

5088-76

4851903 0038115 0

INTERNATIONAL STANDARD



ISO 5088

H-36-10

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

## Textiles — Ternary fibre mixtures — Quantitative analysis

*Textiles — Mélanges ternaires de fibres — Analyse quantitative*

First edition — 1976-12-15

UDC 677.014.23 : 543.062

Ref. No. ISO 5088-1976 (E)

Descriptors : textiles, fibres, ternary systems, chemical analysis, quantitative analysis, separation

Price based on 13 pages

ISO 5088-1976 (E)

## FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5088 was drawn up by Technical Committee ISO/TC 38, *Textiles*, and was circulated to the Member Bodies in September 1975.

It has been approved by the Member Bodies of the following countries :

Belgium	Hungary	Spain
Bulgaria	India	Sweden
Canada	Israel	Switzerland
Czechoslovakia	Japan	United Kingdom
Denmark	Netherlands	U.S.A.
Egypt, Arab Rep. of	Norway	U.S.S.R.
Finland	Poland	Yugoslavia
France	Romania	
Germany	South Africa, Rep. of	

The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

Italy  
New Zealand

© International Organization for Standardization, 1976 •

Printed in Switzerland

**CONTENTS**

	Page
0 Introduction . . . . .	1
1 Scope . . . . .	2
2 Methods of chemical analysis. . . . .	2
3 Method of analysis by manual separation. . . . .	6
4 Method of analysis by a combination of manual and chemical means . . . .	7
5 Precision of methods . . . . .	7
6 Test report. . . . .	7
<b>Annexes</b>	
A Examples of the calculation of percentages of the components of certain ternary mixtures using some of the variants described in 2.9.1 . . . . .	8
B Table of typical ternary mixtures which may be analysed using methods of analysis of binary mixtures specified in ISO 1833. . . . .	11

# Textiles — Ternary fibre mixtures — Quantitative analysis

## 0 INTRODUCTION

The methods of quantitative analysis of mixtures of textile fibres are based on two processes: the manual separation and the chemical separation of fibre types.

The method of manual separation should be used whenever possible, since it generally gives more accurate results than the chemical method. It can be used for all textiles whose component fibres do not form an intimate mixture, as, for example, in the case of yarns composed of several elements each of which is made up of one type of fibre, or fabrics in which the warp is of a different type of fibre from the weft, or knitted fabrics capable of being unravelled and made up of yarns of different types.

In general, the methods for quantitative chemical analysis of mixtures of textile fibres are based on the selective solution of the individual components of the mixture. Four variants of this procedure are possible:

— Variant 1: Using two different test specimens, component (a) is dissolved from the first test specimen and component (b) from the second test specimen. The insoluble residues of each test specimen are weighed and the percentage of each soluble component is calculated from the respective losses in mass. The percentage of the third component (c) is calculated by difference.

— Variant 2: Using two different test specimens, a component (a) is dissolved from the first test specimen, and two components (a and b) from the second test specimen. The insoluble residue of the first test specimen is weighed and the percentage of the component (a) is calculated from the loss in mass. The insoluble residue of the second test specimen is weighed: it corresponds to component (c). The percentage of the third component (b) is calculated by difference.

— Variant 3: Using two different test specimens, two components (a and b) are dissolved from the first test specimen and two components (b and c) from the second test specimen. The insoluble residues correspond to the two components (c) and (a) respectively. The percentage of the third component (b) is calculated by difference.

— Variant 4: Using only one test specimen, one of the components is removed, after which the insoluble residue formed by the two other fibres is weighed and the percentage of the soluble component is calculated

from the loss in mass. One of the two fibres of the residue is dissolved, the insoluble component is weighed and the percentage of the second soluble component is calculated from the loss in mass.

Where a choice is possible, it is advisable to use one of the first three variants. Where chemical analysis is used, take care to choose methods prescribing solvents which dissolve only the required fibre or fibres, and leave undissolved the other fibre or fibres.

By way of example, a table is given in annex B which contains a certain number of ternary mixtures, together with methods for analysing binary mixtures which can, in principle, be used for analysing these ternary mixtures.

In order to reduce the possibility of error to a minimum, it is recommended that, whenever possible, chemical analysis using at least two of the four above-mentioned variants should be made.

Mixtures of fibres used during processing and, to a lesser extent, in finished textiles may contain non-fibrous matter such as fats, waxes or dressings, or water-soluble matter either occurring naturally or added to facilitate processing. Non-fibrous matter must be removed before analysis. For this reason, a method of pre-treatment for removing oils, fats, waxes and water-soluble matter is also given.

In addition, textiles may contain resins or other matter added to confer special properties. Such matter, including dyestuffs in exceptional cases, may interfere with the action of the reagent on the soluble components and/or it may be partially or completely removed by the reagents. This type of added matter may thus cause errors and should be removed before the sample is analysed. If it is impossible to remove such added matter, the methods for quantitative chemical analysis given in annex B are no longer applicable.

Dye in dyed fibre is considered to be an integral part of the fibre and is not removed.

Analyses are conducted on the basis of dry mass and a procedure is given for its determination.

The result is expressed by reference to the dry mass or by reference to this mass after application of the conventional recovery rate.

Before proceeding with any analysis, all the fibres present in the mixture must be identified. In some chemical methods, the insoluble components of a mixture may be partially dissolved in the reagent used to dissolve the soluble component or components. Whenever possible, reagents have been chosen that have little or no effect on the insoluble fibres. If a loss in mass is known to occur during the analysis, the result should be corrected; correction factors are given for this purpose. These factors have been determined in several laboratories by treating, with 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 before or during processing. If the 4th variant, in which a textile fibre is subjected to the successive action of two different solvents, must be used, correction factors must be applied for possible losses in mass undergone by the fibre in the two treatments.

At least two determinations should be made, both in the case of manual separation and in the case of chemical separation.

## 1 SCOPE

This International Standard specifies methods of quantitative analysis of various ternary mixtures of fibres.

## 2 METHODS OF CHEMICAL ANALYSIS

### 2.1 Field of application

The field of application of each method for analysing binary mixtures, specified in ISO 1833, indicates the fibres to which the method is applicable.

### 2.2 Reference

ISO 1833, *Textiles – Binary fibre mixtures – Quantitative chemical analysis*.

### 2.3 Principle

After identification of the components of a mixture, the non-fibrous matter is removed by a suitable pre-treatment, and then one or more of the four variants of the process of selective solution described in the Introduction is applied. Except where this presents technical difficulties, it is preferable to dissolve the major fibre component so as to obtain the minor fibre component as the final residue.

### 2.4 Reagents

**2.4.1 Light petroleum**, redistilled, boiling range 40 to 60 °C.

Other reagents are specified in the appropriate clauses of ISO 1833. These shall be chemically pure.

### 2.4.2 Distilled or deionized water.

## 2.5 Apparatus

**2.5.1 Filter crucibles and weighing bottles** large enough to contain such crucibles, or any other apparatus giving identical results.

**2.5.2 Vacuum flask.**

**2.5.3 Desiccator**, containing self-indicating silica gel.

**2.5.4 Ventilated oven**, for drying the specimens at  $105 \pm 3$  °C.

**2.5.5 Analytical balance**, accurate to 0,000 2 g.

**2.5.6 Soxhlet extractor apparatus**, or apparatus giving identical results.

## 2.6 Conditioning and testing atmosphere

Because dry masses are determined, it is unnecessary to condition the specimen. The analysis is carried out in the ambient atmosphere.

## 2.7 Sampling and pre-treatment of laboratory test sample

### 2.7.1 Sampling

Take a laboratory test sample that is representative of the laboratory bulk sample and sufficient to provide all the test specimens, each of at least 1 g, that are required.

### 2.7.2 Pre-treatment

Remove the non-fibrous extractable matter in light petroleum (2.4.1) and water, by treating the air-dry sample in the Soxhlet extractor (2.5.6), with light petroleum for 1 h at a minimum rate of 6 cycles per hour. Allow the light petroleum to evaporate from the sample and then soak the sample in cold water (2.4.2) for 1 h, and then in water at  $65 \pm 5$  °C for 1 h, agitating from time to time. Use a liquor ratio of 1:100. Remove the excess water from the sample by squeezing, suction or centrifuging and 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 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.

In the test report, the methods of pre-treatment used should be described in detail.

## 2.8 Procedure

### 2.8.1 General instructions

#### 2.8.1.1 DRYING

Conduct all drying operations for not less than 4 h and not more than 16 h at  $105 \pm 3$  °C in the ventilated oven (2.5.4) with the oven door closed throughout.

If the drying period is less than 14 h, ensure that constant mass has been obtained. This is considered as having been reached if the variation in mass, after further drying for 60 min, is less than 0,05 %. Avoid handling crucibles and weighing bottles, test specimens or residues with the bare hands during the drying, cooling and weighing operations.

Dry the specimen in a weighing bottle (2.5.1) with its cover beside it. After drying, stopper the weighing bottle before removing it from the oven, and transfer it quickly to the desiccator (2.5.3).

Dry the filter crucible (2.5.1) in a weighing bottle with its cover beside it. After drying, stopper the weighing bottle and transfer it quickly to the desiccator.

Where apparatus other than a filter crucible is used, drying operations should be conducted in the oven so as to determine the dry mass of the fibres without loss.

#### 2.8.1.2 COOLING

Conduct all cooling operations in the desiccator, placed beside the balance, until the cooling of the weighing bottles is complete, and in any case for not less than 2 h.

#### 2.8.1.3 WEIGHING

After cooling, complete the weighing of the weighing bottle or crucible within 2 min of its removal from the desiccator. Weigh to an accuracy of 0,000 2 g.

### 2.8.2 Procedure

Take, from the pre-treated laboratory test sample, specimens weighing at least 1 g. Cut yarn or cloth into lengths of about 10 mm, dissected as much as possible. Dry each specimen in a weighing bottle, cool it in the desiccator and weigh it. Transfer the specimen to the glass vessel specified in the appropriate part of ISO 1833, re-weigh the weighing bottle immediately and calculate the dry mass

of the test specimen by difference. Complete the test procedure as specified in the appropriate part of the method used. After weighing, examine the residue microscopically to check that the treatment has in fact completely removed the soluble fibre or fibres.

## 2.9 Calculation and expression of results

Express the mass of each component as a percentage of the total mass of fibre present in the mixture. Calculate the result on the basis of clean dry mass, to which is applied firstly the agreed conventional recovery rate and secondly the correction factor necessary to take account of loss of matter during pre-treatment and analysis.

### 2.9.1 Calculation of percentages of mass of clean dry fibres, disregarding loss of fibre mass during pre-treatment

#### 2.9.1.1 VARIANT 1

Formulae to be applied where a component of the mixture is removed from one specimen and another component from a second specimen :

$$P_1 = \left[ \frac{d_2}{d_1} - d_2 \times \frac{r_1}{m_1} + \frac{r_2}{m_2} \times \left( 1 - \frac{d_2}{d_1} \right) \right] \times 100$$

$$P_2 = \left[ \frac{d_4}{d_3} - d_4 \times \frac{r_2}{m_2} + \frac{r_1}{m_1} \times \left( 1 - \frac{d_4}{d_3} \right) \right] \times 100$$

$$P_3 = 100 - (P_1 + P_2)$$

where

$P_1$  is the percentage of the first clean dry component (component in the first specimen dissolved in the first reagent);

$P_2$  is the percentage of the second clean dry component (component in the second specimen dissolved in the second reagent);

$P_3$  is the percentage of the third clean dry component (component undissolved in both specimens);

$m_1$  is the dry mass of the first specimen after pre-treatment;

$m_2$  is the dry mass of the second specimen after pre-treatment;

$r_1$  is the dry mass of the residue after removal of the first component from the first specimen in the first reagent;

$r_2$  is the dry mass of the residue after removal of the second component from the second specimen in the second reagent;

$d_1$  is the correction factor for loss in mass, in the first reagent, of the second component undissolved in the first specimen<sup>1)</sup>;

$d_2$  is the correction factor for loss in mass, in the first reagent, of the third component undissolved in the first specimen<sup>1)</sup>;

$d_3$  is the correction factor for loss in mass, in the second reagent, of the first component undissolved in the second specimen<sup>1)</sup>;

$d_4$  is the correction factor for loss in mass, in the second reagent, of the third component undissolved in the second specimen<sup>1)</sup>.

### 2.9.1.2 VARIANT 2

Formulae to be applied in the case where a component (a) is removed from the first test specimen, leaving as residue, the other two components (b + c), and the two components (a + b) are removed from the second test specimen, leaving as residue the third component (c).

$$P_1 = 100 - (P_2 + P_3)$$

$$P_2 = 100 \times \frac{d_1 r_1}{m_1} - \frac{d_1}{d_2} \times P_3$$

$$P_3 = \frac{d_4 r_2}{m_2} \times 100$$

where

$P_1$  is the percentage of the first clean dry component (component of the first specimen soluble in the first reagent);

$P_2$  is the percentage of the second clean dry component (component soluble, at the same time as the first component of the second specimen, in the second reagent);

$P_3$  is the percentage of the third clean dry component (component insoluble in both specimens);

$m_1$  is the dry mass of the first specimen after pre-treatment;

$m_2$  is the dry mass of the second specimen after pre-treatment;

$r_1$  is the dry mass of the residue after removal of the first component from the first specimen in the first reagent;

$r_2$  is the dry mass of the residue after removal of the first and second components from the second specimen in the second reagent;

$d_1$  is the correction factor for loss in mass in the first reagent, of the second component undissolved in the first specimen<sup>1)</sup>;

$d_2$  is the correction factor for loss in mass, in the first reagent, of the third component undissolved in the first specimen<sup>1)</sup>;

$d_4$  is the correction factor for loss in mass, in the second reagent, of the third component undissolved in the second specimen<sup>1)</sup>.

### 2.9.1.3 VARIANT 3

Formulae to be applied where two components (a + b) are removed from a specimen, leaving as residue the third component (c), then two components (b + c) are removed from another specimen leaving as residue, the first component (a) :

$$P_1 = \frac{d_3 r_2}{m_2} \times 100$$

$$P_2 = 100 - (P_1 + P_3)$$

$$P_3 = \frac{d_2 r_1}{m_1} \times 100$$

where

$P_1$  is the percentage of the first clean dry component (component dissolved in the first specimen in the first reagent);

$P_2$  is the percentage of the second clean dry component (component dissolved in the first specimen in the first reagent and in the second specimen in the second reagent);

$P_3$  is the percentage of the third clean dry component (component dissolved in the second specimen in the second reagent);

$m_1$  is the dry mass of the first specimen after pre-treatment;

$m_2$  is the dry mass of the second specimen after pre-treatment;

$r_1$  is the dry mass of the residue after removal of the first and second components from the first specimen with the first reagent;

$r_2$  is the dry mass of the residue after removal of the second and third components from the second specimen with the second reagent;

1) The values of  $d$  are indicated in the relevant clauses of ISO 1833.

$d_2$  is the correction factor for loss in mass with the first reagent, of the third component undissolved in the first specimen<sup>1)</sup>;

$d_3$  is the correction factor for loss in mass, with the second reagent, of the first component undissolved in the second specimen<sup>1)</sup>.

#### 2.9.1.4 VARIANT 4

Formulae to be applied where two components are successively removed from the mixture using the same test specimen :

$$P_1 = 100 - (P_2 + P_3)$$

$$P_2 = \frac{d_1 r_1}{m} \times 100 - \frac{d_1}{d_2} \times P_3$$

$$P_3 = \frac{d_3 r_2}{m} \times 100$$

where

$P_1$  is the percentage of the first clean dry component (first soluble component);

$P_2$  is the percentage of the second clean dry component (second soluble component);

$P_3$  is the percentage of the third clean dry component (insoluble component);

$m$  is the dry mass of the test specimen after pre-treatment;

$r_1$  is the dry mass of the residue after removal of the first component by the first reagent;

$r_2$  is the dry mass of the residue after removal of the first and second components by the first and second reagents;

$d_1$  is the correction factor for loss in mass of the second component in the first reagent<sup>1)</sup>;

$d_2$  is the correction factor for loss in mass of the third component in the first reagent<sup>1)</sup>;

$d_3$  is the correction factor for loss in mass of the third component in the first and second reagents<sup>2)</sup>.

**2.9.2 Calculation of the percentage of each component with adjustment by conventional recovery rates and, where appropriate, by correction factors for losses in mass during pre-treatment operations.**

$$A = 1 + \frac{a_1 + b_1}{100} \quad B = 1 + \frac{a_2 + b_2}{100} \quad C = 1 + \frac{a_3 + b_3}{100}$$

hence

$$P_{1A} = \frac{P_1 A}{P_1 A + P_2 B + P_3 C} \times 100$$

$$P_{2A} = \frac{P_2 B}{P_1 A + P_2 B + P_3 C} \times 100$$

$$P_{3A} = \frac{P_3 C}{P_1 A + P_2 B + P_3 C} \times 100$$

where

$P_{1A}$  is the percentage of the first clean dry component, including moisture content and loss in mass during pre-treatment;

$P_{2A}$  is the percentage of the second clean dry component, including moisture content and loss in mass during pre-treatment;

$P_{3A}$  is the percentage of the third clean dry component, including moisture content and loss in mass during pre-treatment;

$P_1$  is the percentage of the first clean dry component obtained by one of the formulae given in 2.9.1;

$P_2$  is the percentage of the second clean dry component obtained by one of the formulae given in 2.9.1;

$P_3$  is the percentage of the third clean dry component obtained by one of the formulae given in 2.9.1;

$a_1$  is the conventional recovery rate of the first component;

$a_2$  is the conventional recovery rate of the second component;

$a_3$  is the conventional recovery rate of the third component;

$b_1$  is the percentage of loss in mass during pre-treatment of the first component;

$b_2$  is the percentage of loss in mass during pre-treatment of the second component;

$b_3$  is the percentage of loss in mass during pre-treatment of the third component.

1) The values of  $d$  are indicated in the relevant clauses of ISO 1833.

2) Wherever possible,  $d_3$  should be determined in advance by experimental methods.



Where a special pre-treatment is used, the values of  $b_1$ ,  $b_2$  and  $b_3$  should be determined if possible, by submitting each of the pure fibre constituents to the 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.

Where no clean separate constituent fibres used in the manufacture of the material to be analysed are available, average values of  $b_1$ ,  $b_2$  and  $b_3$  as obtained from tests performed on clean fibres similar to those in the mixture under examination must be used. If normal pre-treatment by extraction with light petroleum and water is applied, correction factors  $b_1$ ,  $b_2$  and  $b_3$  may generally be ignored except in the case of unbleached cotton, unbleached flax and unbleached hemp where the loss due to pre-treatment is usually accepted as 4 % and in the case of polypropylene where 1 % is admitted.

In the case of other fibres, by convention, no allowance is made in the calculation for the loss during normal pre-treatment.

### 2.9.3 Note

Some examples of calculation are given in annex A.

## 3 METHOD OF ANALYSIS BY MANUAL SEPARATION

### 3.1 Field of application

The method is applicable to textile fibres of all types provided that they do not form an intimate mixture and that it is possible to separate them by hand.

### 3.2 Principle

After identification of the textile components, the non-fibrous matter is removed by a suitable pre-treatment and then the fibres are separated by hand, dried and weighed and the proportion of each fibre in the mixture is calculated.

### 3.3 Reagents

**3.3.1 Light petroleum**, redistilled, boiling range 40 to 60 °C.

**3.3.2 Distilled or deionized water.**

### 3.4 Apparatus

**3.4.1 Weighing bottle**, or other apparatus giving identical results.

**3.4.2 Desiccator**, containing self-indicating silica gel.

**3.4.3 Ventilated oven**, for drying specimens at  $105 \pm 3$  °C.

**3.4.4 Analytical balance**, accurate to 0,000 2 g.

**3.4.5 Soxhlet extractor**, or other apparatus giving identical results.

**3.4.6 Needle.**

**3.4.7 Twist tester**, or other apparatus giving identical results.

### 3.5 Conditioning and testing atmosphere

See 2.6.

### 3.6 Sampling and pre-treatment of laboratory test sample

#### 3.6.1 Sampling

See 2.7.1.

#### 3.6.2 Pre-treatment

See 2.7.2.

### 3.7 Procedure

#### 3.7.1 Analysis of yarn

Take from the pre-treated sample a specimen of mass not less than 1 g. For a very fine yarn, the analysis may be made on a minimum length of 30 m, whatever its mass. Cut the yarn into pieces of suitable length and separate the fibre types by means of the needle (3.4.6) and, if necessary, the twist tester (3.4.7). The fibre types so obtained are placed in the pre-weighed weighing bottles (3.4.1) and dried at  $105 \pm 3$  °C to constant mass as specified in 2.8.1.

#### 3.7.2 Analysis of cloth

Take from the pre-treated sample a specimen of mass at least 1 g (not including selvages), with edges carefully trimmed to avoid fraying and running parallel with weft or warp yarns or, in the case of knitted fabrics, in the line of the wales and courses. Separate the different types of fibre and collect them in the pre-weighed weighing bottles (3.4.1) and proceed as specified in 3.7.1.

### 3.8 Calculation and expression of results

Express the mass of each component fibre as a percentage of the total mass of the fibre in the mixture. Calculate the result on the basis of clean dry mass to which are applied the conventional recovery rate and the correction factor necessary to take account of loss of mass during pre-treatment operations.

### 3.8.1 Calculation of the percentage mass of clean dry fibre disregarding loss in fibre mass during pre-treatment

$$P_1 = \frac{100 m_1}{m_1 + m_2 + m_3} = \frac{100}{1 + \frac{m_2 + m_3}{m_1}}$$

$$P_2 = \frac{100 m_2}{m_1 + m_2 + m_3} = \frac{100}{1 + \frac{m_1 + m_3}{m_2}}$$

$$P_3 = 100 - (P_1 + P_2)$$

where

$P_1$  is the percentage of the first clean dry component;

$P_2$  is the percentage of the second clean dry component;

$P_3$  is the percentage of the third clean dry component;

$m_1$  is the clean dry mass of the first component;

$m_2$  is the clean dry mass of the second component;

$m_3$  is the clean dry mass of the third component.

### 3.8.2 Calculation of the percentage of each component with adjustment by conventional recovery rates and, where appropriate, by correction factors for losses in mass during pre-treatment

See 2.9.2.

## 4 METHOD OF ANALYSIS BY A COMBINATION OF MANUAL SEPARATION AND CHEMICAL MEANS

Wherever possible, manual separation should be used taking account of the proportions of components separated before proceeding to any chemical treatment of each of the separated components.

## 5 PRECISION OF METHODS

The precision indicated in each method of analysis of binary mixtures relates to the reproducibility (see ISO 1833, sub-clause 1.9). This refers to the reliability, that is to say the closeness of agreement between experimental values obtained by operators in different laboratories or at different times using the same method and on specimens of an identical consistent mixture. 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 of a series of analyses made in different laboratories would, given a normal and correct application of the method to an identical and homogeneous mixture, be exceeded only in 5 cases out of 100.

To determine the precision of analysis of a ternary mixture, the values indicated in the methods for analysis of binary mixtures which have been used to analyse the ternary mixture are applied in the usual way.

Given that, in the four variants of the quantitative chemical analysis of ternary mixtures, provision is made for two dissolutions (using two separate specimens for the first three variants and a single specimen for the fourth variant) and, assuming that  $E_1$  and  $E_2$  denote the respective precisions of the two methods for analysing binary mixtures, the precision of the results for each component is shown in the following table :

Component fibres \ Variants	1	2 and 3	4
a	$E_1$	$E_1$	$E_1$
b	$E_2$	$E_1 + E_2$	$E_1 + E_2$
c	$E_1 + E_2$	$E_2$	$E_1 + E_2$

If the fourth variant is used, the precision may be found to be lower than that calculated by the method indicated above, owing to possible action of the first reagent on the residue consisting of components (b) and (c), which would be difficult to evaluate.

## 6 TEST REPORT

The test report shall include the following particulars :

- the variant or variants used to carry out the analysis, the methods, reagents, and correction factors;
- the detailed procedure of any special pre-treatment (see 2.7.2);
- the individual results and the arithmetic mean of the results to the first decimal place;
- wherever possible, the precision of the method for each component calculated as shown in the table in clause 5.

## ANNEX A

**EXAMPLES OF THE CALCULATION OF PERCENTAGES OF THE COMPONENTS OF CERTAIN TERNARY MIXTURES USING SOME OF THE VARIANTS DESCRIBED IN 2.9.1.**

**A.1 VARIANT 1**

Consider the case of a fibre mixture which, when qualitatively analysed, gave the following components : carded wool, polyamide, unbleached cotton.

Suppose that, using this variant, i.e. using two different specimens and removing one component (a = wool) by dissolution from the first test specimen and a second component (b = polyamide) from the second test specimen, the following results are obtained :

- |   |                            |
|---|----------------------------|
| 1. Dry mass of the first specimen after pre-treatment :   | $m_1 = 1,600\ 0\ \text{g}$ |
| 2. Dry mass of the residue after treatment with alkaline sodium hypochlorite (polyamide + cotton) : | $r_1 = 1,416\ 6\ \text{g}$ |
| 3. Dry mass of the second specimen after pre-treatment :  | $m_2 = 1,800\ 0\ \text{g}$ |
| 4. Dry mass of the residue after treatment with formic acid (wool + cotton) :                       | $r_2 = 0,900\ 0\ \text{g}$ |

Treatment with alkaline sodium hypochlorite does not entail any loss in mass of polyamide, while bleached cotton loses 3 %, therefore  $d_1 = 1,0$  and  $d_2 = 1,03$ .

Treatment in formic acid does not entail any loss in mass of wool or unbleached cotton, therefore  $d_3 = 1,0$  and  $d_4 = 1,0$ .

**A.1.1 Dry masses**

If the values obtained by chemical analysis and the correction factors are substituted in the formula indicated in 2.9.1.1, the following result is obtained :

$$P_1 (\text{wool}) = \left[ \frac{1,03}{1,0} - 1,03 \times \frac{1,416\ 6}{1,600\ 0} + \frac{0,900\ 0}{1,800\ 0} \times \left( 1 - \frac{1,03}{1,0} \right) \right] \times 100 = 10,30$$

$$P_2 (\text{polyamide}) = \left[ \frac{1,0}{1,0} - 1,0 \times \frac{0,900\ 0}{1,800\ 0} \times \frac{1,416\ 6}{1,600\ 0} \times \left( 1 - \frac{1,0}{1,0} \right) \right] \times 100 = 50,00$$

$$P_3 (\text{cotton}) = 100 - (10,30 + 50,00) = 39,70$$

The percentages of the various clean dry fibres in the mixture are as follows :

wool	10,30 %
polyamide	50,00 %
cotton	39,70 %

**A.1.2 Masses after application of allowance for moisture**

The percentages shall be corrected according to the formulae in 2.9.2 in order to take account also of the conventional recovery rate and the correction factors for any losses in mass after pre-treatment.

Supposing that the bleached cotton sustains a loss in mass of 4 % after pre-treatment in petroleum ether and water, and that for example the conventional recovery rate to be applied is 17 % for wool, 6,25 % for polyamide and 8,5 % for cotton, one obtains :

$$P_{1A} \text{ (wool)} = \frac{10,30 \times \left(1 + \frac{17,0 + 0,0}{100}\right)}{10,30 \times \left(1 + \frac{17,00 + 0,0}{100}\right) + 50,00 \times \left(1 + \frac{6,25 + 0,0}{100}\right) + 39,70 \times \left(1 + \frac{8,5 + 4,0}{100}\right)} \times 100 = 10,97$$

$$P_{2A} \text{ (polyamide)} = \frac{50,00 \times \left(1 + \frac{6,25 + 0,0}{100}\right)}{109,8385} \times 100 = 48,37$$

$$P_{3A} \text{ (cotton)} = 100 - (10,97 + 48,37) = 40,66$$

The composition of the mixture is thus as follows :

wool	11,0 %
polyamide	48,4 %
cotton	40,6 %
	<u>100,0 %</u>

## A.2 VARIANT 4

Consider the case of a fibre mixture which, when qualitatively analysed, gave the following components : carded wool, viscose, unbleached cotton.

Supposing that, using variant 4, i.e. successively removing two components from the mixture of one single test specimen, the following results are obtained :

1. Dry mass of test specimen after pre-treatment :  $m_1 = 1,6000 \text{ g}$
2. Dry mass of test specimen after the first treatment with alkaline sodium hypochlorite (viscose + cotton) :  $r_1 = 1,4166 \text{ g}$
3. Dry mass of residue after the second treatment of the residue  $r_1$  with formic acid/zinc chloride (cotton) :  $r_2 = 0,6630 \text{ g}$

Treatment with alkaline sodium hypochlorite does not entail any loss of mass in viscose, while unbleached cotton loses 3 %, therefore  $d_1 = 1,0$  and  $d_2 = 1,03$ . After treatment in formic acid/zinc chloride, the mass of cotton decreases by 2 % so that  $d_3 = (1,03 \times 1,02) = 1,0506$  rounded to 1,05 ( $d_3$  being the correction factor for the respective loss or increase in mass of the third component in the first and second reagent).

### A.2.1 Dry masses

Using the formulae indicated in 2.9.1.4, the values obtained by chemical analysis and the correction factors are substituted and the following result is obtained :

$$P_2 \text{ (viscose)} = \frac{1,0 \times 1,4166}{1,6000} \times 100 - \frac{1,00}{1,03} \times 43,51 = 46,32 \%$$

$$P_3 \text{ (cotton)} = \frac{1,05 \times 0,6630}{1,6000} \times 100 = 43,51 \%$$

$$P_1 \text{ (wool)} = 100 - (46,32 + 43,51) = 10,17 \%$$

**A.2.2 Mass after application of conventional recovery rates**

As has already been indicated for variant 1, the percentages must be corrected by the formulae in 2.9.2. Taking the same values as above, one has :

$$P_{1A} (\text{wool}) = \frac{10,17 \times \left(1 + \frac{17,0 + 0,0}{100}\right)}{10,17 \times \left(1 + \frac{17,0 + 0,0}{100}\right) + 46,32 \times \left(1 + \frac{13,0 + 0,0}{100}\right) + 43,51 \times \left(1 + \frac{8,5 + 4,0}{100}\right)} \times 100 = 10,51 \%$$

$$P_{2A} (\text{viscose}) = \frac{46,32 \times \left(1 + \frac{13,0 + 0,0}{100}\right)}{113,21} \times 100 = 46,24 \%$$

$$P_{3A} (\text{cotton}) = 100 - (10,51 + 46,24) = 43,25 \%$$

The composition of the mixture is thus :

wool	10,5 %
viscose	46,2 %
cotton	43,3 %
	<u>100,0 %</u>

## ANNEX B

TABLE OF TYPICAL TERNARY MIXTURES WHICH MAY BE ANALYSED USING METHODS  
OF ANALYSIS OF BINARY MIXTURES SPECIFIED IN ISO 1833  
(for illustration purposes)

Mixture No.	Component fibres			Variant No. *	Corresponding clause of ISO 1833 (showing reagents used)
	1st component	2nd component	3rd component		
1	wool or hair	viscose, cupro or certain types of modal	cotton	1 and/or 4	3 (alkaline sodium hypochlorite) and 5 (zinc chloride/formic acid)
2	wool or hair	polyamide 6 or 6.6	cotton, viscose, cupro or modal	1 and/or 4	3 (alkaline sodium hypochlorite) and 6 (formic acid 80 % (m/m))
3	wool, hair or silk	certain chlorofibres	cotton, viscose, cupro or modal	1 and/or 4	3 (alkaline sodium hypochlorite) and 12 (carbon disulphide/acetone 55,5/44,5 (V/V))
4	wool or hair	polyamide 6 or 6.6	polyester, polypropylene, acrylic or glass fibre	1 and/or 4	3 (alkaline sodium hypochlorite) and 6 (formic acid 80 % (m/m))
5	wool, hair or silk	certain chlorofibres	polyester, acrylic, polyamide or glass fibre	1 and/or 4	3 (alkaline sodium hypochlorite) and 12 (carbon disulphide/acetone 55,5/44,5 (V/V))
6	silk	wool or hair	polyester	2	17** (sulphuric acid 75 % (m/m)) and 3 (alkaline sodium hypochlorite)
7	polyamide 6 or 6.6	acrylic	cotton, viscose, cupro or modal	1 and/or 4	6 (formic acid 80 % (m/m)) and 11 (dimethylformamide)
8	certain chlorofibres	polyamide 6 or 6.6	cotton, viscose, cupro or modal	1 and/or 4	11 (dimethylformamide) and 6 (formic acid 80 % (m/m)) or 12 (carbon disulphide/acetone 55,5/44,5 (V/V)) and 6 (formic acid 80 % (m/m))
9	acrylic	polyamide 6 or 6.6	polyester	1 and/or 4	11 (dimethylformamide) and 6 (formic acid 80 % (m/m))
10	acetate	polyamide 6 or 6.6	cotton, viscose, cupro or modal	4	2 (acetone) and 6 (formic acid 80 % (m/m))
11	certain chlorofibres	acrylic	polyamide	2 and/or 4	12 (carbon disulphide/acetone 55,5/44,5 (V/V)) and 11 (dimethylformamide)
12	certain chlorofibres	polyamide 6 or 6.6	acrylic	1 and/or 4	12 (carbon disulphide/acetone 55,5/44,5 (V/V)) and 6 (formic acid 80 % (m/m))
13	polyamide 6 or 6.6	cotton, viscose, cupro or modal	polyester	4	6 (formic acid 80 % (m/m)) and 10 (sulphuric acid 75 % (m/m))
14	acetate	cotton, viscose, cupro or modal	polyester	4	2 (acetone) and 10 (sulphuric acid 75 % (m/m))

\* When variant 4 is used, first remove the first component using the first reagent.

\*\* See Addendum 1 to ISO 1833.

TABLE (continued)

Mixture No.	Component fibres			Variant No.*	Corresponding clause of ISO 1833 (showing reagents used)
	1st component	2nd component	3rd component		
15	acrylic	cotton, viscose, cupro or modal	polyester	4	11 (dimethylformamide) and 10 (sulphuric acid 75 % (m/m))
16	acetate	wool, hair or silk	cotton, viscose, cupro, modal, polyamide, polyester, acrylic	4	2 (acetone) and 3 (alkaline sodium hypochlorite)
17	triacetate	wool, hair or silk	cotton, viscose, cupro, modal, polyamide, polyester, acrylic	4	9 (dichloromethane) and 3 (alkaline sodium hypochlorite)
18	acrylic	wool, hair or silk	polyester	1 and/or 4	11 (dimethylformamide) and 3 (alkaline sodium hypochlorite)
19	acrylic	silk	wool or hair	4	11 (dimethylformamide) and 17** (sulphuric acid 75 % (m/m))
20	acrylic	wool, hair or silk	cotton, viscose, cupro or modal	1 and/or 4	11 (dimethylformamide) and 3 (alkaline sodium hypochlorite)
21	wool, hair or silk	cotton, viscose, cupro or modal	polyester	4	3 (alkaline sodium hypochlorite) and 10 (sulphuric acid 75 % (m/m))
22	viscose, cupro or certain types of modal	cotton	polyester	2 and/or 4	5 (zinc chloride/formic acid) and 10 (sulphuric acid 75 % (m/m))
23	acrylic	viscose, cupro or certain types of modal	cotton	4	11 (dimethylformamide) and 5 (zinc chloride/formic acid)
24	certain chlorofibres	viscose, cupro or certain types of modal	cotton	1 and/or 4	12 (carbon disulphide/acetone 55,5/44,5 (V/V)) and 5 (zinc chloride/formic acid) or 11 (dimethylformamide) and 5 (zinc chloride/formic acid)
25	acetate	viscose, cupro or certain types of modal	cotton	4	2 (acetone) and 15 (zinc chloride/formic acid)
26	triacetate	viscose, cupro or certain types of modal	cotton	4	9 (dichloromethane) and 5 (zinc chloride/formic acid)
27	acetate	silk	wool or hair	4	7 (acetone 70 % (V/V)) and 17** (sulphuric acid 75 % (m/m))
28	triacetate	silk	wool or hair	4	9 (dichloromethane) and 17** (sulphuric acid 75 % (m/m))
29	acetate	acrylic	cotton, viscose, cupro or modal	4	2 (acetone) and 11 (dimethylformamide)
30	triacetate	acrylic	cotton, viscose, cupro or modal	4	9 (dichloromethane) and 11 (dimethylformamide)

\* When variant 4 is used, first remove the first component using the first reagent.

\*\* See Addendum 1 to ISO 1833.

TABLE (concluded)

Mixture No.	Component fibres			Variant No.*	Corresponding clause of ISO 1833 (showing reagents used)
	1st component	2nd component	3rd component		
31	triacetate	polyamide 6 or 6.6	cotton, viscose, cupro or modal	4	9 (dichloromethane) and 6 (formic acid 80 % (m/m))
32	triacetate	cotton, viscose, cupro or modal	polyester	4	9 (dichloromethane) and 10 (sulphuric acid 75 % (m/m))
33	acetate	polyamide 6 or 6.6	polyester or acrylic	4	2 (acetone) and 6 (formic acid 80 % (m/m))
34	acetate	acrylic	polyester	4	2 (acetone) and 11 (dimethylformamide)
35	certain chlorofibres	cotton, viscose, cupro or modal	polyester	4	11 (dimethylformamide) and 10 (sulphuric acid 75 % (m/m)) or 12 (carbon disulphide/acetone 55,5/44,5 (V/V)) and 10 (sulphuric acid 75 % (m/m))

\* When variant 4 is used, first remove the first component using the first reagent.