INTERNATIONAL STANDARD

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Textiles — Quantitative chemical analysis —

Part 15:

Mixtures of jute and certain animal fibres (method by determining nitrogen content)

Textiles — Analyse chimique quantitative —

Partie 15: Mélanges de jute et de certaines fibres animales (méthode par dosage de l'azote)



Reference number ISO 1833-15:2006(E)

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Foreword

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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.

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 1833-15 was prepared by Technical Committee ISO/TC 38, Textiles.

This first edition of ISO 1833-15 cancels and replaces Clause 14 of ISO 1833:1977.

ISO 1833:1977 will be cancelled and replaced by ISO 1833-1, ISO 1833-3, ISO 1833-4, ISO 1833-5, ISO 1833-6, ISO 1833-7, ISO 1833-8, ISO 1833-9, ISO 1833-10, ISO 1833-11, ISO 1833-12, ISO 1833-13, ISO 1833-14, ISO 1833-15, ISO 1833-16, ISO 1833-17, ISO 1833-18 and ISO 1833-19.

ISO 1833 consists of the following parts, under the general title Textiles — Quantitative chemical analysis:

- Part 1: General principles of testing
- Part 2: Ternary fibre mixtures
- Part 3: Mixtures of acetate and certain other fibres (method using acetone)
- Part 4: Mixtures of certain protein and certain other fibres (method using hypochlorite)
- Part 5: Mixtures of viscose, cupro or modal and cotton fibres (method using sodium zincate)
- Part 7: Mixtures of polyamide and certain other fibres (method using formic acid)
- Part 8: Mixtures of acetate and triacetate fibres (method using acetone)
- Part 9: Mixtures of acetate and triacetate fibres (method using benzyl alcohol)
- Part 10: Mixtures of triacetate or polylactide and certain other fibres (method using dichloromethane)
- Part 11: Mixtures of cellulose and polyester fibres (method using sulfuric acid)
- Part 12: Mixtures of acrylic, certain modacrylics, certain chlorofibres, certain elastanes and certain other fibres (method using dimethylformamide)
- Part 13: Mixtures of certain chlorofibres and certain other fibres (method using carbon disulfide/acetone)

ISO 1833-15:2006(E)

- Part 15: Mixtures of jute and certain animal fibres (method by determining nitrogen content)
- Part 16: Mixtures of polypropylene fibres and certain other fibres (method using xylene)
- Part 17: Mixtures of chlorofibres (homopolymers of vinyl chloride) and certain other fibres (method using sulfuric acid)
- Part 18: Mixtures of silk and wool or hair (method using sulfuric acid)
- Part 19: Mixtures of cellulose fibres and asbestos (method by heating)
- Part 21: Mixtures of chlorofibres, certain modacrylics, certain elastanes, acetates, triacetates and certain other fibres (method using cyclohexanone)

The following parts are under preparation:

- Part 6: Mixtures of viscose or certain types of cupro or modal or lyocell and cotton fibres (method using formic acid and zinc chloride)
- Part 20: Mixtures of elastane and certain other fibres (method using dimethylacetamide)
- Part 22: Mixtures of viscose or certain types of cupro or modal or lyocell and flax fibres (method using formic acid and zinc chlorate)
- Part 23: Mixtures of polyethylene and polypropylene (method using cyclohexanone)
- Part 24: Mixtures of polyester and some other fibres (method using phenol and tetrachloroethane)

Textiles — Quantitative chemical analysis —

Part 15:

Mixtures of jute and certain animal fibres (method by determining nitrogen content)

1 Scope

This part of ISO 1833 specifies a method, by determining the nitrogen content, to calculate the proportion of each component, after the removal of non-fibrous matter, in textiles made of binary mixtures of

| | jute | |
|-----|------|--|
| and | I | |

animal fibres.

The animal-fibre component may consist solely of hair or wool, or of any mixtures of the two.

This part of ISO 1833 is not applicable to products in which dyestuffs or finishes contain nitrogen.

NOTE Because this method differs in principle from the general method based on selective solubility set out in ISO 1833-1, it is given in a form that is complete in itself.

2 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 1833-1, Textiles — Quantitative chemical analysis — Part 1: General principles of testing

3 Principle

The nitrogen content of the mixture is determined, and from this and the known or assumed nitrogen contents of the two components, the proportion of each component is calculated.

Reagents 4

All reagents shall be of recognized analytical reagent quality.

- 4.1 Toluene.
- 4.2 Methanol.
- 4.3 Sulfuric acid, $\rho = 1.84 \text{ g/ml.}^{1)}$
- Potassium sulfate. 1) 4.4
- Selenium dioxide. 1) 4.5
- Sodium hydroxide solution, 400 g/l. 4.6

Dissolve 400 g of sodium hydroxide in 400 ml to 500 ml of water, and dilute to 1 l with water.

4.7 Mixed indicator.

Dissolve 0,1 g of methyl red in 95 ml of ethanol and 5 ml of water, then mix with 0,5 g of bromocresol green dissolved in 475 ml of ethanol and 25 ml of water.

Boric acid solution. 4.8

Dissolve 20 g of boric acid in 1 l of water.

4.9 Sulfuric acid, 0,01 mol/l standard volumetric solution.

Apparatus 5

Use the apparatus described in ISO 1833-1 together with those given in 5.1, 5.2 and 5.3.

- 5.1 Kjeldahl digestion flask, capacity 200 ml to 300 ml.
- 5.2 Kjeldahl distillation apparatus with steam injection.
- 5.3 Titration apparatus, allowing a precision of 0,05 ml.

Sampling and pre-treatment of sample

6.1 Sampling

Take a laboratory test sample that is representative of the laboratory bulk sample and sufficient to provide all the specimens, each of about 1 g, that are required. Treat the sample as described in 6.2.

Pre-treatment of sample

Extract the air-dry sample in a Soxhlet apparatus with a mixture of 1 volume of toluene and 3 volumes of methanol for 4 h at a minimum rate of 5 cycles per hour.

¹⁾ These reagents should be free of nitrogen.

Allow the solvent to evaporate from the sample in air, and remove the last traces in an oven at (105 ± 3) °C. Extract the sample in water (50 ml per gram of sample) by boiling under reflux for 30 min. Filter, return the sample to the flask and repeat the extraction with fresh water.

Filter, remove excess water from the sample by squeezing, suction, or centrifuging, and then allow the sample to become air-dry.

SAFETY PRECAUTIONS — The toxic effects of toluene and methanol shall be borne in mind, and full precautions shall be taken in their use.

7 Test procedure

Follow the general procedure given in ISO 1833-1 as regards the selection, drying and weighing of the specimen. Then proceed as follows.

Take from the pre-treated sample a test specimen weighing about 1 g. Dry the specimen in a weighing bottle, cool it in a desiccator, and weigh it.

Transfer the specimen to the dry Kjeldahl digestion flask, reweigh the weighing bottle immediately and obtain the dry mass of the specimen by the difference.

To the specimen in the digestion flask, add in the following order 2,5 g of potassium sulfate, 0,1 g to 0,2 g of selenium dioxide and 10 ml of sulfuric acid (4.3). Heat the flask, gently at first, until the whole of the fibre is destroyed, and then more vigorously until the solution becomes clear and almost colourless. Heat for a further 15 min.

Allow the flask to cool, dilute the contents carefully with 10 ml to 20 ml of water, cool, transfer the contents quantitatively to a 200 ml graduated flask and make up to volume with water to form the digest solution.

Place about 20 ml of boric acid solution in a 100 ml conical flask and place the flask under the condenser of the Kjeldahl distillation apparatus so that the delivery tube dips just below the surface of the boric acid solution.

Transfer exactly 10 ml of digest solution to the distillation flask, add not less than 5 ml of sodium hydroxide solution to the funnel, lift the stopper slightly, and allow the sodium hydroxide solution to run slowly into the flask. If the digest solution and sodium hydroxide solution remain as two separate layers, mix them by gentle agitation. Heat the distillation flask gently and pass into it steam from the steam injection apparatus (Kjeldahl apparatus).

Collect about 20 ml of distillate, lower the receiver so that the tip of the delivery tube is about 20 mm above the surface of the liquid, and distil for 1 min more. Rinse the tip of the delivery tube with water, catching the washings in the receiver. Remove the receiver and replace it with a second receiver containing about 10 ml of boric acid solution, and collect about 10 ml of distillate.

Titrate the two distillates separately with sulfuric acid (4.9), using the mixed indicator. Record the total titre for the two distillates. If the titre for the second distillate is more than 0,2 ml, reject the result and repeat the distillation, using a fresh aliquot of digest solution.

Carry out a blank determination, i.e. digestion and distillation using the reagents only.

8 Calculation and expression of results

Calculate the percentage nitrogen content in the dry specimen as follows: 8.1

$$A = \frac{14\left(V_1 - V_2\right)c}{m_0}$$

where

is the percentage nitrogen content in the clean dry specimen;

is the total volume, in millilitres, of sulfuric acid (4.9) used in the determination;

is the total volume, in millilitres, of sulfuric acid (4.9) used in the blank determination;

is the concentration of the sulfuric acid (4.9), expressed in moles per litre;

is the dry mass, in grams, of the specimen.

Using the values of 0,22 % for the nitrogen content of jute and 16,2 % for the nitrogen content of animal fibre, both values being expressed on the dry mass of the fibre, calculate the composition of the mixture as follows:

$$P_{\mathsf{A}} = \frac{A - 0.22}{16.2 - 0.22} \times 100$$

where P_{A} is the percentage animal fibre in the clean dry specimen.

Precision

On a homogeneous mixture of textile materials the confidence limits of the results obtained by this method are not greater than \pm 1 for the confidence level of 95 %.

ISO 1833-15:2006(E)

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