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ISO

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.

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.

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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 µm.

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 $65 \pm 5^\circ\text{C}$ 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 $105 \pm 3^\circ\text{C}$ 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

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

where

- P_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

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

where

- P_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.