast for all fibres except for cellulose diacetate and triacetate, for which n-decane is recommended.

It is recommended that all fibres be examined as soon after mounting as possible. Some fibres if left for a period may be penetrated by the mountant, or they may swell which makes fibre diameter measurements incorrect, or the mountant may evaporate.

7.1.3.3 Factors governing refractive indices

Factors governing the refractive index of fibres are the chemical nature of the molecules, the physical arrangement of these molecules, the wavelength of incident light, moisture content, and other substances that may be present in the fibre. In order to make accurate determinations it is necessary to use plane-polarised light under conditions of controlled temperature and relative humidity.

Birefringent substances exhibit different indices of refraction for a given wavelength depending on the direction of light passing through them, as well as upon its direction of transmission. For positive birefringent fibres the maximum and minimum refractive index corresponds to the long axis of the fibres and at right angles to the axis respectively. For negative birefringent fibres the reverse occurs.

7.1.3.4 Behaviour under polarised light

Determination of the behaviour under polarised light of a fibre can be carried out by mounting the fibre in a mountant of known refractive index (Table 2), then viewing under polarised light such that the microscope provides light polarised in the 6-12 o'clock direction.

Align the fibre in the direction of the light and set the microscope to provide axial illumination. Focussing carefully on the outlines of the fibre adjust the focus to just above the fibre. For cylindrical fibres, if the refractive index is higher than that of the mountant the fibre will act like a lens and a bright line of light will move into the middle of the fibre as the focus is raised. If the refractive index is lower that that of the mountant the light will flare out as the focus is raised and the middle of the fibre will become darker.

The test works best on round fibres, for flat ribbons it may be easier to see movement of a bright line at the outlines of the fibre.

Rotating the specimen 45° and setting the microscope to provide cross polars allows birefringence to be seen. Record if the fibre appears very bright (strong birefringence), dim (weak birefringence), or dark (no birefringence).

Repeat the test using different mountants (see Table 2). As the refractive index of the liquid approaches that of the fibre the fibre becomes less distinct until almost invisible. From the table match the liquid to the fibre for identification. This technique is particularly useful for the identification of polyester.

Compare the observations made with the Table 1 to identify possible fibres.