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

Part 3: Blend quantification using spectral analysis method

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

Partie 3: Quantification du mélange par une méthode d'analyse spectrale

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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 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 for producers to attach information on the type of fibres used and their mixing ratio to textile products.

Therefore, it is desirable that qualitative methods of all fibres used in textile products and quantitative methods in the case where fibres are mixed (all combinations that can be assumed) exist as test standards.

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

However, cupro and lyocell are difficult to qualify. 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. That is, even if we know that unknown fibre is a cupro or lyocell, we cannot identify which one is.

Therefore, it is difficult to distinguish cupro or lyocell if the cupro or lyocell exists in the textile product or the possibility that cupro and lyocell are mixed completely cannot be denied.

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 201915-2 and this document specify the methods for the composition analysis. The methods used is determined by the instrument availability and experience.

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

Part 3:
Blend quantification using spectral analysis method

1 Scope

This document specifies quantitative testing methods of fibres that consist of cupro and lyocell by using infrared spectroscopy (IR) analysis and multivariate analysis.

This testing method is applied only for cupro or lyocell or a mix of both. The other fibres, such as cotton, viscose, etc. are identified using the test method of ISO/TR 11827 and removed using the relevant part of the ISO 1833 series.

2 Normative references

There are no normative references in this document.

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
calibration model
result of calculation by using the partial least squares (PLS) regression between the IR absorption data and the composition of cupro fibre in percentage

4 Principle

Prepare the calibration specimen with the composition of cupro or lyocell is known in advance and set the objective variables as the composition of cupro in percentage. Measure the ATR-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 (PLS) regression by using the obtained IR data and the composition percentage of cupro.

Then, prepare the testing specimen the same way as the calibration specimen. Measure IR absorption and obtain IR data. Input the data into the calibration model and obtain the composition percentage result for cupro.

5 Apparatus and material

5.1 Infrared spectroscopy (IR) instrument, capable of performing measurement by attenuated total reflection (ATR).

5.2 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.

5.3 Cryogenic grinder.

5.4 Liquid nitrogen.

6 Procedure

6.1 Development of a calibration model

6.1.1 Preparation of specimens

6.1.1.1 Prepare cupro fibre and lyocell fibre separately.

6.1.1.2 Freeze the cupro and lyocell separately by liquid nitrogen (5.4) and shatter them by cryogenic grinder (5.3) for about 1 min into powder.

6.1.1.3 Blend the powders to make a calibration specimen with the ratios such as cupro/lyocell, 100/0, 80/20, 60/40, 40/60, 20/80 and 0/100. These are used for the objective variables (as 100 %, 80 %, 60 %, 40 %, 20 % and 0 % as for the composition of cupro. These objective valuables are named corresponding reference value in Annex A.

The other ratio can be used. If other compositions are used, record it in the test report.

6.1.1.4 Mold the blended powder into three tablets for one blend for the development of the calibration model.

6.1.2 Measurement of IR on the calibration specimens

Measure IR absorption on all specimens by using IR instrument (5.1) according to ATR method with the following condition and record the data.

— Wave number range: 800 cm^{-1} to 3 000 cm^{-1}

— Wave number interval: 1,0 cm^{-1}

6.1.3 Development of the calibration model

6.1.3.1 Assign the objective variables as the composition of cupro in percentage as shown in 6.1.1.3.

6.1.3.2 Calculate the partial least squares (PLS) regression between the IR absorption data and the objective variables by using the software (5.2) as the multivariate analysis.

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

6.1.4 Optimization of the calibration model

6.1.4.1 Optimize the obtained calibration model by using the software (5.2) of the multivariate analysis according to software instruction.

6.1.4.2 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 A.

6.1.4.3 Compare the SEC and R^2 value with the desired values as accuracies, which were 10 or less for SEC and 0,7 or higher for R^2 in this document.

6.1.4.4 If the accuracies, SEC and R^2 are not met the desired values, take the procedure 6.1.4.2 and 6.1.4.3 again. If the accuracies are met the desired value, proceed to next procedure.

6.1.4.5 Take the cross validation process by using the software (5.2) of the multivariate analysis according to the software instruction to evaluate stability.

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

6.1.4.7 Compare the SECV and R^2 value of the cross validation with the desired values, which were 10 or less for SECV and 0,7 or higher for R^2 of the cross validation in this document.

6.1.4.8 If the accuracies, SECV and R^2 of the cross validation are not met the desired values, take the procedure from 6.1.4.2 to 6.1.4.7 again. If the accuracies and the stability are met the desired value, proceed to next.

6.2 Measurement of test sample

6.2.1 Preparation of specimen

6.2.1.1 Prepare a sufficient amount of test sample to prepare the test specimen for three tablets.

6.2.1.2 Frost and shatter the test sample by using cryogenic grinder (5.3) into powder.

6.2.1.3 Mold the powdery test sample into a tablet.

6.2.1.4 Measure IR absorption and obtain data by ATR method according to the same condition of 6.1.2.

6.2.2 Calculation

6.2.2.1 Input the IR absorption data into calibration model developed in 6.1 and calculate the predicted composition of cupro for the sample.

6.2.2.2 Obtain the 3 data from three specimens and average of the 3 data.

7 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 21915-3:2020;

b) all details necessary to identify the product analysed (such as manufacturer, product type, batch or date of manufacture, as required);

c) the result obtained, expressed as the composition of cupro and lyocell nearest 0,1;

d) details of any deviation from the specified procedure;

e) any unusual features observed;

f) the date of the test.

Annex A
(informative)

Optimization of calibration model

A.1 Optimization of calibration model

A.1.1 General

The calibration model calculated by using the partial least squares (PLS) regression of the multivariate analysis is optimised as using the standard error of calibration (SEC) which is defined and calculated in the software as the following.

A.1.2 Definition of SEC

The standard error of calibration (SEC) is defined as Formula (A.1):

$$\mathrm{SEC} = \sqrt{\frac{\sum\left(\hat{Y}_{\mathrm{cal}} - Y_{\mathrm{ref}}\right)^2}{n_{\mathrm{df}}}} \tag{A.1}$$

where

$\hat{Y}_{\mathrm{cal}}$ is the value predicted by the calibration model for the calibration specimen;

Y_{ref} is the corresponding reference value;

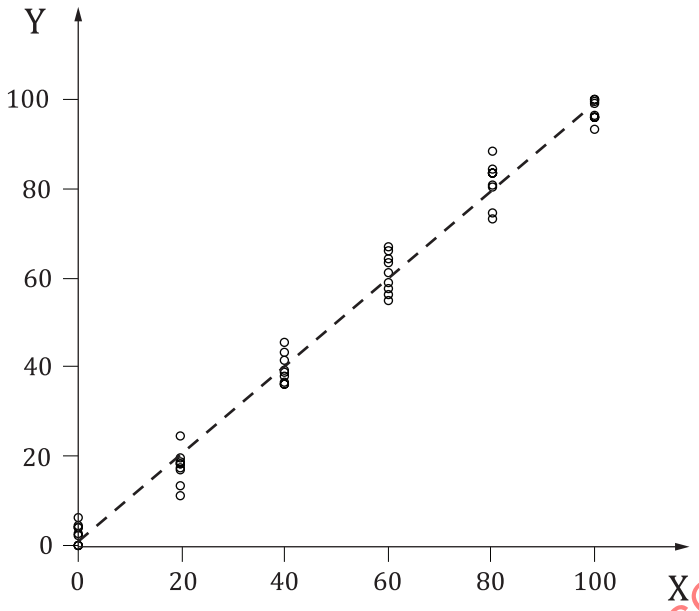
n_{df} is the number of degrees of freedom in the calibration model.

The SEC is used in estimating the expected agreement between values calculated using the calibration model and reference values.

A.1.3 Example of optimization

The SEC and R^2 were calculated and given by the software for the optimization of the calibration model as shown in Figure A.1. The data for the interlaboratory trial was used for the calculation of the optimization. The SEC was obtained as 3,99 and R^2 was 0,99.

The optimization criteria are that the desired values of SEC were 10 or less for SEC and R^2 is 0,7 or higher in this document. The result showed that the calibration model was optimised.



Key

X reference value

Y prediction value of calibration model

Figure A.1 — Reference value and prediction value

A.2 Cross validation of calibration model

The calibration model calculated is validated by using the standard error of cross validation (SECV) which is defined and calculated in the software as the following.

A.2.1 Definition of SECV

The standard error of cross validation (SECV) is defined as Formula (A.2):

$$\mathrm{SECV} = \sqrt{\frac{\sum \left(\hat{Y}_{\mathrm{cv}} - Y_{\mathrm{ref}}\right)^2}{n}} \tag{A.2}$$

where

$\hat{Y}_{\mathrm{cv}}$ is the value predicted by the calibration model in cross validation for the calibration specimen;

Y_{ref} is the corresponding reference value;

n is the number of calibration specimen.

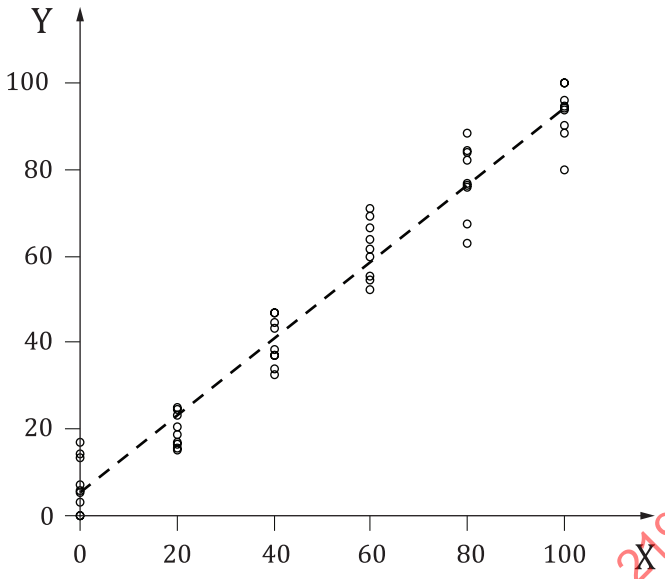
The SECV is used in estimating the stability of the calibration model for unknown samples.

A.2.2 Example of cross validation

After the optimization of the calibration model, the SECV and R^2 were calculated and given by the software for the cross validation of the calibration model as shown in Figure A.2.

The SECV was obtained as 7,31 and R^2 was 0,98.

The cross validation criteria is that the desired values of SECV was 10 or less and R^2 is 0,7 or higher in this document. The result showed that the calibration model was validated.



Key

X reference value

Y prediction value of cross validation

Figure A.2 — Reference value and prediction value (cross validation)

Annex B
(informative)

Interlaboratory test result

B.1 General

The interlaboratory test was performed from January to February 2018 with 4 laboratories. The results are shown in below.

B.2 Test sample

The 6 test samples were prepared by the organizer. The detail of samples is shown in Table B.1.

Table B.1 — Details of sample

Sample name	Composition of sample (%)	
	Cupro	Lyocell
#1	0	100
#2	20	80
#3	40	60
#4	60	40
#5	80	20
#6	100	0

The tablet samples were prepared by using the powder of cupro and lyocell according to the testing procedure 6.1.1 and delivered to testing laboratories.

B.3 Test result

The prediction values are obtained as Table B.2.

Table B.2 — Interlaboratory test result

Sample name	Declared fibre composition	Test result [cupro (%)]					
	cupro (%)	Lab1	Lab2	Lab3	Lab4	Total average	Standard deviation
#1	0	1,7	5,5	1,8	1,7	1,7	0,1
#2	20	11,6	22,3	23,4	25,0	20,5	6,1
#3	40	51,1	34,9	39,7	30,2	39,0	9,0
#4	60	66,2	52,8	63,3	52,1	58,6	7,2
#5	80	90,0	77,2	77,1	85,1	82,3	6,3
#6	100	98,4	98,4	97,4	91,6	98,1	0,6

The shaded results in Table B.2 are excluded from the calculation of the total average and standard deviation because the z' scores exceed 2,0 as shown in Table B.3.