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Cotton fibres — Test method for sugar content — Spectrophotometry

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National foreword

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**Cotton fibres — Test method for sugar
content — Spectrophotometry**

*Fibres de coton — Méthode d'essai pour la teneur en sucre —
Spectrophotométrie*



Reference number
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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).

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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*, Subcommittee SC 23, *Fibres and yarns*.

Introduction

Cotton fibre with normal sugar content might not affect the spinning process. If the sugar content is too high, it might cause storage mildew and metamorphism. During the spinning process, it might also cause twining and breakage, and lower yarn quality and production efficiency. Spectrophotometry is used as a quantitative determination method to detect the total sugar content, and 3,5-dihydroxytoluene-sulfuric acid solution is used as the colour developer. This International Standard supplies the basic information for sugar content of cotton fibres.

Cotton fibres — Test method for sugar content — Spectrophotometry

WARNING — The use of this International Standard might involve the use of hazardous materials, operations, and equipment. This International Standard does not purport to address all the safety risks associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of local regulatory limitations prior to use.

1 Scope

This International Standard specifies a test method to determine the total sugar content in cotton fibres. Spectrophotometry is used as a quantitative determination method, and 3,5-dihydroxytoluene-sulfuric acid solution is used as a colour developer. This International Standard is applicable to cotton fibres.

2 Normative references

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

ISO 139:2005, *Textiles — Standard atmospheres for conditioning and testing*

ISO 1130:1975, *Textile fibres — Some methods of sampling for testing*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

3 Terms and definitions

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

3.1

percentage of sugars

percentage content of total sugars contained in cotton fibres (including reducing sugar and non-reducing sugar) in the total mass of cotton fibres under the standard atmosphere

4 Principle

Under the action of the non-ionic surfactant (fatty acid alkanolamide), sugars in cotton fibres are dissolved in an aqueous solution, then degraded into furfural derivatives in concentrated acid medium (H₂SO₄). They are later combined with 3,5-dihydroxytoluene to form an orange-yellow complex. Quantify the content of sugars in the cotton fibres by the spectrophotometric method in comparison with the calibration curve at the wavelength of 425 nm.

5 Apparatus

5.1 Spectrophotometer, with a wavelength range of 200 nm to 800 nm.

5.2 Electronic balance, selected from the following.

5.2.1 Measuring range ≥ 10 g, accurate to 0,001 g.

5.2.2 Measuring range ≥ 50 g, accurate to 0,1 g.

5.3 **Water bath**, capable of maintaining a constant temperature of (70 ± 2) °C.

5.4 **Conical flask**, of capacity 250 ml, with ground stopper or iodine flask.

5.5 **Pipettes**, of capacity 1 ml, 2 ml and 5 ml.

An automatic pipette system of the same accuracy as manual pipettes may be used.

5.6 **Volumetric flasks**, of capacity 50 ml and 100 ml.

5.7 **Graduated cylinders**, of capacity 100 ml and 1 000 ml.

5.8 **Beakers**, of capacity 100 ml and 1 000 ml.

5.9 **Colorimetric tube**, of capacity 25 ml.

5.10 **Vacuum air pump**.

5.11 **Filter**, made from heat resistant glass having a pore size between 40 μm and 100 μm (pore type P100 in accordance with ISO 4793:1980).

5.12 **Mechanical shaker**, providing rotation or movement sufficient to obtain a ready exchange of liquid between the cotton fibres and the solution used in preparing the extract.

The amplitudes of to-and-fro and rotational mechanical shaker should be set as 30 mm~70 mm and 20 mm~60 mm, respectively. A to-and-fro movement at a rate of 60 min^{-1} or a rotational frequency of 30 min^{-1} has been found satisfactory.

5.13 **Cuvettes**, 1 cm.

6 Reagents

6.1 **Distilled water or grade 3 water**, complying with ISO 3696:1987.

6.2 **Sulfuric acid**, analytical grade, 1,84 g/ml.

6.3 **3,5-Dihydroxytoluene**, analytical grade.

6.4 **Fatty acid alkanolamide**, technical grade.

6.5 **D-fructose**, analytical grade.

6.6 **Reagents preparation**.

6.6.1 **3,5-Dihydroxytoluene/sulfuric acid solution (0,2 %)**.

Weigh and dissolve 0,2 g of 3,5-dihydroxytoluene with 100 g of H_2SO_4 (approximately 54 ml) in a 100 ml beaker with continuous stirring. This solution should be freshly prepared.

6.6.2 Fatty acid alkanolamide solution for determination (0,4 g/l).

Dissolve 0,4 g of fatty acid alkanolamide in 1 000 ml of water and store it in a beaker. Mix well.

6.6.3 Fatty acid alkanolamide solution for extraction (0,05 g/l).

Dilute 100 ml of the solution (prepared in [6.6.2](#)) with water to 700 ml in a beaker. Mix well.

7 Sampling

7.1 Sample in accordance with ISO 1130:1975. Store the samples to avoid the effect of temperature and humidity.

7.2 Randomly select a test specimen, no less than 50 g, from the laboratory samples. Remove any coarse impurities and mix thoroughly.

7.3 As specified in ISO 139:2005, test the test specimen after conditioning it in the standard atmosphere no less than 48 h (or until the difference between successive weighings made at intervals of 2 h does not exceed 0,25 %).

7.4 Weigh accurately three portions of the prepared specimen (each portion should weigh $2,000 \text{ g} \pm 0,001 \text{ g}$), recorded as *m*, and reserve the residual sample for further use.

8 Procedure

8.1 Blank test

8.1.1 Pipette 1,0 ml of 0,4 g/l fatty acid alkanolamide solution ([6.6.2](#)) into a 25 ml colorimetric tube ([5.9](#)).

8.1.2 Add 2,0 ml of 3,5-dihydroxytoluene/sulfuric acid solution ([6.6.1](#)) immediately to the colorimetric tube preheated to 70 °C in a water bath ([5.3](#)), and mix them by shaking to form a mixed solution, and again keep the mixed solution at a constant temperature of 70 °C for 40 min. Remove the tube from the bath. Subsequently, add further 20 ml of the fatty acid alkanolamide solution ([6.6.2](#)) to the mixed solution, mix them by shaking the tube, immediately cool down the resultant solution to room temperature and make it up to the mark with the 0,4 g/l fatty acid alkanolamide solution ([6.6.2](#)) and allow it to stand for 5 min to form a final solution. Transfer an appropriate amount of the final solution into a 1 cm cuvette to form a blank reagent. Measure and record the absorbance of the blank reagent at a wavelength of 425 nm using a Spectrophotometer ([5.1](#)).

8.2 Calibration curve preparation

8.2.1 Standard solutions preparation

8.2.1.1 Stock standard solution

Prepare a 2,0 mg/ml stock solution of D-fructose by dissolving 0,200 g D-fructose with 100 ml water.

8.2.1.2 Standard solution

Dilute 0,5 ml, 1,0 ml, 1,5 ml, 2,0 ml, 2,5 ml, 3,0 ml, 4,0 ml, and 5,0 ml of the stock standard solution (see [8.2.1.1](#)), with water in 50 ml volumetric flasks. These solutions contain 0,02 mg/ml, 0,04 mg/ml, 0,06 mg/ml, 0,08 mg/ml, 0,10 mg/ml, 0,12 mg/ml, 0,16 mg/ml, and 0,20 mg/ml of D-fructose, respectively.

8.2.2 Testing of standard solution

Pipette 1,0 ml of each of the standard solutions into a different 25 ml colorimetric tube, and treat each aliquot in the different tube in the same way as that described in [8.1.2](#).

8.2.3 Calibration curve

Prepare a calibration curve with the concentration of the sugar standard solution as the abscissa and the corresponding absorbance (obtained by subtracting the absorbance of the blank reagent from the absorbance of the sugar standard solution) as the vertical coordinate.

Since the sensitivity of fatty acid alkanolamide solution may change slightly over a long period of time, it is good practice to run a calibration curve weekly for correcting slight changes in the standard curve.

8.3 Determination of sugar content for test specimens

8.3.1 Place the three test specimens (see [7.4](#)) in three separate 250 ml flasks. Add 200 ml of the 0,05 g/l fatty acid alkanolamide solution ([6.6.3](#)) to each flask. Shake for 10 min in the mechanical shaker ([5.12](#)). Turn over the cotton fibre with a glass rod. Shake the flasks for another 10 min and then filter the three test specimen solutions respectively through the filter ([5.11](#)) using vacuum air pump ([5.10](#)).

8.3.2 Pipette 1,0 ml of each of the three test specimen solutions (see [8.3.1](#)) into a separate 25 ml colorimetric tube. Treat in the same way as [8.1.2](#).

9 Calculation and expression of the results

9.1 For each test specimen, correct the absorbance of the test specimen by subtracting the absorbance of the blank reagent from the absorbance of the test specimen. Determine the concentration of the sample solution from the calibration curve. Calculate the percentage of sugars according to the following formula.

$$x = \frac{200 \times c}{1000 \times m} \times 100$$

where

- x is the percentage of sugars, in %;
- c is the concentration of the sugar solution (mg/ml) as read from the calibration curve;
- m is the mass of the test specimen in grams.

9.2 Calculate the mean of the measured values, rounding to two decimal places.

10 Test report

The test report shall include the following information:

- a) a reference to this International Standard, i.e. ISO 18068;
- b) date of receipt and date of analysis;
- c) description of the sample tested;
- d) mass of the test specimens;

- e) range of the calibration graph;
- f) percentage of sugars from the sample;
- g) any deviation, by agreement or otherwise, from the procedure specified.

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