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Textiles — Quantitative chemical analysis —

Part 28:

Mixtures of chitosan with certain other fibres (method using diluted acetic acid) iTeh STANDARD PREVIEW

> (statiles Analyse chimique quantitative — Partie 28: Mélanges de chitosane avec certaines autres fibres (méthode à l'acide acétique dilué)

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

A list of all parts in the ISO1833 series can be found on the ISO website.7d-4d56-9360-

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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 <u>www.iso.org/members.html</u>.

Textiles — Quantitative chemical analysis —

Part 28: Mixtures of chitosan with certain other fibres (method using diluted acetic acid)

1 Scope

This document specifies a method, using diluted acetic acid, to determine the mass percentage of chitosan fibres, after elimination of non-fibrous matter, in textiles made of mixtures of:

chitosan fibre

with

certain other fibres.

This method is applicable to fibre mixtures of chitosan fibre with cellulose fibres (cotton, linen, ramie, viscose, modal, lyocell), protein fibres (wool, cashmere, silk), or synthetic fibres (polyester, polyamide, acrylic).

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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 1833-1, Textiles — Quantitative chemical analysis — Part 1: 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 <u>https://www.iso.org/obp</u>

— IEC Electropedia: available at http://www.electropedia.org/

3.1

chitosan fibre

chitin fibre in which at least 55 % acetylated groups have been deacetylated

Note 1 to entry: Chitin as generic name, see ISO 2076.

Note 2 to entry: In the textile industry, the deacetylation degree is generally more than 90 %.

Note 3 to entry: See <u>Figure 1</u>.



Key

- 1 chitin
- 2 deacetylation
- 3 chitosan

Figure 1 — Chitosan fibre

4 Principle

The chitosan fibre is dissolved out from a known dry mass of the mixture with diluted acetic acid. The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of chitosan fibre is found by the difference.

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Annex A presents the statistical data for chitosan content. **D PREVIEW**

5 Reagents

Use the reagents described in ISO 1833-1 together with those given in <u>5.1</u> and <u>5.2</u>.

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5.1 Diluted acetic acid, dilute 20 ml of acetic acid (p = 1,05-g/ml at 20 °C) to 1 l with water.

5.2 Diluted ammonia solution, dilute 80 ml of ammonia solution (ρ = 0,88 g/ml at 20 °C) to 1 l with water.

6 Apparatus

Use the apparatus described in ISO 1833-1 together with those given in 6.1 and 6.2.

6.1 Conical flask with stopper, minimum capacity 500 ml.

6.2 Mechanical shaker with water-bath, providing reciprocating movement with the capability to maintain at temperature of 90 °C to 95 °C.

7 Test procedure

Follow the general procedure described in ISO 1833-1, and then proceed as follows.

To the specimen contained in the conical flask (6.1), add 200 ml of diluted acetic acid (5.1) per gram of specimen, insert the stopper, agitate vigorously to wet out the specimen and shake continuously for 60 min in the mechanical shaker (6.2) at 90 °C to 95 °C.

Filter the contents of the flask through the weighed filter crucible and transfer any residual fibres to the crucible by washing out the flask with a little more diluted acetic acid of 90 °C to 95 °C. Drain the crucible using suction. Wash the residue successively with water of 90 °C to 95 °C, twice with dilute

ammonia solution (5.2), and finally with cool water, draining the crucible using suction after each addition. Do not apply suction until each washing liquid has drained under gravity.

Dry the crucible and residue, then cool and weigh them.

8 Calculation and expression of results

Calculate the results as described in the general instructions of ISO 1833-1.

The value of d is 1,00, except for wool, cotton, linen, viscose, modal, lyocell, and acrylic for which d is 1,01, and for cashmere and silk for which d is 1,02.

9 Precision

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

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Annex A

(informative)

Statistic data of interlaboratory trial

The interlaboratory trial was carried out among 8 laboratories from Belgium, China, France, Germany, Italy, Korea (2 laboratories) and Portugal.

Six samples prepared were the mixtures of chitosan separately blended with cotton, polyester, polyamide, wool, acrylic and viscose. In the trial, two specimens from each sample were tested and the test result was expressed by dry mass fraction. The chitosan content obtained was analysed as statistic data.

The statistical data of mean value, repeatability and reproducibility are shown in <u>Table A.1</u>. The robust mean x^* and the robust standard deviation s^* are calculated according to the algorithm A of ISO 13528:2015. The repeatability r is calculated according to the algorithm S of ISO 13528:2015. The variance intergroup s_L^2 is deduced from s^* and r. The reproducibility R is deduced from r and s_L [ISO 5725-5:1998, Formula (74)].

Z'-scores of the 8 laboratories were in satisfactory cases (|Z'| < 2), except for 2 in the case of the sample n°5.

Sample No.	Fibre composition	Mean value (n = 8)	Standard C deviation	n.al) s _r	r	s _R	R
1	cotton/chitosan	10,9 <u>IS</u>	<u>0 1833–38:2019</u>	0,4	1,1	1,3	3,8
2	polyester/chitosan	tandards.teh.ai/catalog	/standards/sist/183	$0^{1443b_{\overline{4}}35/6}_{4}$	1-4d56-9360	0,7	2,1
3	polyamide/chitosan	8,9	0,6	0,3	0,8	0,6	1,8
4	wool/chitosan	10,5	1,4	0,3	0,7	1,4	4,0
5	acrylic/chitosan	13,6	0,4	0,2	0,6	0,4	1,1
6	viscose/chitosan	14,8	1,2	0,5	1,3	1,3	3,6
where							

Table A.1 — Statistical data for Chitosan content (Dry mass fraction, %)

s_r is the standard deviation of the repeatability;

r is the limit of the repeatability;

s_R is the standard deviation of the reproducibility;

R is the limit of the reproducibility.

Bibliography

- [1] ISO 2076, Textiles Man-made fibres Generic names
- [2] ISO 13528:2015, Statistical methods for use in proficiency testing by interlaboratory comparison
- [3] ISO 5725-5:1998, Accuracy (trueness and precision) of measurement methods and results Part 5: Alternative methods for the determination of the precision of a standard measurement method

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