

## II

*(Acts whose publication is not obligatory)*

## COUNCIL

## COUNCIL DIRECTIVE

of 26 February 1973

on the approximation of the laws of the Member States relating to the quantitative analysis of ternary fibre mixtures

(73/44/EEC)

THE COUNCIL OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community, and in particular Article 100 thereof;

Having regard to the proposal from the Commission;

Whereas the Council Directive of 26 July 1971 <sup>(1)</sup> on the approximation of the laws of the Member States relating to textile names lays down provisions on labelling based on the fibre composition of textile products;

Whereas the methods used for official tests in the Member States to determine the fibre composition of textile products should be uniform, as regards both the pre-treatment of the sample and the quantitative analysis of the product;

Whereas Article 13 of the abovementioned Council Directive provides that the sampling and analytical methods to be used in all Member States for the purpose of determining the fibre composition of products will be specified in separate Directives; whereas under these circumstances, the Council

adopted in its Directive of 17 July 1972 <sup>(2)</sup> on the approximation of the laws of the Member States relating to certain methods for the quantitative analysis of binary fibre mixtures, provisions concerning the preparation of test samples and test specimens which are applicable to ternary fibre mixtures;

Whereas the object of this Directive is to lay down provisions governing the quantitative analysis of ternary fibre mixtures;

Whereas the particular methods relating to the analysis of certain binary mixtures are described in detail in the Directive of 17 July 1972; whereas present experience does not permit the specification of a single standard procedure as yet; whereas several variants must be proposed for the selective dissolution of the components;

Whereas general rules valid for the analysis of all ternary mixtures must however be worked out; whereas the object of these rules is to specify the various methods which could suitably be applied and the method of calculating the percentage composition for each variant;

Whereas technical specifications must be adapted rapidly to keep pace with technical progress; whereas to this end the procedure laid down in Article 6 of the Directive of 17 July 1972 should be applied;

<sup>(1)</sup> OJ No L 185, 16. 8. 1971, p. 16.

<sup>(2)</sup> OJ No L 173, 31. 7. 1972, p. 1.

HAS ADOPTED THIS DIRECTIVE:

*Article 1*

This Directive concerns the quantitative analysis of ternary fibre mixtures by the methods of manual separation, chemical separation or a combination of both.

*Article 2*

With regard to the preparation of test samples and test specimens, the provisions contained in Annex I to the Council Directive of 17 July 1972 on the approximation of the laws of the Member States relating to certain methods for the quantitative analysis of binary fibre mixtures shall be applicable.

*Article 3*

The Member States shall take all necessary steps to ensure that the provisions laid down — both in Annex I to this Directive and in Annex I to the Directive referred to in Article 2 — be applied in official tests, to determine the composition of textile products composed of a ternary fibre mixture put on the market in accordance with the Council Directive of 26 July 1971 on the approximation of the laws of the Member States relating to textile names.

*Article 4*

Any laboratory responsible for the testing of ternary mixtures shall show in the test report all the factors mentioned in point V of Annex I.

*Article 5*

Any amendments to the specifications in Annexes I, II and III that are necessary to conform with technical progress shall be adopted in accordance with the procedure laid down in Article 6 of the Directive of 17 July 1972.

*Article 6*

1. Member States shall put into force the provisions necessary to comply with this Directive within a period of eighteen months of its notification and shall immediately inform the Commission thereof.

2. Member States shall ensure that the text of the main provisions of internal law adopted by them in the field governed by this Directive are communicated to the Commission.

*Article 7*

This Directive is addressed to the Member States.

Done at Brussels, 26 February 1973.

*For the Council*

*The President*

E. GLINNE

## ANNEX I

## QUANTITATIVE ANALYSIS OF TERNARY FIBRE MIXTURES

## GENERAL

**Introduction**

Methods for the quantitative analysis of fibre mixtures 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 only 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 made up of yarns of different types.

In general, the methods of quantitative chemical analysis are based on the selective solution of the individual components. There are four possible variants of this method:

1. Using two different test specimens, a component (a) is dissolved from the first test specimen, and another component (b) from the second test specimen. The insoluble residues of each specimen are weighed and the percentage of each of the two soluble components is calculated from the respective losses in mass. The percentage of the third component (c) is calculated by difference.
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.
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.
4. Using only one test specimen, after removal of one of the components, 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, the expert responsible for the analysis must take care to select methods employing solvents which dissolve only the correct fibre(s), leaving the other fibre(s) intact.

By way of example, a table is given in Annex III 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 abovementioned 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 III attached are no longer applicable.

Dye in dyed fibres 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 determining dry mass.

The result is obtained by applying to the dry mass of each fibre the recovery rates listed in Annex II to the Directive on the approximation of the laws of the Member States relating to textile names.

Before proceeding with any analysis, all the fibres present in the mixture must be identified. In some chemical methods, the insoluble component of a mixture may be partially dissolved in the reagent used to dissolve the soluble component(s). Wherever 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 undergraded fibres and different correction factors may be necessary if the fibres have been degraded before or during processing. If the fourth 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.

## I. GENERAL INFORMATION ON METHODS FOR THE QUANTITATIVE CHEMICAL ANALYSIS OF TERNARY FIBRE MIXTURES

Information common to the methods given for the quantitative chemical analysis of ternary fibre mixtures.

### I.1. *Scope and field of application*

The field of application of each method for analysing binary fibre mixtures specifies to which fibres the method is applicable. (See Annex II, Directive relating to certain methods for the quantitative analysis of binary fibre mixtures).

### I.2. *Principle*

After the identification of the components of a mixture, the non-fibrous material is removed by 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 final residue.

I.3. *Apparatus and Reagents*

I.3.1. *Apparatus*

- I.3.1.1. Filter crucibles and weighing bottles large enough to contain such crucibles, or any other apparatus giving identical results.
- I.3.1.2. Vacuum flask.
- I.3.1.3. Desiccator containing self-indicating silica gel.
- I.3.1.4. Ventilated oven for drying specimens at  $105 \pm 3$  °C.
- I.3.1.5. Analytical balance, accurate to 0.0002 g.
- I.3.1.6. Soxhlet extractor or other apparatus giving identical results.

I.3.2. *Reagents*

- I.3.2.1. Light petroleum, redistilled, boiling range 40 to 60 °C.
- I.3.2.2. Other reagents are specified in the appropriate parts of the text of the method. All reagents used should be chemically pure.
- I.3.2.3. Distilled or deionized water.

I.4. *Conditioning and testing atmosphere*

Because dry masses are determined, it is unnecessary to condition the specimens or to conduct analyses in a conditioned atmosphere.

I.5. *Laboratory test sample*

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.

I.6. *Pre-treatment of the laboratory test sample*

Where a substance not to be taken into account in the percentage calculations (See Article 12 (2) (d) of the Directive relating to textile names) is present, it should first be removed by a suitable method that does not affect any of the component fibres.

For this purpose, non-fibrous matter which can be extracted with light petroleum and water is removed by treating the air-dry test sample in a Soxhlet extract selector with light petroleum for one hour at a minimum rate of 6 cycles per hour. Allow the light petroleum to evaporate from the sample which is then directly extracted with water by soaking for one hour at room temperature followed by soaking at  $65 \pm 5$  °C for a further one hour, agitating the liquid from time to time, specimen/water ratio 1 : 100. 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 substituting for the water method described above a suitable method that does not substantially alter any of the fibre constituents. However for some unbleached natural vegetable fibres (eg 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 contains finishes insoluble in both light petroleum and water.

Analytical reports should include full details of the methods of pre-treatment used.

I.7. *Test procedure*

I.7.1. *General instructions*

I.7.1.1. *Drying*

Conduct all drying operations for not less than 4 hours and not more than 16 hours at  $105 \pm 3$  °C in a ventilated oven with the oven door closed throughout. If the drying period is less than 14 hours, the specimen must be checkweighed to determine whether its mass is constant. The mass may be considered as constant if, after a further drying period of 60 minutes, its variation is less than 0.05 %.

Avoid handling crucibles and weighing bottles, specimens or residues with bare hands during the drying, cooling and weighing operations.

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

Dry the filter crucible in a weighing bottle with its cover beside it in the oven. After drying, close 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.

I.7.1.2. *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 hours.

I.7.1.3. *Weighing*

After cooling, complete the weighing of the weighing bottle within 2 minutes of its removal from the desiccator; weigh to an accuracy of 0.0002 g.

I.7.2. *Procedure*

Take from the pre-treated laboratory test sample a test specimen of at least 1 g (in mass). Cut yarn or cloth into lengths of about 10 mm, dissected as much as possible. Dry the specimen(s) in (a) weighing bottle(s) cool it (them) in the desiccator and weigh it (them). Transfer the specimen(s) to the glass vessel(s) specified in the appropriate section of the Community method, reweigh the weighing bottle(s) immediately and obtain the dry mass(es) of the specimen(s) by difference; complete the test as specified in the appropriate section of the applicable method. Examine the residue(s) microscopically to check that the treatment has in fact completely removed the soluble fibre(s).

I.8. *Calculation and expression of results*

Express the mass of each component as a percentage of the total mass of fibre in the mixture. Calculate the results on the basis of clean dry mass, adjusted by (a) the conventional recovery rates and (b) the correction factors necessary to take account of loss of non-fibrous matter during pre-treatment and analysis.

I.8.1. Calculation of percentages of mass of clean dry fibres disregarding loss of fibre mass during pre-treatment.

## I.8.1.1. — VARIANT 1 —

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

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

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

$$P_3\% = 100 - (P_1\% + P_2\%);$$

$P_1\%$  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>.

## I.8.1.2. — VARIANT 2 —

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

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

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

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

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

<sup>(1)</sup> The values of d are indicated in the corresponding sections of the Directives relating to the various methods of analysing binary mixtures.

- $P_2\%$  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>.

#### I.8.1.3. — 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):

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

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

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

- $P_1\%$  is the percentage of the first clean dry component (component dissolved by the reagent);
- $P_2\%$  is the percentage of the second clean dry component (component dissolved by the reagent);
- $P_3\%$  is the percentage of the third clean dry component (component dissolved in the second specimen by the 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;
- $d_2$  is the correction factor for loss in mass with the first reagent of the third component undissolved in the first specimen <sup>(1)</sup>;

<sup>(1)</sup> The values of d are indicated in the corresponding sections of the Directives relating to the various methods of analysing binary mixtures.



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

#### I.8.1.4. — VARIANT 4 —

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

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

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

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

$P_1\%$  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 specimen after pre-treatment;

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

$r_2$  is the dry mass of the residue after elimination 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>.

I.8.2. Calculation of the percentage of each component with adjustment by conventional recovery rates and, where appropriate, correction factors for losses in mass during pre-treatment operations:

Given:

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

then:

$$P_{1A}\% = \frac{P_1A}{P_1A + P_2B + P_3C} \times 100$$

$$P_{2A}\% = \frac{P_2B}{P_1A + P_2B + P_3C} \times 100$$

$$P_{3A}\% = \frac{P_3C}{P_1A + P_2B + P_3C} \times 100$$

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

<sup>(1)</sup> The values of  $d$  are indicated in the corresponding sections of the Directives relating to the various methods of analysing binary mixtures.

<sup>(2)</sup> Wherever possible  $d_3$  should be determined in advance by experimental methods.

- $P_{2A}\%$  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 I.8.1;
- $P_2$  is the percentage of the second clean dry component obtained by one of the formulae given in I.8.1;
- $P_3$  is the percentage of the third clean dry component obtained by one of the formulae given in I.8.1;
- $a_1$  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 of the first component during pre-treatment;
- $b_2$  is the percentage of loss in mass of the second component during pre-treatment;
- $b_3$  is the percentage of loss in mass of the third component during pre-treatment.

Where a special pre-treatment is used the values  $b_1$ ,  $b_2$  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 those 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 as 1 %.

In the case of other fibres, losses due to pre-treatment are usually disregarded in calculations.

### I.8.3. *Note*

Calculation examples are given in Annex II to this Directive.

## II. *METHOD OF QUANTITATIVE ANALYSIS BY MANUAL SEPARATION OF TERNARY FIBRE MIXTURES*

### II.1. *Scope*

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

### II.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 in order to calculate the proportion of each fibre in the mixture.

### II.3. *Apparatus*

- II.3.1. Weighing bottles or other apparatus giving identical results.
- II.3.2. Desiccator containing self-indicating silica gel.
- II.3.3. Ventilated oven for drying specimens at  $105 \pm 3$  °C.
- II.3.4. Analytical balance accurate to 0.0002 g.
- II.3.5. Soxhlet extractor, or other apparatus giving identical results.
- II.3.6. Needle.
- II.3.7. Twist tester or similar apparatus.

### II.4. *Reagents*

- II.4.1. Light petroleum, redistilled, boiling range 40°—60 °C.
- II.4.2. Distilled or deionized water.

### II.5. *Conditioning and testing atmosphere*

See I.4.

### II.6. *Laboratory test sample*

See I.5.

### II.7. *Pre-treatment of laboratory test samples*

See I.6.

### II.8. *Procedure*

#### II.8.1. *Analysis of yarn*

Take from the pre-treated laboratory test 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 a suitable length and separate the fibre types by means of a needle and, if necessary, a twist tester. The fibre types so obtained are placed in pre-weighed weighing bottles and dried at  $105 \pm 3$  °C to constant mass, as described in I.7.1 and I.7.2.

#### II.8.2. *Analysis of cloth*

Take from the pre-treated laboratory test sample a specimen of mass not less than 1 g, not including a selvage 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 fibres and collect them in pre-weighed weighing bottles and proceed as described in II.8.1.

### II.9. *Calculation and expression of results*

Express the mass of each component fibre as a percentage of the total mass of the fibres in the mixture. Calculate the results on the basis of clean dry mass, adjusted by (a) the conventional recovery rates and (b) the correction factors necessary to take account of losses in mass during pre-treatment operations.

- II.9.1. Calculation of percentage masses of clean dry fibre, disregarding loss in fibre mass during pre-treatment:

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

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

$$P_3\% = 100 - (P_1\% + P_2\%);$$

$P_1\%$  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.

- II.9.2. For 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 I.8.2.

### III. *METHOD OF QUANTITATIVE ANALYSIS OF TERNARY FIBRE MIXTURES BY A COMBINATION OF MANUAL SEPARATION AND CHEMICAL SEPARATION*

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 separate components.

### IV. *PRECISION OF THE METHODS*

The precision indicated in each method of analysis of binary mixtures relates to the reproducibility (see Annex II of the Directive relating to certain methods for the quantitative analysis of binary textile fibre mixtures).

Reproducibility 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 and obtaining individual results on specimens of an identical homogeneous 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 in 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 the analysis of a ternary mixture the values indicated in the methods for the 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 precision of the two methods for analysing binary mixtures, the precision of the results for each component is shown in the following table:

Component fibre	Variants	1	2 and 3	4
	a		$E_1$	$E_1$
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 degree of 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.

#### V. TEST REPORT

- V.1. Indicate the variant(s) used to carry out the analysis, the methods, reagents and correction factors.
- V.2. Give details of any special pre-treatments (See I.6).
- V.3. Give the individual results and the arithmetic mean, each to the 1st decimal place.
- V.4. Wherever possible, state the precision of the method for each component, calculated according to the table in section IV.

## ANNEX II

EXAMPLES OF THE CALCULATION OF PERCENTAGES OF THE COMPONENTS OF CERTAIN TERNARY MIXTURES USING SOME OF THE VARIANTS DESCRIBED IN POINT I.8.1 OF ANNEX I

Consider the case of a fibre mixture which when qualitatively analysed gave the following components: 1. carded wool; 2. nylon (polyamide); 3. unbleached cotton.

## VARIANT No 1

Using this variant, that is using two different specimens and removing one component (a = wool) by dissolution from the first specimen and a second component (b = polyamide) from the second specimen the following results can be obtained:

1. Dry mass of the first specimen after pre-treatment  $(m_1) = 1.6000 \text{ g}$
2. Dry mass of the residue after pre-treatment with alkaline sodium hypochlorite (polyamide + cotton)  $(r_1) = 1.4166 \text{ g}$
3. Dry mass of the second specimen after pre-treatment  $(m_2) = 1.8000 \text{ g}$
4. Dry mass of the residue after treatment with formic acid (wool + cotton)  $(r_2) = 0.9000 \text{ g}$

Treatment with alkaline sodium hypochlorite does not entail any loss in mass of polyamide, while unbleached cotton loses 3 %, therefore  $d_1 = 1.0$  and  $d_2 = 1.03$ .

Treatment with formic acid does not entail any loss in mass of wool or unbleached cotton, therefore  $d_3$  and  $d_4 = 1.0$ .

If the values obtained by chemical analysis and the correction factors are substituted in the formula under point I.8.1.1 of Annex I, the following result is obtained:

$$P_1\% (\text{wool}) = \left[ \frac{1.03}{1.0} - 1.03 \times \frac{1.4166}{1.6000} + \frac{0.9000}{1.8000} \times \left( 1 - \frac{1.03}{1.0} \right) \right] \times 100 = 10.30$$

$$P_2\% (\text{polyamide}) = \left[ \frac{1.0}{1.0} - 1.0 \times \frac{0.9000}{1.8000} + \frac{1.4166}{1.6000} \times \left( 1 - \frac{1.0}{1.0} \right) \right] \times 100 = 50.00$$

$$P_3\% (\text{cotton}) = 100 - (10.30 + 50.00) = 39.70$$

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

Wool	10.30 %
Polyamide	50.00 %
Cotton	39.70 %

These percentages must be corrected according to the formulae under point I.8.2 of Annex I in order to take account also of the conventional recovery rates and of the correction factors for any losses in mass after pre-treatment.

As indicated in Annex II to the Directive relating to textile names, the conventional recovery rates are as follows: carded wool 17.0 %, polyamide 6.25 %, cotton 8.5 %, also, unbleached cotton shows a loss in mass of 4 % after pre-treatment with light petroleum and water. Thus:

$$P_{1A}\% \text{ (wool)} = \frac{10.30 \times \left(1 + \frac{17.0 + 0.0}{100}\right)}{10.30 \times \left(1 + \frac{17.0 + 0.0}{100}\right) + 50.00 \times \left(1 + \frac{6.25 + 0.0}{100}\right) + 39.70 \times \left(1 + \frac{8.5 + 4.0}{100}\right)} \times 100 = 10.97$$

$$P_{2A}\% \text{ (polyamide)} = \frac{50.00 \times \left(1 + \frac{6.25 + 0.0}{100}\right)}{109.8385} \times 100 = 48.37$$

$$P_{3A}\% \text{ (cotton)} = 100 - (10.97 + 48.37) = 40.66.$$

The composition of the yarn is therefore as follows:

Polyamide	48.4 %
Cotton	40.6 %
Wool	11.0 %
	<u>100.0 %</u>

#### VARIANT 4

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

Suppose that using variant 4, that is successively removing two components from the mixture of one single specimen, the following results are obtained:

1. Dry mass of the specimen after pre-treatment (m<sub>1</sub>) = 1.6000 g
2. Dry mass of the residue after the first treatment with alkaline sodium hypochlorite (viscose + cotton): (r<sub>1</sub>) = 1.4166 g
3. Dry mass of the residue after the second treatment of the residue r<sub>1</sub> with zinc chloride/formic acid (cotton): (r<sub>2</sub>) = 0.6630 g

Treatment with alkaline sodium hypochlorite does not entail any loss of mass in viscose, while unbleached cotton loses 3 %, therefore d<sub>1</sub> = 1.0 and d<sub>2</sub> = 1.03.

As a result of treatment with zinc chloride/formic acid, the mass of cotton increases by 4 %, so that d<sub>3</sub> = (1.03 × 0.96) = 0.9888 rounded to 0.99, (d<sub>3</sub> being the correction factor for the respective loss or increase in mass of the third component in the first and second reagents).

If the values obtained by chemical analysis and the correction factors are substituted in the formulae given in point I.8.1.4 of Annex I the following result is obtained:

$$P_2\% \text{ (viscose)} = \frac{1.0 \times 1.4166}{1.6000} \times 100 - \frac{1.0}{1.03} \times 40.98 = 48.75 \%$$

$$P_3\% \text{ (cotton)} = \frac{0.99 \times 0.6630}{1.6000} \times 100 = 41.02 \%$$

$$P_1\% \text{ (wool)} = 100 - (48.75 + 41.02) = 10.23 \%$$

As has already been indicated for variant 1, these percentages must be corrected by the formulae indicated in point I.8.2 of Annex I.

$$P_{1A} \% (\text{wool}) = \frac{10.23 \times \left(1 + \frac{17.00 + 0.0}{100}\right)}{10.23 \times \left(1 + \frac{17.00 + 0.0}{100}\right) + 48.75 \times \left(1 + \frac{13 + 0.0}{100}\right) + 41.02 \times \left(1 + \frac{8.5 + 4.0}{100}\right)} \times 100 = 10.57\%$$

$$P_{2A} \% (\text{viscose}) = \frac{48.75 \times \left(1 + \frac{13 + 0.0}{100}\right)}{113.2041} \times 100 = 48.65\%$$

$$P_{3A} \% (\text{cotton}) = 100 - (10.57 + 48.65) = 40.78\%$$

The composition of the mixture is therefore:

Viscose	48.6 %
Cotton	40.8 %
Wool	10.6 %
	<u>100.0 %</u>



ANNEX III  
TABLE OF TYPICAL TERNARY MIXTURES WHICH MAY BE ANALYSED USING  
COMMUNITY METHODS OF ANALYSIS OF BINARY MIXTURES  
(for illustration purposes)

Mixture No	Component fibres			Variant (1)	Number of Community binary method and reagent
	1st component	2nd component	3rd component		
1	wool or hair	viscose, cupro or certain types of modal	cotton	1 and/or 4	2 (alkaline sodium hypochlorite) and 3 (zinc chloride/formic acid)
2	wool or hair	polyamide 6 or 6-6	cotton, viscose, cupro or modal	1 and/or 4	2 (alkaline sodium hypochlorite) and 4 (formic acid 80 % w/w)
3	wool, hair or silk	certain chlorofibres	viscose, cupro, modal or cotton	1 and/or 4	2 (alkaline sodium hypochlorite) and 9 (carbon disulphide/acetone 55.5/44.5 w/w)
4	wool or hair	polyamide 6 or 6-6	polyester, polypropylene, acrylic or glass fibre	1 and/or 4	2 (alkaline sodium hypochlorite) and 4 (formic acid 80 % w/w)
5	wool, hair or silk	certain chlorofibres	polyester, acrylic, polyamide or glass fibre	1 and/or 4	2 (alkaline sodium hypochlorite) and 9 (carbon disulphide/acetone 55.5/44.5 w/w)
6	silk	wool or hair	polyester	2	11 (sulphuric acid 75 % w/w) and 2 (alkaline sodium hypochlorite)
7	polyamide 6 or 6-6	acrylic	cotton, viscose, cupro or modal	1 and/or 4	4 (formic acid 80 % w/w) and 8 (dimethylformamide)
8	certain chlorofibres	polyamide 6 or 6-6	cotton, viscose, cupro or modal	1 and/or 4	8 (dimethylformamide) and 4 (formic acid 80 % w/w) or 9 (carbon disulphide/acetone 55.5/44.5 w/w) and 4 (formic acid 80 % w/w)
9	acrylic	polyamide 6 or 6-6	polyester	1 and/or 4	8 (dimethylformamide) and 4 (formic acid 80 % w/w)
10	acetate	polyamide 6 or 6-6	viscose, cotton, cupro or modal	4	1 (acetone) and 4 (formic acid 80 % w/w)
11	certain chlorofibres	acrylic	polyamide	2 and/or 4	9 (carbon disulphide/acetone 55.5/44.5 w/w) and 8 (dimethylformamide)

Mixture No	Component fibres			Variant (1)	Number of Community binary method and reagent
	1st component	2nd component	3rd component		
12	certain chlorofibres	polyamide 6 or 6-6	acrylic	1 and/or 4	9 (carbon disulphide/acetone 55.5/44.5 w/w) and 4 (formic acid 80 % w/w)
13	polyamide 6 or 6-6	viscose, cupro, modal or cotton	polyester	4	4 (formic acid 80 % w/w) and 7 (sulphuric acid 75 % w/w)
14	acetate	viscose, cupro, modal or cotton	polyester	4	1 (acetone) and 7 (sulphuric acid 75 % w/w)
15	acrylic	viscose, cupro, modal or cotton	polyester	4	8 (dimethylformamide) and 7 (sulphuric acid 75 % w/w)
16	acetate	wool, hair or silk	cotton, viscose, cupro, modal, polyamide, polyester, acrylic	4	1 (acetone) and 2 (alkaline sodium hypochlorite)
17	triacetate	wool, hair or silk	cotton, viscose, cupro, modal, polyamide, polyester, acrylic	4	6 (dichloromethane) and 2 (alkaline sodium hypochlorite)
18	acrylic	wool, hair or silk	polyester	1 and/or 4	8 (dimethylformamide) and 2 (alkaline sodium hypochlorite)
19	acrylic	silk	wool or hair	4	8 (dimethylformamide) and 1 (sulphuric acid 75 % w/w)
20	acrylic	wool, hair or silk	cotton, viscose, cupro or modal	1 and/or 4	8 (dimethylformamide) and 2 (alkaline sodium hypochlorite)
21	wool, hair or silk	cotton, viscose, modal, cupro	polyester	4	2 (alkaline sodium hypochlorite) and 7 (sulphuric acid 75 % w/w)
22	viscose, cupro or certain types of modal	cotton	polyester	2 and/or 4	3 (zinc chloride/formic acid) and 7 (sulphuric acid 75 % w/w)
23	acrylic	viscose, cupro or certain types of modal	cotton	4	8 (dimethylformamide) and 3 (zinc chloride/formic acid)
24	certain chlorofibres	viscose, cupro or certain types of modal	cotton	1 and/or 4	9 (carbon disulphide/acetone 55.5/44.5 w/w) and 3 (zinc chloride/formic acid) or 8 (dimethylformamide) and 3 (zinc chloride/formic acid)

Mixture No	Component fibres			Variant (1)	Number of Community binary method and reagent
	1st component	2nd component	3rd component		
25	acetone	viscose, cupro or certain types of modal	cotton	4	1 (acetone) and 3 (zinc chloride/formic acid)
26	triacetate	viscose, cupro or certain types of modal	cotton	4	6 (dichloromethane) and 3 (zinc chloride/formic acid)
27	acetate	silk	wool or hair	4	1 (acetone) and 11 (sulphuric acid 75 % w/w)
28	triacetate	silk	wool or hair	4	6 (dichloromethane) and 11 (sulphuric acid 75 % w/w)
29	acetate	acrylic	cotton, viscose, cupro or modal	4	1 (acetone) and 8 (dimethylformamide)
30	triacetate	acrylic	cotton, viscose, cupro or modal	4	6 (dichloromethane) and 8 (dimethylformamide)
31	triacetate	polyamide 6 or 6-6	cotton, viscose, cupro or modal	4	6 (dichloromethane) and 4 (formic acid 80 % w/w)
32	triacetate	cotton, viscose, cupro or modal	polyester	4	6 (dichloromethane) and 7 (sulphuric acid 75 % w/w)
33	acetate	polyamide 6 or 6-6	polyester or acrylic	4	1 (acetone) and 4 (formic acid 80 % w/w)
34	acetate	acrylic	polyester	4	1 (acetone) and 8 (dimethylformamide)
35	certain chlorofibres	cotton, viscose, cupro or modal	polyester	4	8 (dimethylformamide) and 7 (sulphuric acid 75 % w/w) or 9 (carbon disulphide/acetone 55.5/44.5 w/w) and 7 (sulphuric acid 75 % w/w)

(1) When variant 4 is used, first remove the first component using the first reagent.