

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 textile fibre mixtures

(72/276/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 1971¹ 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 its quantitative analysis;

Whereas Article 13 of the above Directive provides that the sampling and analysing 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, initially, methods applicable to certain binary mixtures of textile fibres should be laid down, to be followed by directives specifying methods for mixtures of more than two textile fibres or of fibres of the same chemical composition (in particular microscopic analysis);

Whereas developments in technology necessitate frequent adaptation of the technical specifications defined in the separate directives on methods of textile analysis; whereas, in order to facilitate the implementation of measures required to that effect, a

procedure should be laid down establishing, within a Committee for the Adaptation of Methods of Textile Analysis to Developments in Technology, close cooperation between Member States and the Commission;

Whereas, in the case of binary mixtures for which there is no uniform method of analysis at Community level, the laboratory responsible for the test may determine the composition of such mixtures using any valid method at its disposal, indicating in the analysis report the result obtained and, in so far as this is known, the degree of accuracy of the method used;

HAS ADOPTED THIS DIRECTIVE:

Article 1

This Directive concerns methods for the quantitative analysis of certain binary textile fibre mixtures, including the preparation of test samples and test specimens.

Article 2

'Test sample' means a sample of a suitable size for analysis, taken from laboratory bulk samples in turn taken from a batch of articles for analysis.

'Test specimen' means that part of the test sample required to give an individual test result.

Article 3

The Member States shall take all necessary steps to ensure that, in accordance with the Directive of 26 July 1971, the provisions in Annexes I and II on methods for the quantitative analysis of certain binary mixtures, including the preparation of test samples and test specimens, are applied in all official

¹ OJ No L 185, 16.8.1971, p. 16.

tests to determine the composition of textile products put on the market.

Article 4

Any laboratory responsible for the testing of binary mixtures for which there is no uniform method of analysis at Community level shall determine the composition of such mixtures by using any valid method at its disposal, indicating in the analysis report the result obtained and, in so far as this is known, the degree of accuracy of the method used.

Article 5

1. There is hereby set up a Committee for the Adaptation of Methods of Textile Analysis to Developments in Technology (hereinafter called the 'Committee'); it shall consist of representatives of the Member States with a representative of the Commission as Chairman.

2. The Commission shall adopt its own rules of procedure.

3. Adaptations to developments in technology of the methods of quantitative analysis provided for in Annex II shall be made in accordance with the procedure laid down in Article 6.

Article 6

1. Where the procedure laid down in this Article is invoked, matters shall be referred to the Committee by its Chairman, either on his own initiative or at the request of the representative of a Member State.

2. The representative of the Commission shall submit to the Committee a draft of the measures to be adopted. The Committee shall deliver its opinion on the draft within a time limit set by the Chairman having regard to the urgency of the matter. Opinions shall be delivered by a majority of twelve votes, the

votes of the Member States being weighted as provided in Article 148 (2) of the Treaty.

The Chairman shall not vote.

3. (a) The Commission shall adopt the measures envisaged where they are in accordance with the opinion of the Committee.

(b) Where the measures envisaged are not in accordance with the opinion of the Committee, or if no opinion is delivered, the Commission shall without delay propose to the Council the measures to be adopted.

The Council shall act by a qualified majority.

(c) If, within three months of the proposal being submitted to it, the Council has not acted, the proposed measures shall be adopted by the Commission.

Article 7

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

2. Member States shall ensure that the texts of the main provisions of national law which they adopt in the field governed by this Directive are communicated to the Commission.

Article 8

This Directive is addressed to the Member States.

Done at Brussels, 17 July 1972.

For the Council

The President

P. LARDINOIS

ANNEX I

PREPARATION OF TEST SAMPLES AND TEST SPECIMENS TO DETERMINE THE FIBRE COMPOSITION OF TEXTILE PRODUCTS

1. FIELD OF APPLICATION

This Annex gives procedures for obtaining laboratory test samples of a suitable size for pre-treatment for quantitative analysis (i.e. of a mass not exceeding 100 g) from laboratory bulk samples, and for selecting test specimens from the laboratory test samples that have been pre-treated to remove non-fibrous matter¹.

2. DEFINITIONS

- 2.1 **Bulk source** — That quantity of material which is judged on the basis of one series of test results. This may comprise, for example, all the material in one delivery of cloth; all the cloth woven from a particular beam; a consignment of yarn, a bale or a group of bales of raw fibre.
- 2.2 **Laboratory bulk sample** — That portion of the bulk source taken to be representative of the whole, and which is available to the laboratory. The size and nature of the laboratory bulk sample should be sufficient to overcome adequately the variability of the bulk source and to facilitate ease of handling in the laboratory².
- 2.3 **Laboratory test sample** — That portion of the laboratory bulk sample that is subjected to pre-treatment to remove non-fibrous matter, and from which test specimens are taken. The size and nature of the laboratory test sample should be sufficient to overcome adequately the variability of the laboratory bulk sample³.
- 2.4 **Test specimen** — The portion of material required to give an individual test result, and selected from the laboratory test sample.

3. PRINCIPLE

The laboratory test sample is selected so that it is representative of the laboratory bulk sample.

The test specimens are taken from the laboratory test sample in such a way that each of them is representative of the laboratory test sample.

4. SAMPLING FROM LOOSE FIBRES

- 4.1 **Unorientated fibres** — Obtain the laboratory test sample by selecting at random tufts from the laboratory bulk sample. Mix thoroughly the whole of the laboratory test sample by means of a laboratory carder.⁴ Subject the web or mixture, including loose fibres and fibres adhering to the equipment used for mixing, to pre-treatment. Then select test specimens, in proportion to the respective masses, from the web or mixture, from the loose fibres and from the fibres adhering to the equipment.

If the card web remains intact after pre-treatment, select the test specimens in the manner described in 4.2. If the card web is disturbed by the pre-treatment, select each test specimen by removing at random at least 16 small tufts of suitable and approximately equal size and then combine them.

- 4.2 **Orientated fibres (cards, webs, slivers, rovings)** — From randomly selected parts of the laboratory bulk sample cut not less than ten cross-sections each of mass approximately 1 g. Subject the laboratory test sample so formed to the pre-treatment. Recombine the cross-sections by laying them side by side and obtain the test specimen by cutting through them so as to take a portion of each of the ten lengths.

¹ In some cases it is necessary to pre-treat the individual test specimen.

² For made-up and finished articles see section 7.

³ See 1.

⁴ The laboratory carder may be replaced by a fibre blender, or the fibres may be mixed by the method of 'tufts and rejects'.

5. SAMPLING YARN

5.1 Yarn in packages or in hanks — Sample all the packages in the bulk laboratory sample.

Withdraw the appropriate continuous equal lengths from each package either by winding skeins of the same number of turns on a wrap-reel,¹ or by some other means. Unite the lengths side by side either as a single skein or as a tow to form the laboratory test sample, ensuring that there are equal lengths from each package in the skein or tow.

Subject the laboratory test sample to the pre-treatment.

Take test specimens from the laboratory test sample by cutting a bunch of threads of equal length from the skein or tow, taking care to see that the bunch contains all the threads in the sample.

If the tex of the yarn is t and the number of packages selected from the laboratory bulk sample is n , then to obtain a test sample of 10 g, the length of yarn to be withdrawn from each package is $\frac{10^6}{Nt}$ cm.

If nt is high, i.e. more than 2000, wind a heavier skein and cut it across in two places to make a tow of suitable mass. The ends of any sample in the form of a tow should be securely tied before pre-treatment and test specimens taken from a place remote from the tie bands.

5.2 Yarn on warp — Take the laboratory test sample by cutting a length from the end of the warp, not less than 20 cm long and comprising all the yarns in the warp except the selvedge yarns, which are rejected. Tie the bunch of threads together near one end. If the sample is too large for pre-treatment as a whole divide it into two or more portions, each tied together for pre-treatment, and reunite the portions after each has been pre-treated separately. Take a test specimen by cutting a suitable length from the laboratory test sample from the end remote from the tie band, and comprising all the threads in the warp. For warp of N threads of tex t , the length of a specimen of mass 1 g is $\frac{10^6}{Nt}$ cm.

6. SAMPLING FABRIC

6.1 From a laboratory bulk sample consisting of a single cutting representative of the cloth

— Cut a diagonal strip from one corner to the other and remove the selvages. This strip is the laboratory test sample. To obtain a laboratory test sample of x g, the strip area shall be $\frac{x \cdot 10^4}{G}$ cm², where G is the mass of the cloth in g/m².

Subject the laboratory test sample to the pre-treatment and then cut the strip transversely into four equal lengths and superimpose them.

Take test specimens from any part of the layered material by cutting through all the layers so that each specimen contains an equal length of each layer.

If the fabric has a woven design, make the width of the laboratory test sample, measured parallel to the warp direction, not less than one warp repeat of the design. If, with this condition satisfied, the laboratory test sample is too large to be treated as a whole, cut it into equal parts, pre-treat them separately, and superimpose these parts before selection of the test specimen, taking care that corresponding parts of the design do not coincide.

6.2 From a laboratory bulk sample consisting of several cuttings

— Treat each cutting as described in 6.1, and give each result separately.

7. SAMPLING MADE-UP AND FINISHED ARTICLES

The bulk laboratory sample is normally a complete made-up or finished article or a representative fraction of one.

¹ If the packages can be mounted in a convenient creel a number can be wound simultaneously.

Where appropriate determine the percentage of the various parts of the article not having the same fibre content, in order to check compliance with Article 9 of the Directive on the approximation of the laws of the Member States relating to textile names.

Select a laboratory test sample representative of the part of the made-up or finished article, whose composition must be shown by the label. If the article has several labels, select laboratory test samples representative of each part corresponding to a given label.

If the article whose composition is to be determined is not uniform, it may be necessary to select laboratory test samples from each of the parts of the article and to determine the relative proportions of the various parts in relation to the whole article in question.

Then calculate the percentages taking into account the relative proportions of the sampled parts.

Subject the laboratory test samples to the pre-treatment.

Then select test specimens representative of the pre-treated laboratory test samples.

$$P_1A = \frac{100 P_1 \left(1 + \frac{a_1 + b_1}{100}\right)}{P_1 \left(1 + \frac{a_1 + b_1}{100}\right) + (100 - P_1) \left(1 + \frac{a_2 + b_2}{100}\right)}$$

where

P_1A is the percentage of insoluble component, adjusted by conventional recovery factors and for loss of mass during pre-treatment;

P_1 is the percentage of clean, dry insoluble component as calculated from the formula shown in I.8.1;

a_1 is the conventional recovery factor for the insoluble component (see Annex II of the Directive on textile names);

a_2 is the conventional recovery factor for the soluble component (see Annex II of the Directive on textile names);

b_1 is the percentage loss of insoluble component caused by the pre-treatment;

b_2 is the percentage loss of soluble component caused by the pre-treatment.

The percentage of the second component ($P_2 A$) is equal to $100 - P_1 A$.

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 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 and b_2 , as obtained from tests performed on clean fibres similar to those in the mixture under examination, should be used.

If normal pre-treatment by extraction with light petroleum and water is applied, correction factors b_1 and b_2 may generally be ignored, except in the case of unbleached cotton, unbleached flax and unbleached hemp, where the loss due to the pre-treatment is conventionally taken as 4%, and in the case of polypropylene, where it is taken as 1%.

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

ANNEX II

METHODS FOR QUANTITATIVE ANALYSIS OF CERTAIN BINARY FIBRE MIXTURES

1. GENERAL

Introduction

Methods for the quantitative analysis of fibre mixtures are based on two main processes, the manual separation and the chemical separation of fibres.

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 fibre of the warp is of a different kind to that of the weft, or knitted fabrics capable of being unravelled made up of yarns of different types.

In general, the methods of chemical quantitative analysis are based on the selective solution of the individual components. After the removal of a component the insoluble residue is weighed, and the proportion of the soluble component is calculated from the loss in mass. This first part of the Annex gives the information common to the analyses by this method of all fibre mixtures dealt with in the Annex, whatever their composition. It should thus be used in conjunction with the succeeding individual sections of the Annex, which contain the detailed procedures applicable to particular fibre mixtures. Occasionally, an analysis is based on a principle other than selective solution; in such cases full details are given in the appropriate section.

Mixtures of fibres during processing and, to a lesser extent, 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 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 component and/or it may be partially or completely removed by the reagent. 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 this Annex are no longer applicable.

Dye in dyed fabrics 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 factors 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 should have been identified. 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 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. The procedures given apply to single determinations.

At least two determinations on separate test specimens should be made, both in the case of manual separation and in the case of chemical separation. For confirmation, unless technically impossible, it is recommended to use alternative procedures whereby the constituent that was the residue in the standard method is dissolved out first.

I. *General information on methods for the quantitative chemical analysis of textile fibre mixtures*

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

I.1 *Scope and field of application*

The field of application for each method specifies to which fibres the method is applicable.

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 of the components, usually by selective solution.¹ The insoluble residue is weighed and the proportion of soluble component calculated from the loss in mass. Except where this presents technical difficulties, it is preferable to dissolve the fibre present in the greater proportion, thus obtaining the fibre present in the smaller proportion as residue.

I.3 *Materials and equipment*

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 $150^{\circ} \pm 3^{\circ}\text{C}$.

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 sections of each 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 specimen 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.

¹ Method 13 is an exception. It is based on a determination of the content of a constituent substance of one of the two components.

1.6 *Pre-treatment of laboratory test sample¹*

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

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 extractor with light petroleum for 1 hour at a minimum rate of 6 cycles per hour. Allow the light petroleum to evaporate from the sample, which is then extracted by direct treatment consisting in soaking the specimen in water at room temperature for 1 hour and then soaking it in water at 65 ± 5 °C for a further hour, agitating the liquor from time to time. Use a liquor: specimen ratio of 100:1. 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 (e.g. 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.

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

1.7 *Test procedure*

1.7.1 *General instructions*

1.7.1.1 *Drying*

Conduct all drying operations for not less than 4 hours and not more than 16 hours at 105 ± 3 °C in a ventilated oven with the oven door closed throughout. If the drying period is less than 14 hours, the specimen must be weighed to check that its mass has become constant. The mass may be considered to have become 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 in the oven should be conducted in such a way as to enable the dry mass of the fibres to be determined without loss.

1.7.1.2 *Cooling*

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

1.7.1.3 *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.

1.7.2 *Procedure*

Take from the pre-treated laboratory test sample a test specimen weighing at least 1 g. Cut yarn or cloth into lengths of about 10 mm, dissected as much as possible. Dry the specimen in a weighing bottle, cool it in the desiccator and weigh it. Transfer the

¹ See Annex I.1.

specimen to the glass vessel specified in the appropriate section of the relevant Community method, reweigh the weighing bottle immediately and obtain the dry mass of the specimen by difference. Complete the test as specified in the appropriate section of the applicable method. Examine the residue microscopically to check that the treatment has in fact completely removed the soluble fibre.

I.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. The percentage of soluble component is obtained by difference. Calculate the results on the basis of clean, dry mass, adjusted by (a) the relevant recovery factors and (b) the correction factors necessary to take account of loss of matter during pre-treatment and analysis.

Calculations should be made by applying the formula given in I.8.2.

I.8.1 Calculation of percentage of insoluble component on clean, dry mass basis, disregarding loss of fibre mass during pre-treatment.

$$P_1 = \frac{100 \text{ rd}}{m}$$

where

P_1 is the percentage of clean, dry insoluble component;

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

r is the dry mass of the residue;

d is the correction factor for loss of mass of the insoluble component in the reagent during the analysis.

Suitable values of d are given in the appropriate section of each method.

Such values of d are of course the normal values applicable to chemically undegraded fibres.

I.8.2 Calculation of percentage of insoluble component on clean, dry mass basis, with adjustment by conventional recovery factors and, where appropriate, correction factors for loss mass during pre-treatment.

II. *Method of quantitative analysis by manual separation*

II.1 *Field of application*

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 constituents of the textile, the non-fibrous material is removed by 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 bottle or any other apparatus giving identical results.

II.3.2 Desiccator containing self-indicating silica gel.

II.3.3 Ventilated oven for drying specimens at 105 ± 3 °C.

II.3.4 Analytical balance, accurate to 0.0002 g.

II.3.5 Soxhlet extractor, or other apparatus giving an identical result.

II.3.6 Needle.

II.3.7 Twist tester or similar apparatus.

II.4 *Reagents*

II.4.1 Light petroleum, redistilled, boiling range 40, to 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 sample*

See I.6.

II.8 *Procedure*

II.8.1 *Analysis of yarn*

Select from the pre-treatment 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 ± 3 °C until a constant mass is obtained, as described in I.7.1 and I.7.2.

II.8.2 *Analysis of cloth*

Select from the pre-treated laboratory test sample, well away from all selvages, a specimen of mass not less than 1 g, 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 wales and courses. Separate the different fibre types, 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 fibre constituent 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 relevant recovery factors and (b) the correction factors necessary to take account of loss of matter during pre-treatment.

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

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

where

P_1 is the percentage of the first 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.

II.9.2 For calculation of the percentage of each component with adjustment by conventional recovery factors and, where appropriate, by correction factors for loss of matter during pre-treatment, see I.8.2.

III.1 *Precision of the methods*

The precision indicated in individual methods relates to the reproducibility.

The 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 consistent mixture.

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

By this is meant that the difference between two results in a series of analysis made in different laboratories would, given a normal and correct application of the method to an identical and consistent mixture, be exceeded only in 5 cases out of a 100.

III.2 *Test report*

III.2.1 State that the analysis was conducted in accordance with this method.

III.2.3 Give details of any special pre-treatment (see I.6).

III.2.3 Give the individual results and the arithmetic mean, each to an accuracy of 0.1.

2. SPECIAL METHODS — SUMMARY TABLE

Method	Field of application		Reagent	Page No
No 1	Acetate	Certain other fibres	Acetone	798
No 2	Certain protein fibres	Certain other fibres	Alkaline sodium hypochlorite	799
No 3	Viscose, cupro or certain types of modal	Cotton	Zinc chloride Formic acid	800
No 4	Nylon 6 or nylon 6.6	Certain other fibres	Formic acid, 80% m/m	801
No 5	Acetate	Triacetate	Benzyl alcohol	802
No 6	Triacetate	Certain other fibres	Dichloromethane	803
No 7	Certain cellulose fibres	Polyester	Sulphuric acid, 75% m/m	804
No 8	Acrylics, certain modacrylics or certain chlorofibres	Certain other fibres	Dimethylformamide	805
No 9	Certain chlorofibres	Certain other fibres	Carbon disulphide/ acetone, 55.5/44.5 v/v	806
No 10	Acetate	Certain chlorofibres	Glacial acetic acid	807
No 11	Silk	Wool or hair	Sulphuric acid, 75% m/m	808
No 12	Certain cellulose fibres	Wool or hair	Sulphuric acid, 70% m/m	809
No 13	Jute	Certain animal fibres	Nitrogen content method	811

METHOD No 1

ACETATE AND CERTAIN OTHER FABRICS

(Acetone method)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

1. acetate (17)
with
2. wool (1), animal hair (2 and 3), silk (4), cotton (5), flax (7), true hemp (8), jute (9), abaca (10), alfa (11), coir (12), broom (13), kenaf (14), ramie (15), sisal (16), cupro (19), modal (20), protein (21), viscose (23), acrylic (24), nylon (28) and polyester (29).

In no circumstances is the method applicable to acetate fibres which have been deacetylated on the surface.

2. PRINCIPLE

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

3. APPARATUS AND REAGENTS (additional to those specified in the general instructions)

3.1 Apparatus

Glass-stoppered conical flasks of at least 200 ml capacity.

3.2 Reagent

Acetone.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the specimen contained in a glass-stoppered conical flask of at least 200 ml capacity, add 100 ml of acetone per gramme of specimen, shake the flask, stand it for 30 minutes at room temperature, stirring from time to time, and then decant the liquid through the weighed filter crucible.

Repeat the treatment twice more (making three extractions in all), but for periods of 15 minutes only, so that the total time of treatment in acetone is one hour. Transfer the residue to the filter crucible. Wash the residue in the filter crucible with acetone and drain with suction. Refill the crucible with acetone and allow to drain under gravity.

Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of d is 1.00.

6. PRECISION

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

METHOD No 2

CERTAIN PROTEIN FIBRES AND CERTAIN OTHER FIBRES

(Method using alkaline sodium hypochlorite)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

1. certain protein fibres, namely: wool (1), animal hair (2 and 3), silk (4), regenerated protein based on casein (21)
with
2. cotton (5), cupro (19), modal (20), viscose (23), acrylic (24), chlorofibres (25), nylon (28), polyester (29), polypropylene (31) and glass fibre (38).

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

2. PRINCIPLE

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

3. APPARATUS AND REAGENTS (other than those specified in the general instructions)

3.1 Apparatus

Glass beaker of at least 500 ml capacity.

3.2 Reagents

(i) *Hypochlorite reagent*

This reagent consists of a freshly prepared, approximately molar, solution (33 to 37 g of active chlorine per litre), to which has been added a sufficient quantity of sodium hydroxide of 5 g/l (± 0.5 g/l). Check the concentration of the solution idometrically before each analysis.

(ii) *Acetic acid, dilute solution*

Dilute 5 ml glacial acetic acid to 1 litre with water.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows: to the specimen contained in the glass beaker of at least 500 ml capacity, add 100 ml of hypochlorite reagent per gramme of specimen; stir vigorously to wet out the specimen; leave for 40 minutes at room temperature, stirring vigorously at regular intervals. Filter the contents of the beaker through the weighed filter crucible and transfer any residual fibres to the crucible by washing out the beaker with a little hypochlorite reagent. Drain the crucible with suction and wash the residue successively with water, dilute acetic acid, and finally water, draining the crucible with suction after each addition. Do not apply suction until each washing liquor has drained under gravity. Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of d is 1.00, except for unbleached cotton, for which $d = 1.03$.

6. PRECISION

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

METHOD No 3

VISCOSE, CUPRO OR CERTAIN TYPES OF MODAL AND COTTON

(Method using formic acid and zinc chloride)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

1. viscose (23) or cupro (19), including certain types of modal fibre (20),
with
2. cotton (5).

If a modal fibre is found to be present, a preliminary test should be carried out to see whether it is soluble in the reagent.

This method is not applicable to mixtures in which the cotton has suffered extensive chemical degradation nor when the viscose or cupro is rendered incompletely soluble by the presence of certain dyes or finishes that cannot be removed completely.

2. PRINCIPLE

The viscose, cupro or modal fibre is dissolved from a known dry mass of the mixture, with a reagent consisting of formic acid and zinc chloride. The residue is collected, washed, dried and weighed; its corrected mass is expressed as a percentage of the dry mass of the mixture. The percentage of dry viscose, cupro or modal fibre is found by difference.

3. APPARATUS AND REAGENTS (other than those specified in the general instructions)

3.1 Apparatus

- (i) Glass-stoppered conical flasks of at least 200 ml capacity,
- (ii) Apparatus for maintaining flasks at 40 ± 2 °C.

3.2 Reagents

- (i) Solution containing 20 g of anhydrous zinc chloride and 68 g of anhydrous formic acid made up to 100 g with water (namely 20 parts mass of zinc chloride to 80 parts mass of 85% m/m formic acid);
- (ii) Ammonium hydroxide solution: dilute 20 ml of a concentrated ammonia solution (specific gravity 0.880 g/ml) to 1 litre with water.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows: place the specimen immediately in the flask, pre-heated to 40 °C. Add 100 ml of the solution of formic acid and zinc chloride, pre-heated to 40 °C per gramme of specimen. Insert the stopper and shake the flask vigorously. Keep the flask and its contents at a constant temperature of 40 °C for two hours and a half, shaking the flask at hourly intervals. Filter the contents of the flask through the weighed filter crucible and with the help of the reagent transfer to the crucible any fibres remaining in the flask. Rinse with 20 ml of reagent.

Wash crucible and residue thoroughly with water at 40 °C. Rinse with 100 ml ammonia solution, then with water until all trace of ammonia has disappeared. Do not apply suction until each washing liquor has drained under gravity. Finally, drain the remaining liquid with suction, dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of d for cotton is 0.96.

6. PRECISION

On a homogeneous mixture of textile materials, the confidence limits of results obtained by this method are not greater than ± 2 for a confidence level of 95%.

METHOD No 4

NYLON 6 OR NYLON 6.6 AND CERTAIN OTHER FIBRES

(Method using 80% m/m formic acid)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

1. nylon 6 or nylon 6.6 (28)
with
2. wool (1), animal hair (2 and 3), cotton (5), cupro (19), modal (20), viscose (23), acrylic (24), chlorofibre (25), polyester (29), polypropylene (31) and glass fibre (38).

As mentioned above, this method is also applicable to mixtures with wool, but when the wool content exceeds 25%, method No 2 should be applied (dissolving wool in a solution of alkaline sodium hypochlorite).

2. PRINCIPLE

The polyamide fibre is dissolved out from a known dry mass of the mixture, with formic acid. The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of nylon 6 or nylon 6.6 is found by difference.

3. APPARATUS AND REAGENTS (other than those specified in the general instructions)

3.1 Apparatus

Glass-stoppered conical flask of at least 200 ml capacity.

3.2 Reagents

- (i) Formic acid (80% m/m, relative density at 20 °C: 1.186). Dilute 880 ml of 90% m/m formic acid (relative density at 20 °C: 1.204) to 1 litre with water. Alternatively, dilute 780 ml of 98–100% m/m formic acid (relative density at 20 °C: 1.220) to 1 litre with water.

The concentration is not critical within the range 77% to 83% m/m formic acid.

- (ii) Ammonia, dilute solution: dilute 80 ml of concentrated ammonia solution (relative density at 20 °C: 0.880) to 1 litre with water.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows: to the specimen contained in the conical flask of at least 200 ml capacity, add 100 ml of formic acid per gramme of specimen. Insert the stopper, shake the flask to wet out the specimen. Stand the flask for 15 minutes at room temperature, shaking it at intervals. Filter the contents of the flask through the weighed filter crucible and transfer any residual fibres to the crucible by washing out the flask with a little formic acid reagent. Drain the crucible with suction and wash the residue on the filter successively with formic acid reagent, hot water, dilute ammonia solution, and finally cold water, draining the crucible with suction after each addition. Do not apply suction until each washing liquor has drained under gravity. Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of d is 1.00.

6. PRECISION

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

METHOD No 5

ACETATE AND TRIACETATE

(Method using benzyl alcohol)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

— acetate (17)

with

— triacetate (22)

2. PRINCIPLE

The acetate fibre is dissolved out from a known dry mass of the mixture, with benzyl alcohol at 52 ± 2 °C.

The residue is collected, washed, dried and weighed; its mass is expressed as a percentage of the dry mass of the mixture. The percentage of dry acetate is found by difference.

3. APPARATUS AND REAGENTS (other than those specified in the general instructions)

3.1 Apparatus

(i) Glass-stoppered conical flask of at least 200 ml capacity;

(ii) Mechanical shaker;

(iii) Thermostat or other apparatus for keeping the flask at a temperature of 52 ± 2 °C.

3.2 Reagents

(i) Benzyl alcohol;

(ii) Ethanol.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the specimen contained in the conical flask, add 100 ml of benzyl alcohol per gramme of specimen.

Insert the stopper, secure the flask to the shaker so that it is immersed in the water-bath, kept at 52 ± 2 °C, and shake for 20 minutes at this temperature.

(Instead of using a mechanical shaker, the flask may be shaken vigorously by hand.)

Decant the liquid through the weighed filter crucible. Add a further dose of benzyl alcohol in the flask and shake as before at 52 ± 2 °C for 20 minutes.

Decant the liquid through the crucible. Repeat the cycle of operations a third time.

Finally pour the liquid and the residue into the crucible; wash any remaining fibres from the flask into the crucible with an extra quantity of benzyl alcohol at 52 ± 2 °C. Drain the crucible thoroughly.

Transfer the fibres into a flask, rinse with ethanol and after shaking manually decant through the filter crucible.

Repeat this rinsing operation two or three times. Transfer the residue into the crucible and drain thoroughly. Dry the crucible and the residue and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of d is 1.00.

6. PRECISION

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

METHOD No 6

TRIACETATES AND CERTAIN OTHER FIBRES

(Method using dichloromethane)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

1. triacetate (22)
with
2. wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (19), modal (20), viscose (23), acrylic (24), nylon (28), polyester (29) and glass fibre (38).

Note:

Triacetate fibres which have received a finish leading to partial hydrolysis cease to be completely soluble in the reagent. In such cases, the method is not applicable.

2. PRINCIPLE

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

3. APPARATUS AND REAGENTS (other than those specified in the general instructions)

3.1 Apparatus

Glass-stoppered conical flask of at least 200 ml capacity.

3.2 Reagent

Dichloromethane.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the specimen contained in the 200 ml glass-stoppered conical flask, add 100 ml of dichloromethane per gramme of specimen, insert the stopper, shake the flask every ten minutes to wet out the specimen and stand for thirty minutes at room temperature, shaking the flask at regular intervals. Decant the liquid through the weighed filter crucible. Add 60 ml of dichloromethane to the flask containing the residue, shake manually and filter the contents of the flask through the filter crucible. Transfer the residual fibres to the crucible by washing out the flask with a little more dichloromethane. Drain the crucible with suction to remove excess liquid, refill the crucible with dichloromethane and allow it to drain under gravity. Finally, apply boiling water treatment to the residue, transfer it back into the crucible, drain with suction, dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of d is 1.00, except in the case of polyester, for which the value of d is 1.01.

6. PRECISION

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

METHOD No 7

CERTAIN CELLULOSE FIBRES AND POLYESTER

(Method using 75% m/m sulphuric acid)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

1. cotton (5), flax (7), true hemp (8), ramie (15), cupro (19), modal (20), viscose (23)
with
2. polyester (29).

2. PRINCIPLE

The cellulose fibre is dissolved out from a known dry mass of the mixture, with 75% m/m sulphuric acid. The residue is collected, washed, dried and weighed; its mass is expressed as a percentage of the dry mass of the mixture. The proportion of dry cellulose fibre is found by difference.

3. APPARATUS AND REAGENTS (other than those specified in the general instructions)

3.1 Apparatus

- (i) Glass-stoppered conical flask of at least 500 ml capacity;
- (ii) Thermostat or other apparatus for maintaining the flask at 50 ± 5 °C.

3.2 Reagents

- (i) *Sulphuric acid, 75 ± 2 % m/m*

Prepare by adding carefully, while cooling, 700 ml of sulphuric acid (relative density at 20 °C: 1.84) to 350 ml of distilled water. After the solution has cooled to room temperature, dilute to 1 litre with water.

- (ii) *Ammonia, dilute solution*

Dilute 80 ml of ammonia solution (relative density at 20 °C: 0.88) to 1 litre with water.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the specimen contained in the glass-stoppered conical flask of at least 500 ml capacity, add 200 ml of 75% sulphuric acid per gramme of specimen, insert the stopper and carefully shake the conical flask to wet out the specimen. Maintain the flask at 50 ± 5 °C for one hour, shaking it at regular intervals of roughly ten minutes. Filter the contents of the flask through the weighed filter crucible by means of suction. Transfer any residual fibres by washing out the flask with a little 75% sulphuric acid. Drain the crucible with suction and wash the residue on the filter once by filling the crucible with a fresh portion of sulphuric acid. Do not apply suction until the acid has drained under gravity.

Wash the residue successively several times with cold water, twice with dilute ammonia solution, and then thoroughly with cold water, draining the crucible with suction after each addition. Do not apply suction until each washing liquor has drained under gravity. Finally, drain the remaining liquid from the crucible with suction, dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of d is 1.00.

6. PRECISION

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

METHOD No 8

ACRYLICS, CERTAIN MODACRYLICS OR CERTAIN CHLOROFIBRES
AND CERTAIN OTHER FIBRES

(Method using dimethylformamide)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

1. acrylics (24), certain modacrylics (27), or certain chlorofibres (25)¹
with
2. wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (19), modal (20), viscose (23), nylon (28) and polyester (29).

It is equally applicable to acrylics, and certain modacrylics, treated with pre-metallized dyes, but not to those dyed with afterchrome dyes.

2. PRINCIPLE

The acrylic, modacrylic or chlorofibre is dissolved out from a known dry mass of the mixture, with dimethylformamide heated in a water-bath at boiling-point. The residue is collected, washed, dried and weighed. Its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture and the percentage of dry acrylic, modacrylic or chlorofibre is found by difference.

3. APPARATUS AND REAGENTS (other than those specified in the general instructions)

3.1 Apparatus

- (i) Glass-stoppered conical flask of at least 200 ml capacity;
- (ii) Water bath at boiling point.

3.2 Reagent

Dimethylformamide (boiling point $153 \pm 1^\circ\text{C}$) not containing more than 0.1% water.

This reagent is toxic and the use of a hood is thus recommended.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the specimen contained in the glass-stoppered conical flask of at least 200 ml capacity, add per gramme of specimen 80 ml of dimethylformamide, pre-heated in the water-bath at boiling point, insert the stopper, shake the flask to wet out the specimen and heat in the water-bath at boiling point for one hour. Shake the flask and its contents gently by hand five times during this period.

Decant the liquid through the weighed filter crucible, retaining the fibres in the flask. Add a further 60 ml of dimethylformamide to the flask and heat for a further 30 minutes, shaking the flask and contents gently by hand twice during this period.

Filter the contents of the flask through the filter crucible by means of suction.

Transfer any residual fibres to the crucible by washing out the flask with dimethylformamide. Drain the crucible with suction. Wash the residue twice with cold water, proceeding as follows: fill the crucible, allow it to drain under gravity and then drain with suction.

If the residue consists of nylon or polyester fibre, dry the crucible and the residue and cool weigh them.

¹ The solubility of such modacrylics or chlorofibres in the reagent should be checked before carrying out the analysis.

If the residue consists of regenerated or natural cellulose, silk or wool, transfer it with forceps to a glass-stoppered conical flask of at least 200 ml capacity, add 160 ml of water, and allow to stand for 5 minutes at room temperature, shaking the flask and contents vigorously at regular intervals. Decant the water through the crucible and repeat this washing process three times. After the last wash, filter the contents of the flask through the crucible by means of suction. Transfer any residual fibres by washing out the flask with water. Drain the crucible with suction to remove the water, dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of d is as follows:

silk	1.00
nylon 6, 6.6 or 11	1.01
wool	1.01
cupro	} 1.01
modal	
viscose	
polyester	1.02

6. PRECISION

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

METHOD No 9

CERTAIN CHLOROFIBRES AND CERTAIN OTHER FIBRES

(Method using 55.5/44.5 mixture of carbon disulphide and acetone)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

1. certain chlorofibres (25), namely certain polyvinyl chloride fibres, whether after-chlorinated or not¹ with
2. wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (19), modal (20), viscose (23), acrylic (24), nylon (28), polyester (29), glass fibre (38).

When the wool or silk content of the mixture exceeds 25%, method No 2 should be used.

When the nylon 6 or 6.6 content of the mixture exceeds 25%, method No 4 should be used.

2. PRINCIPLE

The chlorofibre is dissolved out from a known dry mass of the mixture, with an azeotropic mixture of carbon disulphide and acetone. The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of dry polyvinyl chloride fibre is found by difference.

¹ Before carrying out the analysis, the solubility of the chlorofibres in the reagent should be checked.

3. APPARATUS AND REAGENTS (other than those specified in the general instructions)

3.1 Apparatus

- (i) Glass-stoppered flask of at least 200 ml capacity;
- (ii) Mechanical shaker.

3.2 Reagents

- (i) Azeotropic mixture of carbon disulphide and acetone (55.5% by volume carbon disulphide to 44.5% acetone);
As this reagent is toxic, the use of a hood is recommended.
- (ii) Ethanol (92% by volume) or methanol.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the specimen contained in the glass-stoppered conical flask of at least 200 ml capacity, add 100 ml of the azeotropic mixture per gramme of specimen. Seal the flask securely, and shake the flask on a mechanical shaker, or vigorously by hand, for twenty minutes at room temperature. Decant the supernatant liquid through the weighed filter crucible.

Repeat the treatment with 100 ml of fresh reagent. Continue this cycle of operations until no polymer deposit is left on a watch glass when a drop of the extraction liquid is evaporated. Transfer the residue to the filter crucible using more reagent, apply suction to remove the liquid, and rinse the crucible and residue with 20 ml of alcohol and then three times with water. Allow the washing liquor to drain under gravity before draining with suction. Dry the crucible and residue and cool and weigh them.

Note:

With certain mixtures having a high chlorofibre content there may be substantial shrinkage of the specimen during the drying procedure, as a result of which the dissolution of chlorofibre by the solvent is retarded. This does not, however, affect the ultimate dissolution of the chlorofibre in the solvent.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of d is 1.00.

6. PRECISION

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

METHOD No 10

ACETATE AND CERTAIN CHLOROFIBRES

(Method using glacial acetic acid)

1. FIELD OF APPLICATION

This method is applicable, after elimination of non-fibrous matter, to binary mixtures of:

1. acetate (17)
with
2. certain chlorofibres (25), namely polyvinyl chloride, whether after-chlorinated or not.

2. PRINCIPLE

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

3. APPARATUS AND REAGENTS (other than those specified in the general instructions)

3.1 Apparatus

- (i) Glass-stoppered conical flask of at least 200 ml capacity;
- (ii) Mechanical shaker.

3.2 Reagent

Glacial acetic acid (over 99%). This reagent should be handled with care since it is highly caustic.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the specimen contained in the glass-stoppered conical flask of at least 200 ml capacity, add 100 ml glacial acetic acid per gramme of specimen. Seal the flask securely and shake on the mechanical shaker, or vigorously by hand, for twenty minutes at room temperature. Decant the supernatant liquid through the weighed filter crucible. Repeat this treatment twice, using 100 ml of fresh reagent each time, making three extractions in all. Transfer the residue to the filter crucible, drain with suction to remove the liquid and rinse the crucible and the residue with 50 ml of glacial acetic acid, and then three times with water. After each rinse, allow the liquid to drain under gravity before applying suction. Dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the result as described in the general instructions. The value of d is 1.00.

6. PRECISION

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

METHOD No 11

SILK AND WOOL OR HAIR

(Method using 75% m/m sulphuric acid)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

1. silk (4)
with
2. wool (1) or animal hair (2 and 3).

2. PRINCIPLE

The silk fibre is dissolved out from a known dry mass of the mixture, with 75% m/m sulphuric acid.¹

The residue is collected, washed, dried and weighed. Its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of dry silk is found by difference.

¹ Wild silks, such as tussah silk, are not completely soluble in 75% m/m sulphuric acid.

3. APPARATUS AND REAGENTS (other than those specified in the general instructions)

3.1 Apparatus

Glass-stoppered conical flasks of at least 200 ml capacity.

3.2 Reagents

(i) *Sulphuric acid (75 ± 2% m/m)*

Prepare by adding carefully, while cooling, 700 ml sulphuric acid (density at 20 °C: 1.84) to 350 ml distilled water.

After cooling to room temperature, dilute the solution to 1 litre with water.

(ii) *Sulphuric acid, dilute solution:* add 100 ml sulphuric acid (density at 20 °C: 1.84) slowly to 1900 ml distilled water.

(iii) *Ammonia, dilute solution:* dilute 200 ml concentrated ammonia (density at 20 °C: 0.880) to 1000 ml with water.

4. TEST PROCEDURE

Follow the procedure described in the general instruction and proceed as follows:

To the specimen contained in a glass-stoppered conical flask of at least 200 ml capacity, add 100 ml of 75% m/m sulphuric acid per gramme of specimen and insert the stopper. Shake vigorously and stand for thirty minutes at room temperature. Shake again and stand for thirty minutes. Shake a last time and filter the contents of the flask through the weighed filter crucible. Wash any remaining fibres from the flask with the 75% sulphuric acid reagent. Wash the residue on the crucible successively with 50 ml of the dilute sulphuric acid reagent, 50 ml water and 50 ml of the dilute ammonia solution. Each time allow the fibres to remain in contact with the liquid for about 10 minutes before applying suction. Finally rinse with water, leaving the fibres in contact with the water for about 30 minutes. Drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of d is 0.985 for wool.¹

6. PRECISION

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

METHOD No 12

CERTAIN CELLULOSE FIBRES AND WOOL OR HAIR

(Method using 70% m/m sulphuric acid)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

1. cotton (5), flax (7), true hemp (8), ramie (15), cupro (19), modal (20), viscose (23) with
2. wool (1) or animal hair (2 and 3).

When the wool content exceeds 25 %, method No 2, using alkaline sodium hypochlorite, is preferable.

¹ Wild silks, such as tussah silk, are not completely soluble in 75% m/m sulphuric acid.

2. PRINCIPLE

The cellulose fibre is dissolved out from a known dry mass of the mixture, with 70 % m/m sulphuric acid. The residue is collected, washed, dried and weighed; its mass is expressed as a percentage of the dry weight of the mixture. The proportion of dry cellulose fibre is found by difference.

3. APPARATUS AND REAGENTS (other than those described in the general instructions)

3.1 Apparatus

- (i) Beaker of at least 300 ml capacity and glass-stoppered conical flask of at least 300 ml capacity;
- (ii) Thermostat or other apparatus for maintaining the flask at a temperature of 38 ± 1 °C.

3.2 Reagents

- (i) Sulphuric acid (70 ± 2 % m/m).
Add carefully, while cooling, 500 ml sulphuric acid (relative density at 20 °C: 1.84) to 380 ml distilled water.
After cooling to 20 °C, the mixture should have a relative density at 20 °C of 1.62.
- (ii) Sulphuric acid (1% m/m).
Add carefully 6 ml sulphuric acid of relative density 1.84 at 20 °C to 1 litre of water.
- (iii) Solution containing 20 g of sodium bicarbonate per litre.
- (iv) Ethanol (96% by volume).

4. TEST PROCEDURE

Follow the procedure given in the general instructions and proceed as follows:

On the specimen contained in the beaker of at least 300 ml capacity, pour 100 ml of boiling 1% m/m sulphuric acid.

Maintain at boiling point for 10 minutes, stirring with a glass rod.

Filter the contents of the beaker through a weighed filter crucible by means of suction. Transfer the residual fibres by washing out the beaker with water, then drain the crucible with suction.

Transfer the fibres into the conical flask of at least 300 ml capacity. Add per gramme of specimen 200 ml of 70% m/m sulphuric acid, heated to 38 ± 1 °C.

Insert the stopper and shake. Place the flask in the thermostat at a temperature of 38 ± 1 °C for 15 minutes, shaking it at regular intervals of roughly five minutes.

Filter the contents of the flask through the same weighed filter crucible by means of suction. Transfer the residual fibres by washing out the flask with a little 70% m/m sulphuric acid. Drain the crucible by means of suction and wash the residue on the filter a first time by filling the filter with fresh 70% m/m sulphuric acid. Do not apply suction until the acid has drained under gravity. Wash the residue several times with cold water, then with 100 ml of the sodium bicarbonate solution, which is allowed to drain under gravity. Wash thoroughly with cold water and finally pour a little ethanol on the residue, and then drain by means of suction.

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

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of *d* is 1.00 for non-chemically treated wool and hair. It may be in excess of 1.00 for chemically treated wool or hair.

6. PRECISION

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

METHOD No 13

JUTE AND CERTAIN ANIMAL FIBRES

(Method by determining nitrogen content)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to binary mixtures of:

1. jute (9)
with
2. certain animal fibres.

The animal-fibre component may consist solely of hair (2 and 3) or wool (1) or of any mixture of the two. This method is not applicable to textile mixtures containing non-fibrous matter (dyes, finishes, etc.) with a nitrogen base.

2. PRINCIPLE

The nitrogen content of the mixture is determined and from this and the known or assumed nitrogen contents of the two components, the proportion of each component is calculated.

3. APPARATUS AND REAGENTS (other than those specified in the general instructions)

3.1 Apparatus

- (i) Kjeldahl digestion flask, 200–300 ml capacity.
- (ii) Kjeldahl distillation apparatus with steam injection.
- (iii) Titration apparatus, allowing precision of 0.05 ml.

3.2 Reagents

- (i) Toluene
- (ii) Methanol
- (iii) Sulphuric acid, relative density at 20 °C: 1.84¹
- (iv) Potassium sulphate¹
- (v) Selenium dioxide¹
- (vi) Sodium hydroxide solution (400 g/litre). Dissolve 400 g of sodium hydroxide in 400–500 ml of water and dilute to 1 litre with water.
- (vii) Mixed indicator. Dissolve 0.1 g of methyl red in 95 ml of ethanol and 5 ml of water, and mix with 0.5 g of bromocresol green dissolved in 475 ml of ethanol and 25 ml of water.
- (viii) Boric acid solution. Dissolve 20 g boric acid in 1 litre of water.
- (ix) Sulphuric acid, 0.02N (standard volumetric solution).

4. PRE-TREATMENT OF TEST SAMPLE

The following pre-treatment is substituted for the pre-treatment described in the general instructions:

Extract the air-dry sample in a Soxhlet apparatus with a mixture of 1 volume of toluene and 3 volumes of methanol for 4 hours at a minimum rate of 5 cycles per hour. Allow the solvent to evaporate from the sample in air, and remove the last traces in an oven at 105 ± 3 °C. Then extract the sample in water (50 ml per g of sample) by boiling under reflux for 30 minutes. Filter, return the sample to the flask, and repeat the extraction with an identical volume of water. Filter, remove excess water from the sample by squeezing, suction, or centrifuging and then allow the sample to become air-dry.

¹ These reagents should be nitrogen-free.

Note:

The toxic effects of toluene and methanol should be borne in mind and full precautions should be taken in their use.

5. TEST PROCEDURE

5.1 General instructions

Follow the procedure described in the general instructions as regards the selection, drying and weighing of the specimen.

5.2 Detailed procedure

Transfer the specimen to a Kjeldahl digestion flask. To the specimen weighing at least 1 g contained in the digestion flask, add, in the following order, 2.5 g potassium sulphate, 0.1–0.2 g selenium dioxide and 10 ml sulphuric acid (relative density 1.84). Heat the flask, gently at first, until the whole of the fibre is destroyed, and then heat it more vigorously until the solution becomes clear and almost colourless. Heat it for a further fifteen minutes. Allow the flask to cool, dilute the contents carefully with 10–20 ml water, cool, transfer the contents quantitatively to a 200 ml graduated flask and make up to volume with water to form the digest solution.

Place about 20 ml of boric acid solution in a 100 ml conical flask and place the flask under the condenser of the Kjeldahl distillation apparatus so that the delivery tube dips just below the surface of the boric acid solution. Transfer exactly 10 ml of digest solution to the distillation flask, add not less than 5 ml of sodium hydroxide solution to the funnel, lift the stopper slightly and allow the sodium hydroxide solution to run slowly into the flask. If the digest solution and sodium hydroxide solution remain as two separate layers, mix them by gentle agitation. Heat the distillation flask gently and pass it into steam from the generator. Collect about 20 ml of distillate, lower the conical flask so that the tip of the delivery tube of the condenser is about 20 mm above the surface of the liquid and distil for 1 minute more. Rinse the tip of the delivery tube with water, catching the washings in the conical flask. Remove the conical flask and replace it with another conical flask containing roughly 10 ml of boric acid solution and collect about 10 ml distillate.

Titrate the two distillates separately with 0.02N sulphuric acid, used the mixed indicator. Record the total titre for the two distillates. If the titre for the second distillate is more than 0.2 ml, repeat the test and start the distillation again using a fresh aliquot of digest solution.

Carry out a blank determination, i.e. digestion and distillation using the reagents only.

6. CALCULATION AND EXPRESSION OF RESULTS

6.1 Calculate the percentage nitrogen content in the dry specimen as follows:

$$A = \frac{28 (V - b) N}{W},$$

where A = percentage nitrogen in the clean dry specimen,

V = total volume in ml of standard sulphuric acid used in the determination,

b = total volume in ml of standard sulphuric acid used in the blank determination,

N = normality of standard sulphuric acid,

W = dry mass (g) of specimen.

6.2 Using the values of 0.22% for the nitrogen content of jute and 16.2% for the nitrogen content of animal fibre, both percentages being expressed on the dry mass of the fibre, calculate the composition of the mixture as follows:

$$P_A = \frac{A - 0.22}{16.2 - 0.22} \times 100$$

where P_A = percentage of animal fibre in the clean dry specimen.

7. PRECISION

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