COMMISSION OF THE EUROPEAN COMMUNITIES

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Brussels, 24 January 1994

94/0008(COD)

Proposal for a
EUROPEAN PARLIAMENT AND COUNCIL DIRECTIVE

on certain methods for the quantitative analysis
of binary textile fibre mixtures

(presented by the Commission)

EXPLANATORY MEMORANDUM

1. In the context of a people's Europe, the Commission attaches great importance to simplifying and clarifying Community law so as to make it clearer and more accessible to the ordinary citizen, thus giving him new opportunities and the chance to make use of the specific rights it gives him.

   This aim cannot be achieved so long as numerous provisions which have been amended several times, often quite substantially, remain scattered; so that they must be sought partly in the original instrument and partly in later amending ones. Considerable research work, comparing many different instruments, is thus needed to identify the current rules.

   For this reason a consolidation of rules that have frequently been amended is essential if Community law is to be clear and transparent.

2. By its decision of 1 April 1987 the Commission instructed its departments to produce a formal consolidated version of legislative instruments no later than after their tenth amendment, but made it clear that this was a minimum requirement, and that in the interests of clarity and of the ready comprehension of Community law, an effort should be made by each department to consolidate the instruments for which it is responsible at more frequent intervals.

3. The attached proposal of the Commission for a consolidation of Council Directive 72/276/EEC on the approximation of the laws of the Member States relating to certain methods for the quantitative analysis of binary textile fibre mixtures has been drafted in accordance with the fundamental principles agreed by Council, Parliament and the Commission in 1974; it aims at legislative consolidation: the existing Directives would be replaced by one new one, which would leave their substance untouched but would assemble them into a single text, with only the formal amendments required by the operation itself. This codified text will serve as the basis for future legislative developments in this field.

4. As in the past the text supplied here is collated from the original Directives as published in the Official Journal; the use of photocopies means that any improvements to the wording are immediately identifiable. The old numbering of the Articles has been retained in the margin for ease of reference, the new numbering being entered above the Articles: Annex IV contains a concordance table relating the old system of numbering to the new. In order to preserve the dates for transposal of all the Directives concerned, a new Annex (Annex III, part B) lists the deadline for implementation of each of the Directives now being repealed.

COUNCIL DIRECTIVE 72/276/EEC

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

(OJ No L 173, 31.07.1972, p. 1)

modified by Directives

79/76/EEC (OJ n° L 17, 24.01.1979, p. 17)
81/75/EEC (OJ n° L 57, 4.03.1981, p. 23)
87/184/EEC (OJ n° L 75, 17.03.1987, p. 21)

modified by Acts of Accession

of Greece (OJ n° L 291, 19.11.1979, p. 108)

of Spain and Portugal (JO n° L 302, 15.11.1985, p. 218)

Proposal for a
EUROPEAN PARLIAMENT AND COUNCIL DIRECTIVE

on certain methods for the quantitative analysis of binary textile fibre mixtures.

THE EUROPEAN PARLIAMENT AND THE COUNCIL OF THE EUROPEAN UNION,

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

Having regard to the proposal from the Commission,

Having regard of the opinion of the Economic and Social Committee (1),

1) **Whereas Council Directive 72/276/EEC 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**(2), as last amended by Directive 87/184/EEC(3) has been amended frequently and substantially; whereas for reasons of clarity and rationality the said Directive should be consolidated;

(1) OJ No C
(2) OJ No L 173, 31.7.1972, p. 1.
(3) OJ No L 75, 17.3.1987, p. 21.

2) Whereas European Parliament and Council Directive .../.../EC of ..... 19.. on textile names (1) requires labelling to indicate the nature of the fibres in textile products, checks on the conformity of these products with the indications given on the label being carried out by analysis;

1.	87/184/EEC (adapted)

3) 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;

2.	72/276/EEC

4) Whereas Directive .../.../EC 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;

3.	72/276/EEC (adapted)

whereas therefore Annex II of this Directive lays down 15 uniform methods of analysis for most of the textile products composed of binary mixtures that are on the market;

2.	87/184/EEC (adapted)

5) 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;

5.	72/276/EEC

6) 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;

6.	72/276/EEC

7) Whereas the provisions of this Directive are in accordance with the opinion of the Committee for Directives relating to Textile Names and Labelling;

5.	87/184/EEC

(1) OJ No L , p.

8) Whereas this Directive must not affect the obligations of the Member States concerning the deadlines for transposal of the Directives set out in Annex III, part B,

HAVE ADOPTED THIS DIRECTIVE:

72/276/EEC

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

Member States shall take all necessary steps to ensure that, in accordance with the Directive ../../EC, 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 tests to determine the composition of textile products put on the market. (adapted)

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. A Committee for the Adaptation of Methods of Textile Analysis to Developments in Technology (hereinafter called the 'Committee') is hereby set up; 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 technological developments in the methods of quantitative analysis provided for in Annex II shall be made in accordance with the procedure laid down in Article 6.

72/276/EEC (adapted)

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 54 votes, the votes of the Member States being weighted as provided in Article 148 (2) of the Treaty. → Act of Accession ES, PO

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

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

Article 8

The Directives listed in Annex III, part A are ——— repealed, without prejudice to the obligations of the Member States concerning the deadlines for transposal set out in Annex III, part B.

References to the repealed Directives shall be construed as references to this Directive and should be read in accordance with the correlation table set out in Annex IV.

Article 9

This Directive is addressed to the Member States.

This Directive shall enter into force on the twentieth day following that of its publication in the Official Journal of the European Communities.

72/276/EEC

Done at Brussels,

For the European Parliament

The President

For the Council

The President

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 matter[1].

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 laboratory[2].

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 sample[3].

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.

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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 $\frac{10^4}{Nt}$ 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 $\frac{10^5}{Nt}$ 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 selvedges. This strip is the laboratory test sample. To obtain a laboratory test sample of x g, the strip area shall be $\frac{x.10^4}{G}$ 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.

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

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.

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 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.

ANNEX II

72/276/EEC

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 textile names. (adapted)

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° ± 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 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.

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

Where a substance not to be taken into account in the percentage calculations (see Article 12(3) 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. (adapted)

[1] Method 12 is an exception. It is based on a determination of the content of a constituent substance of one of the two components.

2 See Annex I.1.

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.

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 ± 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.

I.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.

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 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

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.

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

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.

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

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.

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 selvedges, 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

72/276/EEC

Method	Field of application		Reagent	
No 1	Acetate	Certain other fibres	Acetone	
No 2	Certain protein fibres	Certain other fibres	Alkaline sodium hypochlorite	
No 3	Viscose, cupro or certain types of modal	Cotton	Zinc chloride Formic acid	
No 4	Polyamide or Nylon	Certain other fibres	Formic acid, 80% m/m	(adapted)
No 5	Acetate	Triacetate	Benzyl alcohol	
No 6	Triacetate	Certain other fibres	Dichloromethane	
No 7	Certain cellulose fibres	Polyester	Sulphuric acid, 75% m/m	
No 8	Acrylics, certain modacrylics or certain chlorofibres	Certain other fibres	Dimethylformamide	
No 9	Certain chlorofibres	Certain other fibres	Carbon disulphide/ acetone, 55·5/44·5 v/v	
No 10	Acetate	Certain chlorofibres	Glacial acetic acid	
No 11	Silk	Wool or hair	Sulphuric acid, 75% m/m	
No 12	Jute	Certain animal fibres	Nitrogen content method	
No 13	polypropylene	certain other fibres	xylene	81/75/EEC
No 14	chlorofibres (homopolymers ofn vinyl chloride)	certain other fibres	concentrated sulphuric acid method	81/75/EEC
No 15	chlorofibres, certain modacrylics, certain elastanes, acetates, triacetalates	certain other fibres	cyclohexanone	87/184/EEC

72/276/EEC

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 (19)

   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), ramie (14), sisal (15), cupro (21), modal (22), protein (23), viscose (25), acrylic (26), polyamide or nylon (30) and polyester (31).

(adapted)

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

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 is 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 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), protein (23)

   with

2. cotton (5), cupro (21), modal (22), viscose (25), acrylic (26), chlorofibres (27), polyamide or nylon (30), polyester (31), polypropylene (33), elastane (39) and glass fibre (40).

If different 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 know dry mass of the mixture, with a hypochlorite solution. 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.

Either lithium hypochlorite or sodium hypochlorite can be used for the preparation of the hypochlorite solution.

Lithium hypochlorite is recommended in cases involving a small number of analyses or for analyses conducted at fairly lengthy intervals. This is because the percentage of hypochlorite in solid lithium hypochlorite — unlike that in sodium hypochlorite — is virtually constant. If the percentage of hypochlorite is known, hypochlorite content need not be checked iodometrically for each analysis, since a constant weighed portion of lithium hypochlorite can be employed.

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

3.1. Apparatus

(i) Erlenmeyer flask with ground-glass stopper, 250 ml;

(ii) Thermostat, adjustable to 20 (± 2) °C.

3.2. Reagents

(i) Hypochlorite reagent

(a) Lithium hypochlorite solution

This consists of a freshly prepared solution containing 35 (± 2) g/l of active chlorine (approximately 1 M), to which 5 (± 0,5) g/l of previously dissolved sodium hydroxide is added. To prepare, dissolve 100 grams of lithium hypochlorite containing 35 % active chlorine (or 115 grams containing 30 % active chlorine) in approximately 700 ml of distilled water, add 5 grams of sodium hydroxide dissolved in approximately 200 ml of distilled water and make up to 1 litre with distilled water. The solution which has been freshly prepared needs not be checked iodometrically;

(b) Sodium hypochlorite solution

This consists of a freshly prepared solution containing 35 (± 2) g/l of active chlorine (approximately 1 M) to which 5 (± 0,5) g/l of previously dissolved sodium hydroxide is added.

Check the active chlorine content of the solution iodometrically before each analysis;

(ii) *Acetic acid, dilute solution*

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

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows: mix approximately 1 gram of the sample with approximately 100 ml of the hypochlorite solution (lithium or sodium hypochlorite) in the 250 ml flask and agitate thoroughly in order to wet out the sample.

Then heat the flask for 40 minutes in a thermostat at 20 °C and agitate continuously, or at least at regular intervals. Since the dissolution of the wool proceeds exothermically, the reaction heat of this method must be distributed and removed. Otherwise, considerable errors may be caused by the incipient dissolution of the non-soluble fibres.

After 40 minutes, filter the flask contents through a weighed glass-filter crucible and transfer any residual fibres into the filter crucible by rinsing the flask 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 with 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, except for cotton, viscose and modal, for which d = 1,01, and unbleached cotton, for which d = 1,03.

6. PRECISION .

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

72/276/EEC

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 (25) or cupro (21), including certain types of modal fibre (22), 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

79/76/EEC

(i) Solution containing 20 g of fused anhydrous zinc chloride and 68 g of anhydrous formic acid made up to 100 g with water (namely 20 parts by mass of fused anhydrous zinc chloride to 80 parts by mass of 85 % m/m formic acid).

Attention is drawn, in this respect, to Annex II (1), point I.3.2.2, which lays down that all reagents used should be chemically pure; in addition, it is essential to use only fused anhydrous zinc chloride.

72/276/EEC

(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.

79/76/EEC

Rinse the fibrous residue in approximately 100 ml of cold ammonia solution (3.2.ii) ensuring that this residue remains wholly immersed in the solution for 10 minutes (1); then rinse thoroughly with cold water.

(1) To ensure that the fibrous residue is immersed in the ammonia solution for 10 minutes, one may for example, use a filter crucible adaptor fitted with a tap by which the flow of the ammonia solution can be regulated.

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.

72/276/EEC

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of 'd' for cotton is 1·02.

79/76/EEC

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%.

72/276/EEC

72/276/EEC

METHOD No 4

POLYAMIDE OR NYLON AND CERTAIN OTHER FIBRES (adapted)

(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. Polyamide or nylon (30)
with (adapted)
2. wool (1), animal hair (2 and 3), cotton (5), cupro (21), modal (22), viscose (25), acrylic (26), chlorofibre (27), polyester (31), polypropylene (33) and glass fibre (40).

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 polyamide or nylon is found by difference. (adapted)

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 (19)

with

— triacetate (24)

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%.

72/276/EEC

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 (24)
   with
2. wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (21), modal (22), viscose (25), acrylic (26), polyamide or nylon (30), polyester (31) and glass fibre (40). (adapted)

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 suction to eliminate excess liquid, then treat the residue with boiling water to eliminate all the solvent, apply suction, dry the crucible and residue, cool and weigh them. 79/76/EEC

72/276/EEC

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 (14), cupro (21), modal (22), viscose (25) with
2. polyester (31).

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

72/276/EEC

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 (26), certain modacrylics (29), or certain chlorofibres (27)[1]

with

2. wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (21), modal (22), viscose (25), Polyamide and polyester (31). or nylon (30) (adapted)

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 ± 1 °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 fibre to the crucible by washing out the beaker with dimethylformamide. Drain the crucible with suction. Wash the residue with about 1 litre of hot water at 70 — 80 °C, filling the crucible each time. After each addition of water, apply suction briefly but not until the water has drained under gravity. If the washing liquor drains through the crucible too slowly, slight suction may be applied.

87/184/EEC

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

72/276/EEC

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

5. CALCULATION AND EXPRESSION OF RESULTS

87/184/EEC

Calculate the results as described in the general instructions. The value of "d" is 1,00 except in the following cases :

wool	1,01
cotton	1,01
cupro	1,01
modal	1,01
polyester	1,01.

72/276/EEC

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 (27), namely certain polyvinal chloride fibres, whether after-chlorinated or not[1]

   with

2. wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (21), modal (22), viscose (25), acrylic (26), polyamide or nylon (30), polyester (31), glass fibre (40). (adapted)

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

When the polyamide or nylon content of the mixture exceeds 25%, method No 4 should be used. (adapted)

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.

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 resh 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.

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

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 elemination of non-fibrous matter, to binary mixtures of:

1. acetate (19)

with

2. certain chlorofibres (27) 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.

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%.

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

METHOD No 12

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: 1·84[1]

(iv) Potassium sulphate[1]

(v) Selenium dioxide[1]

(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.

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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:

$$P_A = \frac{A - 0{\cdot}22}{16{\cdot}2 - 0{\cdot}22} \times 100$$

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%.

METHOD No 13

POLYPROPYLENE FIBRES AND CERTAIN OTHER FIBRES

(Xylene method)

81/75/EEC

1. FIELD OF APPLICATION

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

1. polypropylene fibres (33)

with

2. wool (1), animal hair (2 and 3), silk (4), cotton (5), acetate (19), cupro (21), modal (22), triacetate (24), viscose (25), acrylic (26), polyamide or nylon (30), polyester (31) and glass fibre (40).

2. PRINCIPLE

The polypropylene fibre is dissolved out from a known dry mass of the mixture with boiling xylene. 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 polypropylene 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) Reflux condenser (suitable for liquids of high boiling point), fitting the conical flasks (i).

3.2. Reagent

Xylene distilling between 137 and 142° C.

Note:

This reagent is highly flammable and has a toxic vapour. Suitable precautions must be taken in its use.

4. TEST PROCEDURE

Follow the procedure described in the general instructions, then proceed as follows:

To the specimen contained in the conical flask (3.1. (i)), add 100 ml of xylene (3.2) per gram of specimen. Attach the condenser (3.1. (ii)), bring the contents to the boil and maintain at boiling point for three minutes. Immediately decant the hot liquid through the weighed filter crucible (see note 1). Repeat this treatment twice more, each time using a fresh 50 ml portion of solvent.

Wash the residue remaining in the flask successively with 30 ml of boiling xylene (twice), then with 75 ml of light petroleum (I.3.2.1 of general instructions) (twice). After the second wash with light petroleum, filter the contents of the flask through the crucible, transfer any residual fibres to the crucible with the aid of a small quantity of light petroleum and allow the solvent to evaporate. Dry the crucible and residue, cool and weigh them.

Notes:

1. The filter crucible through which the xylene is to be decanted must be pre-heated.
2. After the treatment with boiling xylene, ensure that the flask containing the residue is cooled sufficiently before the light petroleum is introduced.
3. In order to reduce the fire and toxicity hazards to the operator, a hot extraction apparatus using the appropriate procedures, giving identical results, may be used (1).

(1) See for example the apparatus described in 'Melliand Textilberichte' 56 (1975), pp. 643 to 645.

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 14

CHLOROFIBRES (HOMOPOLYMERS OF VINYL CHLORIDE) AND CERTAIN OTHER FIBRES

81/75/EEC

(Concentrated sulphuric acid method)

1. FIELD OF APPLICATION

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

1. chlorofibres (27) based on homopolymers of vinyl chloride (after-chlorinated or not) with
2. cotton (5), acetate (19), cupro (21), modal (22), triacetate (24), viscose (25), certain acrylics (26), certain modacrylics (29), polyamide or nylon (30) and polyester (31).

The modacrylics concerned are those which give a limpid solution when immersed in concentrated sulphuric acid (relative density 1·84 at 20° C).

This method can be used in place of Method Nos 8 and 9.

2. PRINCIPLE

The constituent other than the chlorofibre (i.e. the fibres mentioned under point 2 of paragraph 1) is dissolved out from a known dry mass of the mixture with concentrated suphuric acid (relative density 1·84 at 20° C). The residue, consisting of the chlorofibre, 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 the second constituent is obtained 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) Glass rod with flattened end.

3.2. Reagents

(i) Sulphuric acid, concentrated (relative density 1·84 at 20° C).

(ii) Sulphuric acid, approximately 50 % (m/m) aqueous solution.
Prepare by adding carefully, while cooling, 400 ml of sulphuric acid (relative density 1·84 at 20° C) to 500 ml of distilled or de-ionized water. After cooling to room temperature, dilute the solution to one litre with water.

(iii) Ammonia, dilute solution.
Dilute 60 ml of concentrated ammonia solution (relative density 0·880 at 20° C) to one litre with distilled water.

4. TEST PROCEDURE

Follow the procedure described in the general instructions, then proceed as follows:

To the specimen contained in the flask (3.1.(i)) add 100 ml of sulphuric acid (3.2.(i)) per gram of specimen.

Allow the contents of the flask to remain at room temperature for 10 minutes and during that time stir the test specimen occasionally by means of the glass rod. If a woven or knitted fabric is being treated, wedge it between the wall of the flask and the glass rod and exert a light pressure in order to separate the material dissolved by the sulphuric acid.

Decant the liquid through the weighed filter crucible. Add to the flask a fresh portion of 100 ml of sulphuric acid (3.2.(i)) and repeat the same operation. Transfer the contents of the flask to the filter crucible and transfer the fibrous residue there with the aid of the glass rod. If necessary, add a little concentrated sulphuric acid (3.2.(i)) to the flask in order to remove any fibres adhering to the wall. Drain the filter crucible with suction; remove the filtrate by emptying or changing the filter-flask, wash the residue in the crucible successively with 50 % sulphuric acid solution (3.2.(ii)), distilled or de-ionized water (1.3.2.3 of the general instructions, ammonia solution (3.2.(iii)) and finally wash thoroughly with distilled or de-ionized water, draining the crucible with suction after each addition. (Do not apply suction during the washing operation, but only after the liquid has drained off by gravity.)

Dry the crucible and residue, cool and weigh them.

Do not apply suction during or between the washing operations. Allow the liquid to drain under gravity and then apply suction.

Finally dry the crucible with the residue, 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 with the following exceptions :

silk 1,01

acrylic 0,98.

6. PRECISION

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

METHOD No 15

87/184/EEC

CHLOROFIBRES, CERTAIN MODACRYLICS, CERTAIN ELASTANES, ACETATES, TRIACETATES AND CERTAIN OTHER FIBRES

(Method using cyclohexanone)

1. FIELD OF APPLICATION

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

1. acetate (19), triacetate (24), chlorofibre (27), certain modacrylics (29), certain elastanes (39)

with

2. wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (21), modal (22), viscose (25), polyamide or nylon (30), acrylic 26 and glass fibre (40).

Where modacrylics or elastanes are present a preliminary test must first be carried out to determine whether the fibre is completely soluble in the reagent.

It is also possible to analyze mixtures containing chlorofibres by using method No 9 or 14.

2. PRINCIPLE

The acetate and triacetate fibres, chlorofibres, certain modacrylics, and certain elastanes are dissolved out from a known dry mass with cyclohexanone at a temperature close to boiling point. The residue is collected, washed, dreid and weighed ; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of chlorofibre, modacrylic, elastane, acetate and triacetate is found by difference.

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

3.1. Apparatus

(i) Hot extraction apparatus suitable for use in the test procedure in section 4. (See figure : this is a variant of the apparatus described in *Melliand Textilberichte* 56 (1975) 643-645);

(ii) Filter crucible to contain the specimen ;

(iii) Porous baffle (porosity grade 1);

(iv) Reflux condenser that can be adapted to the distillation flask ;

(v) Heating device.

3.2. Reagents

(i) Cyclohexanone, boiling point 156 °C ;

(ii) Ethyl alcohol, 50 % by volume.

NB : Cyclohexanone is flammable and toxic. Suitable precautions must be taken in its use.

4. TEST PROCEDURE

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

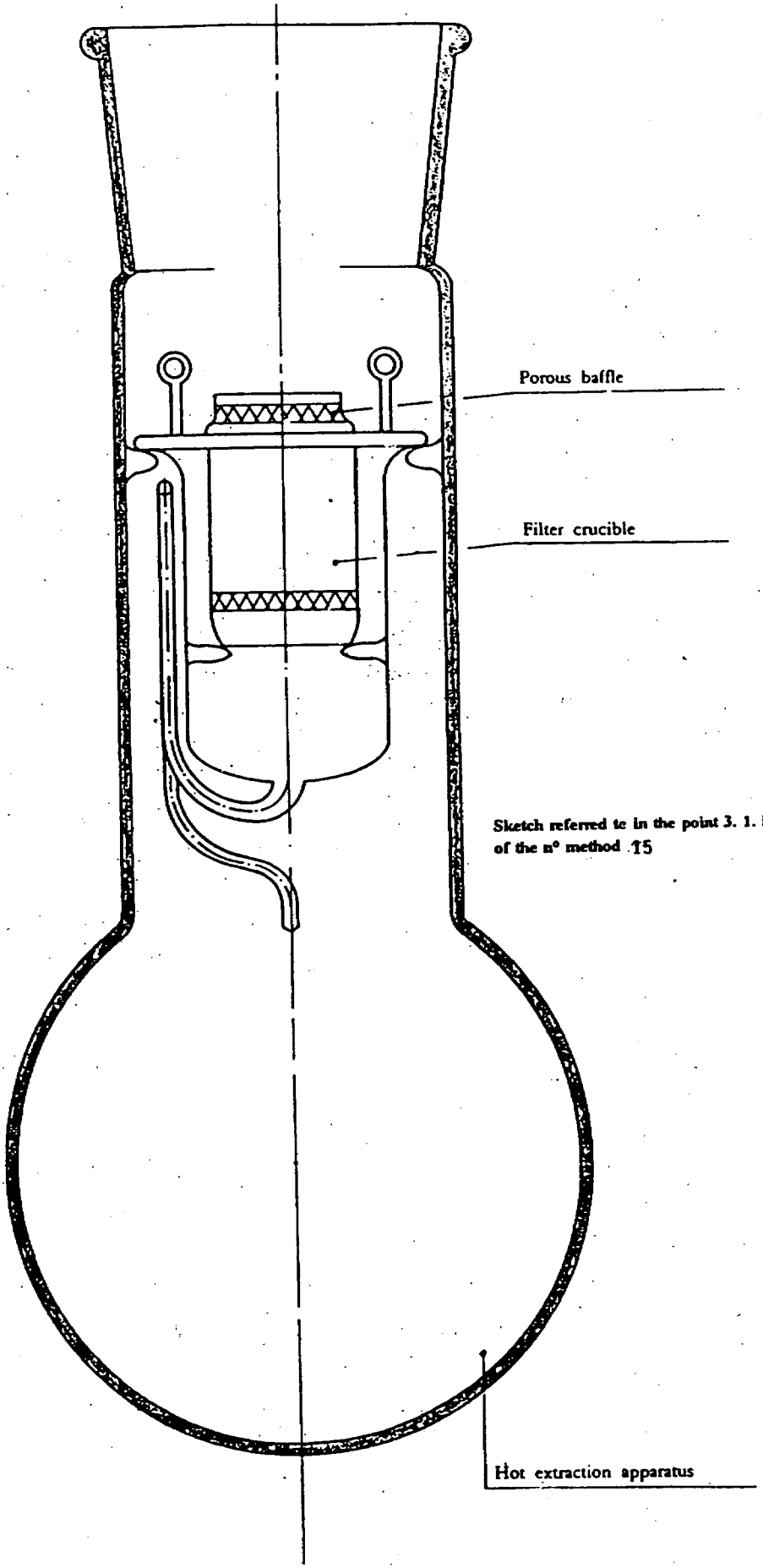
Pour into the distillation flask 100 ml of cyclohexanone per gram of material, insert the extraction container in which the filter crucible, containing the specimen and the porous baffle, slightly inclined, have previously been placed. Insert the reflux condenser. Bring to the boil and continue extraction for 60 minutes at a minimum rate of 12 cycles per hour. After extraction and cooling remove the extraction container, take out the filter crucible and remove the porous baffle. Wash the contents of the filter crucible three or four times with 50 % ethyl alcohol heated to about 60 °C and subsequently with 1 litre of water at 60 °C.

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 %.



Sketch referred to in the point 3. 1. I) of the n° method 15

ANNEX III

PART A

REPEALED DIRECTIVES
(referred to in Article 8)

- Directive 72/276/EEC

and their successive amendments:

- Directive 79/76/EEC

- Directive 81/75/EEC

- Directive 87/184/EEC.

ANNEX III

PART B

TIME LIMITS FOR TRANSPOSAL

Directive	Deadline for implementation
- 72/276/EEC (OJ No L 173, 31.7.1972, p. 1)	18 January 1974
- 79/76/EEC (OJ No L 17, 24.1.1979, p. 17)	28 June 1979
- 81/75/EEC (OJ No L 57, 4.3.1981, p. 23)	27 February 1982
- 87/184/EEC (OJ n° L 75, 17.3.1987, p. 21)	1 September 1988

ANNEX IV

CORRELATION TABLE

This Directive	Directive 72/276/EEC
Article 1	Article 1
Article 2	Article 2
Article 3	Article 3
Article 4	Article 4
Article 5	Article 5
Article 6	Article 6
Article 7	Article 7 (2)
Article 8	---
Article 9	Article 8
Annex I	Annex I
Annex II (1)	Annex II (1)
Annex II (2)	Annex II (2)
Annex II method No 1	Annex II method No 1
Annex II method No 2	Annex II method No 2
Annex II method No 3	Annex II method No 3
Annex II method No 4	Annex II method No 4
Annex II method No 5	Annex II method No 5
Annex II method No 6	Annex II method No 6
Annex II method No 7	Annex II method No 7
Annex II method No 8	Annex II method No 8
Annex II method No 9	Annex II method No 9
Annex II method No 10	Annex II method No 10
Annex II method No 11	Annex II method No 11
Annex II method No 12	Annex II method No 13
Annex II method No 13	Annex II method No 14
Annex II method No 14	Annex II method No 15
Annex II method No 15	Annex II method No 16
Annex III	----
Annex IV	----

47

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