
**Textiles — Qualitative and
quantitative analysis of some cellulose
fibres (lyocell, cupro) and their
blends —**

Part 1:

**Fibre identification using scanning
electron microscopy and spectral
analysis methods**

*Textiles — Analyses qualitative et quantitative de certaines fibres
cellulosiques (lyocell, cupro) et leurs mélanges —*

*Partie 1: Identification des fibres par des méthodes de microscopie
électronique à balayage et d'analyse spectrale*



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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 38, *Textiles*.

A list of all parts in the ISO 21915 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The qualitative and quantitative determination of fibres is important for the distribution of textile products. In many countries, it is legally obligatory to attach information on the type of fibres used and their composition to textile products.

Therefore, the standards for the fibre identification and composition test methods have been developed on the fibres or blends of fibres as possible.

Cupro and lyocell described in this document are regenerated cellulose fibres and can be said to be materials that contribute to a sustainable society in that raw materials are not derived from petroleum.

There is difficulty to identify if the fibre is cupro or lyocell. Because the characteristics of appearance, chemical resistance, infrared spectroscopy (IR) spectrum, etc. are almost the same, the qualitative property according to ISO/TR 11827 and the quantification by the ISO 1833 series cannot be performed in some cases. The identification methods between cupro and lyocell are specified in this document with the new technical aspects.

ISO 21915 is composed of three parts. ISO 21915-1 specifies the identification method of cupro and lyocell by scanning electron microscope and infrared spectrum analysis. Those may be the time-consuming methods to use the composition analysis. ISO 21915-2 and ISO 21915-3 specify the methods for the composition analysis. The method to be used is determined by the instrument availability and experience.

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Textiles — Qualitative and quantitative analysis of some cellulose fibres (lyocell, cupro) and their blends —

Part 1: Fibre identification using scanning electron microscopy and spectral analysis methods

1 Scope

This document specifies the qualitative analysis for cupro and lyocell using the two methods separately

- scanning electron microscope (SEM) method based on the application of ISO 20705, and
- spectral analysis method.

These testing methods are applied only for cupro and lyocell, or those blends. If other fibres are present, those are identified using the test method of ISO/TR 11827 and removed using the relevant part of the ISO 1833 series.

This method is not applicable for the fibre surface that is damaged during the process (e.g. chemically or physically).

2 Normative references

ISO 21915-1:2020

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The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 (all parts), *Textiles — Quantitative chemical analysis*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 20705, *Textiles — Quantitative microscopical analysis — General principles of testing*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

cupro

cellulose fibre obtained by the cuprammonium process

[SOURCE: ISO 2076:2013, 4.1]

3.2

lyocell

cellulose fibre obtained by an *organic solvent* (3.3) *spinning process* (3.4) as defined in ISO 2076

[SOURCE: ISO 2076:2013, 4.2]

3.3

organic solvent

mixture of organic chemicals and water

3.4

solvent spinning

dissolving and spinning without the formation of a derivative

3.5

scanning electron microscope

SEM

electron-optical instrument that examines and analyses the physical information (such as secondary electron, backscattered electron, absorbed electron and X-ray radiation) obtained by generating electron beams and scanning the surface of the specimen in order to determine the structure composition and topography of the sample

3.6

calibration model

result of calculation by using the partial least squares (PLS) regression between the IR absorption data and *dummy variables* (3.7)

3.7

dummy variable

arbitrarily assigned number for *cupro* (3.1) and *lyocell* (3.2) such as 1 for cupro and 0 for lyocell to obtain the *calibration model* (3.6) by using multivariate analysis

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4 Principle

4.1 SEM method

Observe the fibre specimen by using SEM with specific conditions, as described in ISO 20705 (i.e. longitudinal view on SEM), and then find the difference in the shape of the surface appearance. Identify the fibre as cupro or lyocell from the deference of the surface appearance of cupro and lyocell.

4.2 Spectral analysis method

Prepare specimen of cupro and lyocell for creation of the calibration model. Assign the dummy variable for cupro: 1 and lyocell: 0 (these dummy variables are named reference values in [Annex D](#)). Measure the infrared (IR) spectrum at designated condition for the calibration specimens.

Obtain the calibration model by using the software for multivariate analysis to calculate the partial least squares regression (PLS) by using the obtained IR data and the dummy variables. Then, prepare the testing specimen as same as the calibration specimen. Measure IR absorption and obtain IR data. Input the data into the calibration model and obtain the result and round off the data to 1 or 0 and judge if cupro (1) or lyocell (0).

5 Reagents

5.1 SEM method

5.1.1 Methanol, with analytical grade.

5.1.2 Nonionic surfactant.

5.1.3 Sodium carbonate, with analytical grade.

5.1.4 Water, grade 3 water as specified in ISO 3696.

6 Apparatus

6.1 SEM method

6.1.1 SEM, with the specification described below and in accordance with ISO 20705.

The recommended condition to obtain clear appearance of wrinkle on cupro is as follows:

- Accelerating voltage: 10 KV
- Working distance: 10 mm
- Spot size: 35
- Beam current: 30 pA to 100 pA
- Pressure in specimen chamber: 10 Pa to 8 Pa or less
- Resolution of secondary electron image: 20 nm or more
- Magnification: $\times 4\,000$ to $\times 5\,000$

The spot size and beam current setting could be differed among the manufacturers of SEM. It is necessary to find a condition to observe the wrinkle of cupro as shown in [Annex A](#).

6.1.2 Ventilated oven, for drying specimens at $(105 \pm 3) ^\circ\text{C}$.

6.1.3 Microtome, in accordance with ISO 20705.

6.1.4 Razor blade.

6.1.5 Specimen stage (stub), a holder with a diameter of 13 mm made of aluminium or copper.

6.1.6 Double-sided tape.

6.1.7 Glass plate, measuring approximately 150 mm \times 150 mm.

6.1.8 Test tube.

6.1.9 Stainless steel rod.

6.1.10 Sputtering device, using gold and other metals evaporation.

6.2 Spectral analysis method

6.2.1 Infrared spectroscopy (IR) instrument, capable of performing infrared spectrometry.

6.2.2 Software for multivariate calculation, with the following features:

- capable of calculate PLS;
- capable of cross validation of calibration model;
- capable of calculate standard error of calibration (SEC) and its R^2 and standard error of cross validation (SECV) and its R^2 .

7 Procedure

7.1 SEM method

7.1.1 Prior identification

Fibres present in the sample are identified according to ISO/TR 11827, and other fibres shall be removed using the relevant parts of the ISO 1833 series.

7.1.2 Pre-treatment of specimens

7.1.2.1 Prepare approximately 10 cm × 10 cm of fabric or 1 g of fibre or yarn.

7.1.2.2 Pour 100 ml of water (5.1.4) into a beaker and add 0.3 g of nonionic surfactant (5.1.2) and 1 g of sodium carbonate (5.1.3), then boil the solution.

7.1.2.3 Put the specimen into the solution 7.1.2.2 in boiling state and keep it for (30 ± 5) min.

7.1.2.4 Wash the specimen by water and dry in the oven (6.1.2) for 60 min at (105 ± 3) °C.

7.1.3 Cutting

Cut fibres with 0,6 mm ± 0,2 mm in length by using microtome (6.1.3) and razor blade (6.1.4), to prevent aggregation and minimization as uneven dispersion caused by small and crimped fibres.

7.1.4 Preparation of specimen

7.1.4.1 Collect and put the cut specimens into test tube (6.1.8).

7.1.4.2 Put the cut specimens into methanol (5.1.1) of 15 ml and stir with a stainless-steel rod (6.1.9) to disperse and suspend the fibre piece.

7.1.4.3 Pour the cut fibre dispersed and suspended liquid onto glass plate before the fine cut fibre pieces settled.

7.1.4.4 After the reagent evaporated completely, let a round spot with 100 mm in diameter developed on the glass plate (6.1.7), in which so as not to overlap cut fibres and so as to disperse uniformly.

7.1.4.5 Press the specimen stage (stub) (6.1.5) with the double-sided tape (6.1.6) against the fibres.

7.1.4.6 Peel off the specimen stage (stub) (6.1.5) carefully.

7.1.4.7 If needed, apply a gold deposit with 600 Å to 1 000 Å in thickness by the sputtering device.

7.1.4.8 If the fibre fragments condense after evaporation of the reagent, collect the fibre fragments again and repeat procedures 7.1.4.1 to 7.1.4.4.

NOTE 1 Vacuum evaporator can be used instead of thin film sputtering device.

NOTE 2 The thickness of gold deposit is determined by the setting of the sputtering device.

7.1.5 Observation of specimen

7.1.5.1 Observe a specimen on SEM as described in ISO 20705 (6.1.1).

7.1.5.2 Put a specimen stage (stub) (6.1.5) in electronic microscope (6.1.1).

7.1.5.3 Set a working distance between the specimen and the objective lens at about 10 mm.

7.1.5.4 With setting magnification at $\times 10$ or more, bring the upper left end part of the stub on the monitor screen.

7.1.5.5 Set magnification at about $\times 4\,000$ and focus on an image.

7.1.5.6 Scan the stub in a vertical or lateral direction.

7.1.5.7 Observe each fibre on the monitor during scanning. If the thin crimp is observed on the surface of fibre as shown in Figure 1, the fibre is judged as cupro.



Figure 1 — Cupro fibre

If a smooth surface is observed as shown in Figure 2, the fibre is judged as lyocell.