



INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 1833

TEXTILES

BINARY FIBRE MIXTURES QUANTITATIVE CHEMICAL ANALYSIS

1st EDITION

October 1971

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Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

iTeh STANDARD PREVIEW (standards.iteh.ai)

<u>ISO/R 1833:1971</u>

https://standards.iteh.ai/catalog/standards/sist/815362ad-98ae-4078-9746-2d0d06b0664e/iso-r-1833-1971

BRIEF HISTORY

The ISO Recommendation R 1833, Textiles – Binary fibre mixtures – Quantitative chemical analysis, was drawn up by Technical Committee ISO/TC 38, Textiles, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1833, which was circulated to all the ISO Member Bodies for enquiry in January 1970.

The Draft has been approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	India	South Africa, Rep. of
Belgium	Israel	Sweden
Canada	Japan	Switzerland
Czechoslovakia	Netherlands	Turkey
Denmark	New Zealand	U.A.R.
France	Norway	United Kingdom
Germany	Poland	U.S.A.
Greece	Portugal	U.S.S.R.
Hungary —	Romania	

The following Member Body opposed the approval of the Draft :

Italy

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

CONTENTS

	Page
Introduction	5
1. Information common to the methods given for the quantitative chemical analysis of binary fibre mixtures	6
2. Mixtures of acetate and certain other fibres	10
3. Mixtures of certain protein and certain other fibres	11
4. Mixtures of viscose or cupro and cotton fibres using sodium zincate	12
5. Mixtures of viscose or cupro and cotton fibres using formic acid and zinc chloride	14
6. Mixtures of nylon 6 or nylon 6.6 and certain other fibres	15
7. Mixtures of acetate and triacetate fibres using acetone	16
8. Mixtures of acetate and triacetate fibres using benzyl alcohol	17
9. Mixtures of triacetate and certain other fibres	18
10. Mixtures of cellulose and polyester fibres	19
1. Mixtures of acrylic, certain modacrylics or certain chlorofibres and certain other fibres	20
12. Mixtures of certain chlorofibres and certain other fibres	21
13. Mixtures of acetate and certain chlorofibres	22
14. Mixtures of jute and certain animal fibres	23

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ISO/R 1833-1971 (E)

ISO Recommendation

TEXTILES

BINARY FIBRE MIXTURES

QUANTITATIVE CHEMICAL ANALYSIS

INTRODUCTION

In general, the methods described in this ISO Recommendation are based on the selective solution of an individual component. After the removal of a component, the insoluble residue is weighed, and the proportion of soluble component is calculated from the loss in mass. The information common to the analyses by this method of all fibre mixtures, whatever their composition, is given in section 1 of this ISO Recommendation. This should be used in conjunction with the succeeding individual sections of the document which contain the detailed procedures applicable to particular fibre mixtures. Where, occasionally, an analysis is based on a principle other than selective solution, full details are given in the appropriate section.

Mixtures of fibres during processing and, to a lesser extent, finished textiles may contain fats, waxes or dressings, either occurring naturally or added to facilitate processing. Salts and other water-soluble matter may also be present. Some or all of these substances would be removed during analysis, and calculated as the soluble-fibre component. To avoid this error, non-fibrous matter must be removed before analysis and a method of pre-treatment for removing oils, fats, waxes and water-soluble matter is given in section 1.

In addition, textiles may contain resins or other matter added to bond the fibres together or to confer special properties, such as water-repellence or crease-resistance. Such matter, including dyestuffs in exceptional cases, may interfere with the action of the reagent on the soluble component and/or it may be partially or completely removed by the reagent. This type of added matter may also cause errors and should be removed before the sample is analysed. If it is impossible to remove such added matter, the methods of analysis are no longer applicable. Dye in dyed fibres is considered to be an integral part of the fibre and is not removed.

Most textile fibres contain water, the amount depending on the type of fibre and on the relative humidity of the surrounding air. Analyses are conducted on the basis of dry mass, and a procedure for determining the dry mass of test specimens and residues is given in section 1. The result is therefore obtained on the basis of clean, dry fibres.

Provision is made for recalculating the result on the basis of

- (1) agreed allowances for moisture content*, and
- (2) agreed allowances for moisture and also for
 - (a) fibrous matter removed in the pre-treatment, and
 - (b) non-fibrous matter (for example, fibre dressing, processing oil, or size) that can be properly regarded as part of the fibre as an article of commerce.

In some methods, the insoluble component of a mixture may be partially dissolved in the reagent used to dissolve the soluble component. Where possible, reagents have been chosen that have little or no effect on the insoluble fibres. If loss in mass is known to occur during the analysis, the result should be corrected; correction factors for this purpose are given. These correction factors have been determined in several laboratories by treating, in the appropriate reagent as specified in the method of analysis, fibres cleaned by the pre-treatment. These correction factors apply only to undegraded fibres, and different correction factors may be necessary if the fibres have been degraded during processing.

The procedures given apply to single determinations; at least two determinations on separate test specimens should be made, but more may be carried out if desired. Before proceeding with any analysis, all the fibres present in the mixture should have been identified. For confirmation, unless it is technically impossible, it is recommended to use alternative procedures whereby the constituent that would be the residue in the standard method is dissolved out first.

If it is practicable to separate the components of a mixture manually, that method should be used in preference to the chemical methods of analysis given in this ISO Recommendation.

* Suitable values are the official regains, where available, of the respective fibres.

1. INFORMATION COMMON TO THE METHODS GIVEN FOR QUANTITATIVE CHEMICAL ANALYSIS OF BINARY FIBRE MIXTURES

1.1 Scope and field of application

This ISO Recommendation contains methods for the quantitative chemical analysis of various binary mixtures of fibres. The methods given are applicable in general to fibres in any textile form. Where certain textile forms are excepted, these are listed in the "field of application" clause of the individual method.

1.2 Principle

After the identification of the components of a mixture, one component is removed, usually by selective solution, the insoluble residue is weighed, and the proportion of soluble component calculated from the loss in mass. The fibre in the larger proportion is removed first.

1.3 Reagents

All reagents used should be chemically pure.

- 1.3.1 Light petroleum, redistilled, distilling between 40 and 60 °C.
- 1.3.2 Distilled or deionized water.
- 1.3.3 Additional reagents as specified in the appropriate sections of this ISO Recommendation.

1.4 Apparatus

- 1.4.1 Glass filter crucible, capacity 30 to 40 ml, with sealed-in sintered disk filter with pore size of 90 to $150 \,\mu$ m. The crucible should be provided with either a ground glass stopper or a watch-glass cover.
- 1.4.2 Vacuum flask.
- 1.4.3 Desiccator containing self-indicating silica gel.
- 1.4.4 Ventilated oven for drying specimens at 105 ± 3 °C.
- 1.4.5 Analytical balance, accurate to 0.0002 g.
- 1.4.6 Soxhlet extraction apparatus, of sufficient size to give a volume, in millilitres, equal to 20 times the mass, in grammes, of the specimen.
- 1.4.7 Additional apparatus as specified in the appropriate sections of this ISO Recommendation.

1.5 Conditioning and testing atmosphere

Because dry masses are determined, it is unnecessary to condition the specimen. The analysis is carried out under ordinary room conditions.

1.6 Sampling and pre-treatment of sample

1.6.1 Sampling. Take a laboratory test sample* that is representative of the laboratory bulk sample and sufficient to provide all the specimens, each of at least 1 g, that are required. Fabrics may contain yarns of different composition and account must be taken of this fact in the sampling of the fabric. Treat the sample as described in clause 1.6.2.

* See ISO Recommendation R ..., Methods of sampling for chemical testing (at present, document ISO/TC 38 N 378).

1.6.2 Pre-treatment of laboratory test sample. Extract the air-dry sample in a Soxhlet apparatus with light petroleum for 1 hour at a minimum rate of 6 cycles per hour. Allow the light petroleum to evaporate from the sample and then soak the specimen in cold water for 1 hour and then in water at 65 ± 5 °C for a further hour. In both cases use a liquor : specimen ratio of 100 : 1 and agitate the liquor from time to time. Remove the excess water from the sample by squeezing, suction, or centrifuging and then allow the sample to become air-dry.

Where non-fibrous matter cannot be extracted with light petroleum and water, it should be removed by a suitable method that does not substantially alter any of the fibre constituents. However, for some unbleached, natural vegetable fibres (for example jute, coir) it is to be noted that normal pre-treatment with light petroleum and water does not remove all the natural non-fibrous substances; nevertheless, additional pre-treatment is not applied unless the sample does contain finishes insoluble in both light petroleum and water.

1.7 Test procedure

1.7.1 General instructions

- 1.7.1.1 DRYING. Conduct all drying operations for not less than 4 hours and not more than 16 hours at 105 ± 3 °C in a ventilated oven with the oven door closed throughout.
- **1.7.1.2** DRYING OF SPECIMEN. Dry the specimen in a weighing bottle with its stopper beside it. After drying, stopper the weighing bottle before removing it from the oven, and transfer it quickly to a desiccator.
- 1.7.1.3 DRYING OF CRUCIBLE AND RESIDUE. Dry the filter crucible with its stopper or cover beside it in the oven. After drying, close the crucible and transfer it quickly to a desiccator.
- 1.7.1.4 COOLING. Conduct all cooling operations until complete cooling is attained, and in any case for not less than 2 hours with the desiccator beside the balance.
- 1.7.1.5 WEIGHING. After cooling, complete the weighing of the weighing bottle or crucible within 2 minutes of its removal from the desiccator. Weigh to an accuracy of 0.0002 g.

NOTE. - Do not handle the crucibles, specimens or residues with bare hands during the drying, cooling and weighing operations.

1.7.2 Procedure. Take from the pre-treated laboratory test sample a test specimen weighing about 1 g. Cut yarn or dissected cloth into lengths of about 10 mm. Dry the specimen in a weighing bottle, cool it in a desiccator and weigh it. Transfer the specimen to the glass vessel specified in the appropriate part of this ISO Recommendation, reweigh the weighing bottle immediately, and obtain the dry mass of the specimen by difference.

Complete the test procedure as specified in the appropriate section of this ISO Recommendation, and examine the residue microscopically, or otherwise, as appropriate, to check that the treatment has in fact completely removed the soluble fibre.

1.8 Calculation and expression of results

Express the mass of the insoluble component as a percentage of the total mass of fibre in the mixture. Calculate the result on a clean dry mass basis as in clause 1.8.1, or on clean dry mass with agreed percentage additions for moisture as in clause 1.8.2, or on clean dry mass with agreed percentage additions for moisture and

- (a) fibrous matter removed in the pre-treatment, and
- (b) non-fibrous matter as in clause 1.8.3.

Obtain the percentage of the soluble component by difference. State which of the calculation procedures has been used and, if it is the second or third, state the values of the percentage additions.

1.8.1 Method based on clean dry mass

$$P = \frac{100 m_1 d}{m_0}$$

where

P is the percentage of clean dry insoluble component;

 m_0 is the dry mass of the specimen;

 m_1 is the dry mass of the residue;

d is the correction factor of variation in mass of the insoluble component in the reagent. Suitable values of d are given in the appropriate sections of this ISO Recommendation.

1.8.2 Method based on clean dry mass with percentage additions for moisture

$$P_{\rm M} = \frac{100 P (1 + 0.01 a_2)}{P (1 + 0.01 a_2) + (100 - P) (1 + 0.01 a_1)}$$

where

 $P_{\rm M}$ is the percentage of clean insoluble component with percentage additions for moisture;

P is the percentage of clean dry insoluble component;

 a_1 is the percentage addition to the soluble component for moisture;

 a_2 is the percentage addition to the insoluble component for moisture.

1.8.3 Method based on clean dry mass with percentage additions for moisture and

- (a) fibrous matter removed in the pre-treatment, and
- (b) non-fibrous matter

$$P_{\rm A} = \frac{100 P [1 + 0.01 (a_2 + b_2)]}{P [1 + 0.01 (a_2 + b_2)] + (100 - P) [1 + 0.01 (a_1 + b_1)]}$$

where

- $P_{\rm A}$ is the percentage of clean insoluble component in the mixture with percentage additions for moisture and non-fibrous matter;
- *P* is the percentage of clean dry insoluble component;
- a_1 is the percentage addition to the soluble component for moisture;
- a_2 is the percentage addition to the insoluble component for moisture;
- b_1 is the percentage loss of soluble fibrous matter caused by the pre-treatment, and/or the percentage addition to the soluble component for non-fibrous matter;
- b_2 is the percentage loss of insoluble fibrous matter caused by the pre-treatment, and/or the percentage addition to the insoluble component for non-fibrous matter.

The percentage of the second component (P_{2A} %) is equal to $100 - P_{1A}$ %.

Where a special pre-treatment has been used, the values of b_1 and b_2 should be determined, if possible, by submitting each of the pure fibre constituents to pre-treatment applied in the analysis. Pure fibres are those free from all non-fibrous material except that which they normally contain (either naturally or because of the manufacturing process), in the state (unbleached, bleached) in which they are found in the material to be analysed.

1.9 **Precision of the methods**

The precision indicated in individual methods relates to the reproducibility. This refers to the reliability, i.e. the closeness of agreement between experimental values obtained by operators in different laboratories or at different times, using the same method on specimens of an identical, consistent mixture.

The reproducibility is expressed by confidence limits of the results for a confidence level of 95 %.

By this is meant that the difference between two results in a series of analyses made in different laboratories would be exceeded only in 5 cases out of 100, when the standard method is applied to an identical, consistent mixture.

1.10 Test report

The test report should include the following particulars :

- (a) reference to this ISO Recommendation;
- (b) whether the result relates to the overall composition of the material or to an individual component of the assembly;
- (c) details of any special treatment for the removal of size or finish given in addition to the specified pre-treatment;
- (d) the individual results and the arithmetic mean, each to an accuracy of 0.1;
- (e) whether the result is based on
 - (1) clean dry mass;
 - (2) clean dry mass with percentage additions for moisture, giving the values of the percentage additions;
 - (3) clean dry mass with percentage additions for moisture and loss of fibrous matter caused by the pre-treatment, giving the values of the percentage additions;
 - (4) clean dry mass with percentage additions for moisture and non-fibrous matter, giving the values of the percentage additions.

2.1 Field of application

This method is applicable, after removal of non-fibrous matter, to binary mixtures of acetate with wool, silk, regenerated protein, cotton (scoured, kiered, or bleached), cupro, viscose, modal, polyamide, polyester, acrylic and glass fibres. It is not applicable to mixtures containing modacrylic fibres, nor to mixtures containing viscose fibres that have been deactylated on the surface.

2.2 Principle

The acetate is dissolved out from a known dry mass of the mixture, with acetone. 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 acetate is found by difference.

2.3 Additional reagent

Acetone, distilling between 55 and 57 °C.

2.4 Additional apparatus

Conical flask, minimum capacity 200 ml, glass-stoppered.

2.5 Test procedure

Follow the procedure described in clause 1.7.2 and then proceed as follows :

To the specimen contained in the conical flask (2.4) add 100 ml of acetone (2.3) per gramme of specimen, shake the flask, allow it to stand for 30 minutes at room temperature and then decant the liquid through the weighed filter crucible. Repeat the treatment twice more (making three extractions in all) but for periods of 15 minutes only, so that the total time of treatment in acetone is 1 hour. Wash the residue into the filter crucible with acetone, and drain with suction. Refill the crucible with acetone and allow it to drain under gravity. Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

2.6 Calculation and expression of results

Calculate the results as described in clause 1.8. The value of d is 1.00.

2.7 Precision

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

3. MIXTURES OF CERTAIN PROTEIN AND CERTAIN OTHER FIBRES

3.1 Field of application

This method is applicable, after removal of non-fibrous matter, to binary mixtures of certain non-protein fibres and one protein fibre, as follows :

- wool, chemically treated wool, raw and degummed silk, raw and bleached tussah silk, mohair, cashmere, regenerated protein fibres based on casein,

mixed with :

- cotton, cupro, viscose, modal, acrylic, chlorofibres, polyamide, polyester, polypropylene and glass.

If several protein fibres are present, the method gives the total of their amounts but not their individual quantities.

3.2 Principle

The protein fibre is dissolved out from a known dry mass of the mixture with alkaline sodium hypochlorite. 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 protein fibre is found by difference.

3.3 Additional reagents

3.3.1 Hypochlorite reagent. 1 M sodium hypochlorite solution to which has been added a sufficient quantity of sodium hydroxide to bring the concentration of sodium hydroxide to 5 g/l. The solution may be standardized iodometrically but its concentration is not critical within the range 0.9 M to 1.1 M.

3.3.2 Acetic acid, dilute solution. Dilute 5 ml of glacial acetic acid to 1 litre with water.

3.4 Additional apparatus

Glass beaker, minimum capacity 250 ml.

3.5 Test procedure

Follow the procedure described in clause 1.7.2 and then proceed as follows :

To the specimen contained in the glass beaker (3.4) add 100 ml of hypochlorite reagent (3.3.1) per gramme of specimen, stir vigorously to wet out the specimen and leave for 30 minutes, stirring vigorously at intervals. Filter the contents of the beaker through the weighed filter crucible and transfer any residual fibres to the crucible by washing out the beaker with a little hypochlorite reagent. Drain the crucible with suction and wash the residue successively with water, dilute acetic acid (3.3.2), and finally water, draining the crucible with suction after each addition. (Do not apply suction until each washing liquor has drained under gravity.) Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

3.6 Calculation and expression of results

Calculate the results as described in clause 1.8. The value of d is 1.00 except for raw cotton, for which d = 1.03.

3.7 Precision

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