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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

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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Phone: +41 22 749 01 11
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Foreword

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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.

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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

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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

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]

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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

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.

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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: ×4 000 to ×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 ± 3) °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 × 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.

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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.

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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 spattering 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 ×10 or more, bring the upper left end part of the stub on the monitor screen.

7.1.5.5 Set magnification at about ×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.

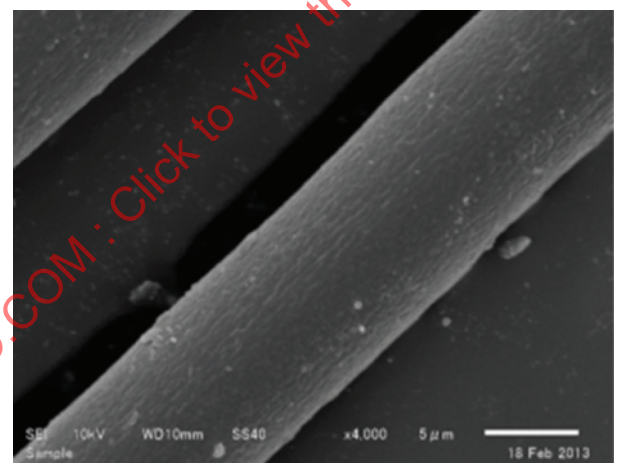

Figure 1 — Cupro fibre

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

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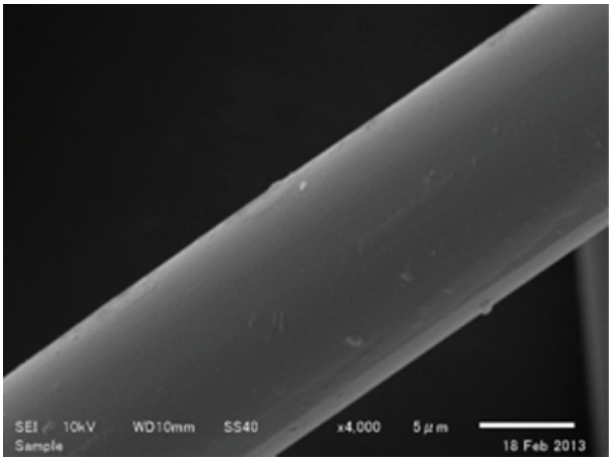

Figure 2 — Lyocell fibre

7.1.6 Qualitative analysis

7.1.6.1 Examine at least 150 fibres in total.

7.1.6.2 If only one fibre type is identified then report the cut specimen as "pure", or either cupro or lyocell.

7.1.6.3 If there is a doubt to judge as pure, observe another 150 fibres as a second stub.

The results of interlaboratory test are shown in Annex B.

This is a qualitative analysis however, if the number of specimens observed is increased, there is a possibility to perform quantitative analysis by SEM method which is described in Annex C.

7.2 Spectral analysis (IR) method

7.2.1 Development of calibration model

7.2.1.1 Preparation of calibration fibres

Prepare cupro samples and lyocell samples separately, at least 10 samples each.

7.2.1.2 Measurement of IR absorption

7.2.1.2.1 Take 3 yarns from one sample and take a single fibre from each yarn and obtain total 3 single fibre specimens.

7.2.1.2.2 Place the yarn specimen on diamond plate and press the fibres by using spatula to make flat.

7.2.1.2.3 Measure the infrared absorption by IR instrument (6.2.1) for the fibre at the following condition.

— Wave number range: 700 cm^{-1} to 4 000 cm^{-1}

— Wave number interval: 1,0 cm^{-1}

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7.2.1.2.4 Measure the IR absorption for all fibre specimens, 3 fibre specimens for 10 yarn samples, total 30 measurements for cupro and lyocell respectively according to the above condition and record the data.

7.2.1.2.5 Check the measured data. If there is obviously different data, delete the data and remeasure.

7.2.1.3 Development of the calibration model

7.2.1.3.1 Assign the dummy variables 1 for cupro and 0 for lyocell as the objective variables.

7.2.1.3.2 Calculate the partial least squares (PLS) regression between the IR absorption data and the dummy variables by using the software (6.2.2) as the multivariate analysis.

7.2.1.3.3 Obtain the calibration model as the result of the calculation of PLS.

7.2.1.4 Optimization of the calibration model

7.2.1.4.1 Optimize the obtained calibration model by using the software (6.2.2) of the multivariate analysis according to software instruction.

7.2.1.4.2 Set up the calibration model by choosing the number of factors using in the calibration model and calculate the standard error of calibration (SEC) and the coefficient of determination (R^2) of the calibration model as shown in Annex D.

7.2.1.4.3 Compare the SEC and R^2 value with the desired values as accuracies, which were 0,5 or less for SEC and 0,7 or higher for R^2 in this document.

7.2.1.4.4 If the accuracies, SEC and R^2 are not met the desired values, take the procedure 7.2.1.4.2 and 7.2.1.4.3 again. If the accuracies are met the desired value, proceed to 7.2.1.4.5.

7.2.1.4.5 Take the cross validation process by using the software (6.2.2) of the multivariate analysis according to the software instruction to evaluate stability.

7.2.1.4.6 Calculate the standard error of the cross validation (SECV) and the coefficient of determination (R^2) of the cross validation as shown in Annex D.

7.2.1.4.7 Compare the SECV and R^2 value of the cross validation with the desired values, which were 0,5 or less for SECV and 0,7 or higher for R^2 of the cross validation in this document.

7.2.1.4.8 If the accuracies, SECV and R^2 of the cross validation are not met the desired values, take the procedure from 7.2.1.4.2 and 7.2.1.4.7 again. If the accuracies and the stability are met the desired value, proceed to 7.2.2.

7.2.2 Measurement of test sample

7.2.2.1 Take 3 yarns from the sample and take a single fibre from each yarn and obtain total 3 single fibre specimens.

7.2.2.2 Measure the IR absorption as described in 7.2.1.2 and record the data.

7.2.3 Calculation of the predicted value and judgment

7.2.3.1 Input the IR data into the calibration model obtained in 7.2.1.

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7.2.3.2 Obtain the predicted value and round off the values to 1 or 0.

7.2.3.3 Judge if the fibre is cupro as the predicted value is 1 or the fibre is lyocell as the predicted value is 0.

7.2.3.4 Record the judged result in the test report.

The results of interlaboratory test are shown in Annex E.

8 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 21915-1:2020;

b) details of the sample fibres to be tested;

c) testing method used;

d) details of the testing results;

e) details of any deviation from the specified procedure;

f) any unusual features observed;

g) the date of the test.

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Annex A
(informative)

Example of the observation condition by SEM

A.1 SEM used

Type of SEM that detects secondary electrons.

A.2 Observation conditions

The parameters of SEM are set as the following.

— Accelerating voltage: 10 KV

— Working distance: 10 mm

— Spot size/Beam current: 20/6 pA, 35/36 pA, and 43/100 pA

— Pressure in specimen chamber: 10 Pa to 8 Pa or less

— Resolution of secondary electron image: 20 nm or more

— Magnification: ×5 000

A.3 Effect of the spot size

The spot sizes are changed at 20, 35 and 43. The photos of the surface appearance of the fibres are shown in Figure A.1. The clarity of the surface appearance is affected significantly by the spot size. Before the test observation, the spot size shall be optimized for the SEM by using the identical cupro fibre. The spot size is no dimensional digit depending on the manufacturer of SEM equipment, which is corresponding to beam current.

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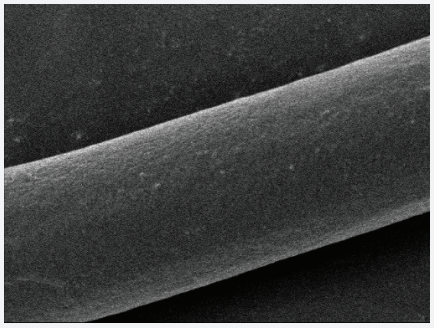
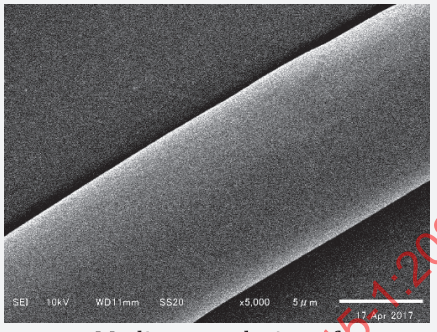
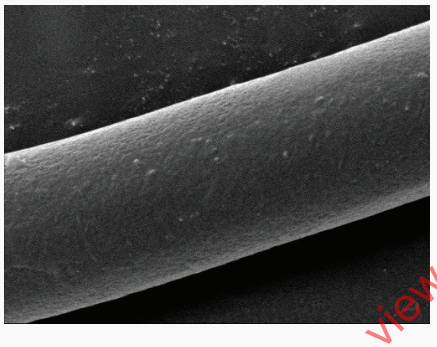
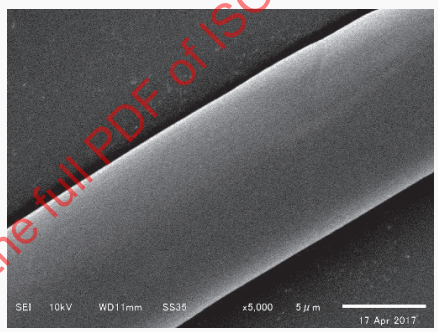
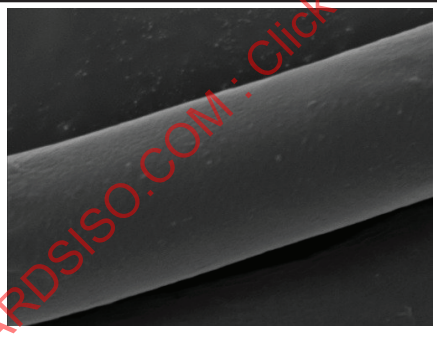
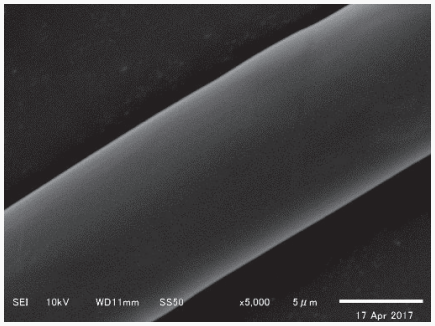
Spot size/ Beam current	Observation by SEM: longitudinal view	
Fibre	**Cupro**	**Lyocell**
20/6 pA	 Medium resolution of the fibre surface image	 Medium resolution of the fibre surface image
35/36 pA	 High resolution	 High resolution
43/100 pA	 Low resolution	 Low resolution

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Figure A.1 — SEM surface appearance of cupro and lyocell with spot size/beam current

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Annex B
(informative)

Interlaboratory test results of SEM method

B.1 General

The interlaboratory test was performed from January to February 2018 with 6 laboratories. The results are shown in below.

B.2 Sample preparation

The samples for the interlaboratory test were prepared as shown in Table B.1.

Table B.1 — Samples for interlaboratory test

Sample	Composition of fibres	
	Cupro (%)	Lyocell (%)
A (yarn)	80	20
B (yarn)	40	60
C (yarn)	0	100
D (yarn)	60	40
E (yarn)	100	0
F (yarn)	20	80
G (fabric)	30	70

B.3 Test result

B.3.1 General

The summary of the test results is shown in Table B.2. From the blend yarns and fabric, both cupro and lyocell were identified significantly and only error was found out for the cupro 100 % and the lyocell 100 % samples as shown in Figure B.3 and Figure B.5.

Table B.2 — Summary of the interlaboratory test results

Sample	Composition of fibres		Test result		
	Cupro (%)	Lyocell (%)	The result	Cupro	Lyocell
A (yarn)	80	20	Figure B.1	○	○
B (yarn)	40	60	Figure B.2	○	○
C (yarn)	0	100	Figure B.3	—	○
D (yarn)	60	40	Figure B.4	○	○
E (yarn)	100	0	Figure B.5	○	—
F (yarn)	20	80	Figure B.6	○	○
G (fabric)	30	70	Figure B.7	○	○

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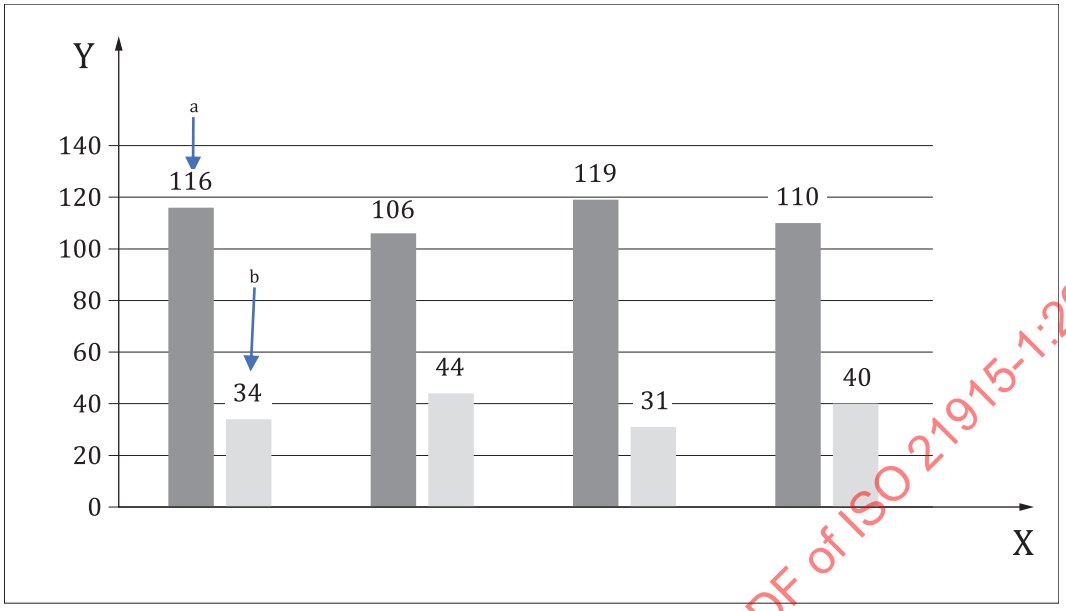
B.3.2 Test results of Sample A

Sample A was prepared as cupro 80 %/lyocell 20 % and the test results are shown in Table B.1.



Key

X laboratory number
Y fibre count
a Cupro.
b Lyocell.

Figure B.1 — Interlaboratory test result of Sample A

B.3.3 Test results of Sample B

The Sample B was prepared as cupro 40 %/lyocell 60 % and the test results are shown in Figure B.2.

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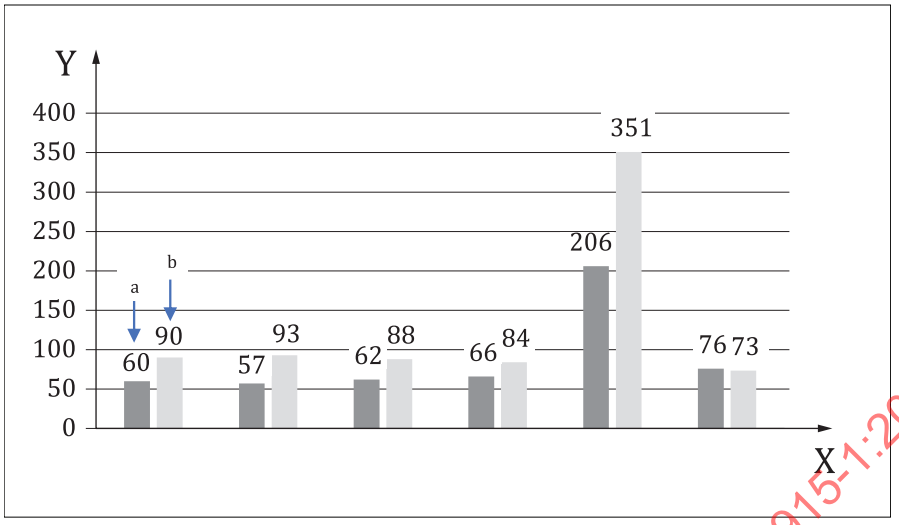


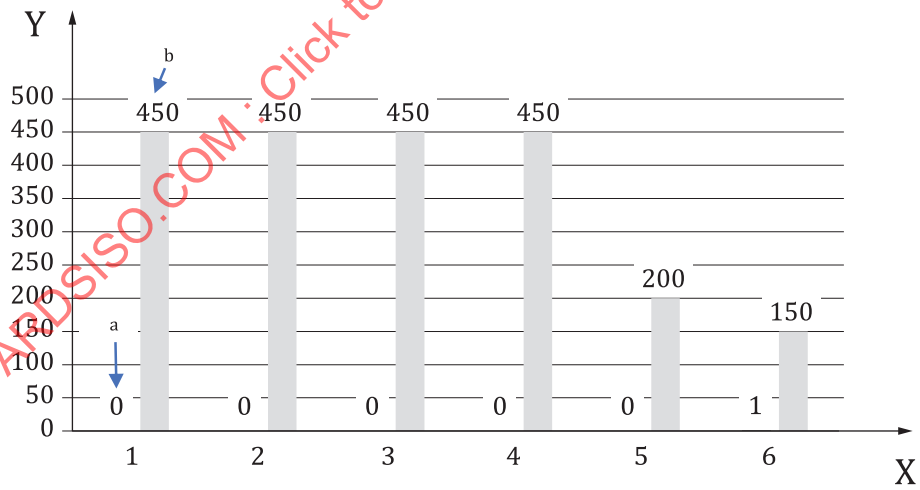
Key

X laboratory number
Y fibre count
a Cupro.
b Lyocell.

Figure B.2 — Interlaboratory test result of Sample B

B.3.4 Test results of Sample C

The Sample C was prepared as lyocell 100 % and the test results are shown in Figure B.3.



Key

X laboratory number
Y fibre count
a Cupro.
b Lyocell.

Figure B.3 — Interlaboratory test result of Sample C

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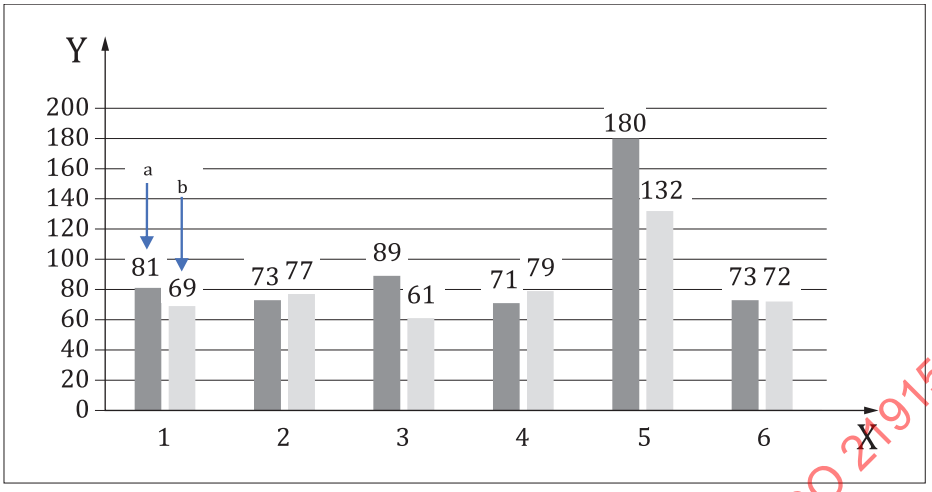
B.3.5 Test results of Sample D

The Sample D was prepared as cupro 60 % and lyocell 40 % and the test results are shown in Figure B.4.



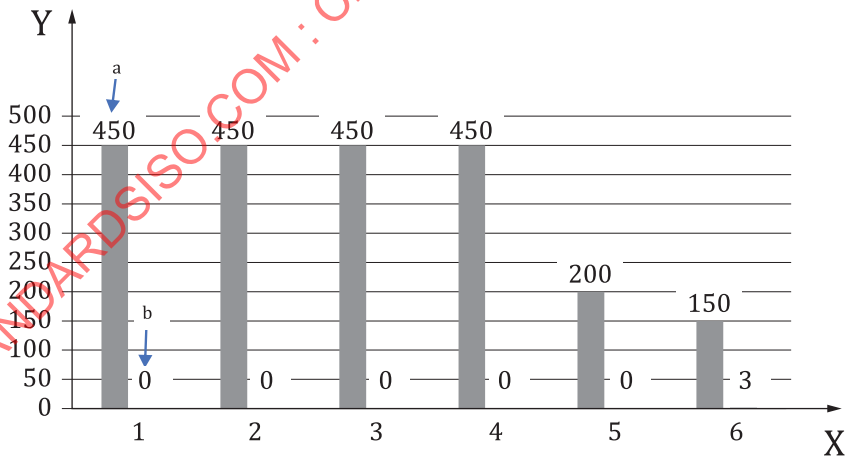
Key

X laboratory number
Y fibre count
a Cupro.
b Lyocell.

Figure B.4 — Interlaboratory test result of Sample D

B.3.6 Test results of Sample E

The Sample E was prepared as cupro 100 % and lyocell 0 % and the test results are shown in Figure B.5.



Key

X laboratory number
Y fibre count
a Cupro.
b Lyocell.

Figure B.5 — Interlaboratory test result of Sample E

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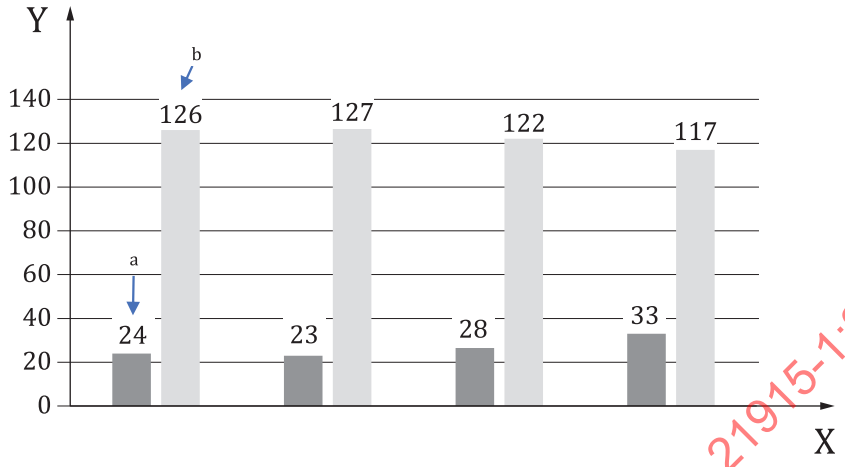
B.3.7 Test results of Sample F

The Sample F was prepared as cupro 20 % and lyocell 80 % and the test results are shown in Figure B.6.



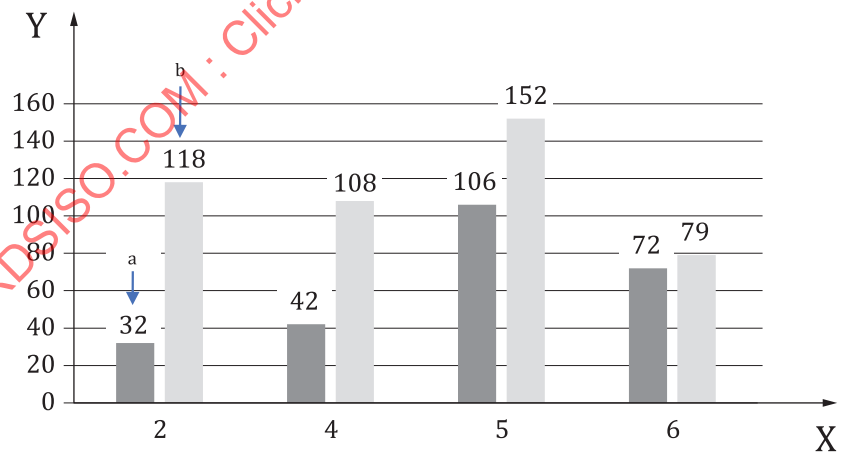
Key

X laboratory number
Y fibre count
a Cupro.
b Lyocell.

Figure B.6 — Interlaboratory test result of Sample F

B.3.8 Test results of Sample G

The Sample G was prepared as cupro 30 % and lyocell 70 % and the test results are shown in Figure B.7.



Key

X laboratory number
Y fibre count
a Cupro.
b Lyocell.

Figure B.7 — Interlaboratory test result of Sample G

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Annex C
(informative)

Quantitative analysis of SEM method

SEM method can also be applied as quantitative analysis of blend of cupro and lyocell.

In this case, as an information that total of all the fibres observed for the test specimen is more than 600 as described in ISO 20705.

However, the quantitative analysis using SEM method takes a lot of time, because the observation magnification is used about ×4 000, as shown in 7.1.5.5. For information, the magnification of SEM described in ISO 17751-2 is ×1 000.

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