

# **RULEBOOK**

## **ON THE MARKING AND LABELING OF TEXTILE PRODUCTS**

*("Official Gazette of the RS", No. 1/2014)*

### **I INTRODUCTORY PROVISIONS**

#### ***Scope of the Rulebook***

##### **Article 1**

This Rulebook lays down requirements concerning the marking and the labelling of fibre composition of textile products, requirements concerning the use of textile fibre names and related marking and labelling, the marking and the labelling of textile products containing non-textile parts of animal origin and requirements concerning the determination of the fibre composition of textile products that are marked or labelled in accordance with this Rulebook, as well as requirements for additional marking and labelling of the procedure and the method used in the maintenance of textile articles of apparel, the information about shrinking during maintenance, as well as the information about the size of textile articles of apparel.

#### ***Definitions***

##### **Article 2**

For the purposes of this Rulebook, the following definitions shall apply:

- 1) *textile product* means any raw, semi-manufactured or manufactured product, either factory or handmade, which is exclusively composed of textile fibres, regardless of the manufacturing process employed;
- 2) *textile fibre* means:
  - (1) a fibre that is characterized by flexibility, fineness and higher ratio of length to maximum transverse dimension, which render it suitable for textile applications, or
  - (2) a flexible strip or tube, of which the apparent width does not exceed 5 mm, including strips cut from wider strips or films, produced from the substances used for the manufacture of the fibres listed in Table 2 of Annex 1 – The list of textile fibre names (hereinafter referred to as: Annex 1), which is printed with this Rulebook and forms an integral part thereof;
- 3) *apparent width* means the width of the strip or tube when folded, flattened or twisted, or the average width where the width is not uniform;

- 4) *textile component* means a part of a textile product with identifiable fibre content;
- 5) *extraneous fibres* means fibres other than those stated on the label or marking of a textile product;
- 6) *lining* means a separate component used in making up garments and other textile products, consisting of a single layer or multiple layers of textile material held in place along one or more of the edges;
- 7) *labelling* means affixing the required information referred to in Article 1 of this rulebook to the textile product by way of attaching a label containing that information;
- 8) *marking* means indicating the required information referred to in Article 1 of this rulebook directly on the textile product by way of embossing, sewing, embroidering, printing or any other technology of application;
- 9) *inclusive labelling* means the use of a single label which contains all the information about the composition of several textile products of the same kind or components included in the inclusive packaging;
- 10) *disposable product* means a textile product designed to be used only once for a limited period of time, and it is not intended for subsequent use for the same or a similar purpose;
- 11) *agreed allowance* means the value of moisture content, expressed in percentage referred to in Article 9 – The Agreed allowance used in the calculation of fibre mass contained in a textile product (hereinafter referred to as: Annex 9) which is printed with this rulebook and forms its integral part thereof, and which is added in the procedure of calculating the percentage of fibre mass contained in a textile product;
- 12) *manufacturer* means a legal person, an entrepreneur or a physical person who manufactures a textile product or a person who presents itself as a manufacturer by placing its company name, a name, a logo, some other recognizable markings or in some other way;
- 13) *representative* means a legal person or an entrepreneur registered in the Republic of Serbia, or a physical person with the place of residence in the Republic of Serbia authorized in writing by the manufacturer of a textile product to perform activities with authorisation with regard to the placing of a textile product on the market of the Republic of Serbia;
- 14) *importer* means a legal person or an entrepreneur registered in the Republic of Serbia, or a physical person with the place of residence in the Republic of Serbia placing textile products from other countries on the market;
- 15) *distributor* means a legal person or an entrepreneur registered in the Republic of Serbia, or a physical person with the place of residence in the Republic of Serbia included in the chain of supply and delivering textile products within the scope of its tasks though it is neither a manufacturer nor an importer;
- 16) *supplier* means a manufacturer, a representative, an importer or a distributor;

17) *making available on the market* means making any textile product available on the market of the Republic of Serbia for the purpose of distribution, consumption or use, whether in return for payment or free of charge;

18) *placing on the market* means making any textile product available for the first time on the market of the Republic of Serbia;

19) *consumer* means any physical person that buys goods or services for the purpose of fulfilling personal demands or household demands;

20) *article of apparel* means any textile product used for dressing or body covering;

21) *commercial documentation* means business records, registers, invoices, accounting money orders, shipping and accompanying documents, receipts, financial statements, as well as annual and periodical reports, audit and reporting protocols, records about production and quality and correspondence including electronic mail relating to business operations of the entity, as well as the information about the trade in any form, including the information saved in electronic form;

22) *this cannot be easily stated* means that resources and costs of such determining exceed economic justification of that procedure;

23) *good manufacturing practice* means the practice of a manufacturer as a caring business entity.

Other definitions used in this rulebook, which are not defined in Paragraph 1 of this article have the meaning defined by laws that arrange technical requirements for products and conformity assessment, consumer protection, trade, general safety of products and standardization.

### ***Scope of application***

#### **Article 3**

This rulebook shall apply to all textile products which are placed on the market of the Republic of Serbia, as well as to products which are, for the purpose of this rulebook, considered textile products, in particular:

1) products containing at least 80% by weight of textile fibres;

2) furniture, umbrella and sunshade coverings containing at least 80% by weight of textile fibres in components of those products which contain textile fibres;

3) the textile components of:

(1) the upper layer or multilayer floor coverings;

(2) mattress coverings,

(3) coverings of camping goods,

provided such textile components constitute at least 80% by weight of textile fibres;

4) textiles incorporated in other products and forming an integral part thereof, where their composition is specified.

### ***Textile products to which this Rulebook shall not apply***

#### **Article**

#### **4**

This rulebook shall not apply to:

- 1) textile products which are manufactured by persons working in their own homes;
- 2) textile products which are manufactured by persons that carry out work from materials supplied without the property;
- 3) customised textile products made up by self-employed tailors.

If requirements for textile products that this rulebook applies to are fully or partially defined by other special regulations, those special regulations shall then apply to those requirements.

## **II TEXTILE FIBRE NAMES AND RELATED LABELLING AND MARKING REQUIREMENTS**

### ***General requirements for marking or labelling textile products***

#### **Article 5**

Textile products shall only be made available on the market provided that such products are labelled or marked in compliance with requirements of this rulebook.

Exceptionally, textile products which are made available on the market within the wholesale trade, as well as textile products which are made available on the market in accordance with the regulation that governs public procurements, can be made available on the market even if they are not marked or labelled, provided that they are accompanied by commercial documents which contain the information referred to in Article 1 of this rulebook.

### ***Textile fibre names***

#### **Article 6**

Only the textile fibre names listed in Annex 1 shall be used for the description of fibre composition on labels and markings of textile products.

Names listed in Annex 1 shall be used for textile fibres the nature of which corresponds to the description set out in that annex.

The names listed in Annex 1 shall not be used for other fibres, whether on their own or as a root or as an adjective.

The term “silk” shall not be used to indicate the shape of fibres or for the purpose of the description of fibres in the form of a filament but which do not correspond to the composition of silk listed in Annex 1 of this rulebook.

### ***Pure textile products***

#### **Article 7**

Only textile products exclusively composed of the same fibre may be labelled or marked as “100%”, “pure” or “all”, with stating the fibre name listed in Annex 1.

Terms listed in Annex 1 of this Article shall not be used for textile products which are not exclusively composed of the same fibre.

A textile product containing no more than 2 % by weight of extraneous fibres may also be treated as exclusively composed of the same fibre, provided this quantity is justified as being technically unavoidable in good manufacturing practice.

The provision referred to in Paragraph 3 of this article shall not be applied to textile products composed of a mixture of textile fibres of which pure fleece wool is one of the components.

A textile product which has undergone a carding process may also be treated as exclusively composed of the same fibre if it contains no more than 5 % by weight of extraneous fibres, provided this quantity is justified as being technically unavoidable in good manufacturing practice.

### ***Products made of pure fleece wool***

#### **Article 8**

A textile product is labelled or marked as “pure fleece wool” in accordance with Annex 3 – Names of textile products made of pure fleece wool, which is printed with this Rulebook and forms an integral part thereof (hereinafter referred to as: Annex 3), provided it is composed exclusively of a wool fibre which has not previously been incorporated in a finished product, which has not been subjected to any spinning or felting (rolling) processes other than those required in the manufacture of that product, and which has not been damaged by treatment or use.

Exceptionally, the name “pure fleece wool” may be used to describe wool contained in a textile product made of textile fibre mixture if all of the following conditions are met:

- 1) all the wool contained in that textile product meets the requirements regarding wool fibres defined in Paragraph 1 of this article;
- 2) pure fleece wool contained in that textile product accounts for not less than 25% of the total weight of that textile product;
- 3) only pure fleece wool and only one other fibre produced by combing procedure of processing is contained in that textile product

The full percentage composition of such textile product referred to in Paragraph 2 of this article shall be given.

The extraneous fibres in the textile products referred to in paragraphs 1 and 2 of this article, including wool products which have undergone a carding process, shall not exceed 0.3% by weight and shall be justified as being technologically unavoidable in good manufacturing practice.

### ***Multi-fibre textile products***

#### **Article 9**

A textile product which contains several types of textile fibres shall be marked or labelled:

- 1) by stating names of all constituent textile fibres in a textile product, in accordance with Annex 1, and
- 2) by stating percentage by weight of all constituent textile fibres in a textile product in descending order.

A fibre which accounts for up to 5 % of the total weight of the textile product, or fibres which collectively account for up to 15 % of the total weight of the textile product, may, where percentage of that (those) other fibres cannot easily be stated at the time of the manufacture, be designated by the term 'other fibres'.

Total percentage of those fibres by weight shall be stated accompanied with the term 'other fibres'.

Products having a pure cotton warp and a pure flax weft, in which the percentage of flax accounts for at least 40 % of the total weight of the unsized fabric may be given the name 'cotton linen union' which must be accompanied by the composition specification 'pure cotton warp — pure flax (or linen) weft'.

For textile products the composition of which is hard to state at the time of their manufacture, the term 'mixed fibres' or the term 'unspecified textile composition' may be used on the label or marking.

Fibres not yet listed in Annex 1 may be designated by the term 'other fibres', immediately preceded or followed by the total percentage of all such fibres by weight of a textile product.

### ***Decorative fibres and fibres with antistatic effect***

#### **Article 10**

Visible, isolable fibres which are purely decorative and do not exceed 7 % of the weight of the finished textile product do not have to be taken into account in the fibre compositions provided for in Articles 7 and 9 of this rulebook.

Metallic fibres and other fibres which are incorporated in order to obtain an antistatic effect and which do not exceed 2 % of the weight of the finished product do not have to be taken into account in the fibre compositions provided for in Articles 7 and 9 of this rulebook.

In the case of textile products the composition of which is hard to state at the time of their manufacture, the percentages provided for in Paragraphs 1 and 2 of this article shall be calculated on the weight of the warp and that of the weft separately.

### ***Multi-component textile products***

#### **Article 11**

Any textile product containing two or more textile components which have different textile fibre contents shall bear a label or marking stating the textile fibre content of each textile component.

The labelling or marking referred to in Paragraph 1 of this article shall not be compulsory for textile components when the following two conditions are fulfilled:

- 1) those textile components are not main linings,
- 2) those textile components represent less than 30% of the total weight of the textile product.

Where two or more textile products have the same fibre content and normally form a single unit, they may bear only one label or marking.

### ***Textile products containing non-textile parts of animal origin***

#### **Article 12**

The presence of non-textile parts of animal origin in textile products shall be indicated by using the phrase 'Contains non-textile parts of animal origin' on the labelling or marking of products containing such non-textile parts whenever they are made available on the market.

The labelling or marking of the composition of textile products shall not be misleading and shall be carried out in such a way that the consumer can easily understand.

### ***Labelling and marking of specific textile products***

#### **Article 13**

The labelling and marking of the composition of specific textile products referred to in Annex 4 – Requirements for the labelling and marking of specific textile products, which is printed with this rulebook and forms an integral part thereof (hereinafter referred to as Annex 4), shall be indicated in accordance with that annex.

### ***Labels and markings***

#### **Article 14**

Textile products shall be labelled or marked to give an indication of their fibre composition whenever they are made available on the market.

The labelling and marking of textile products shall be durable, easily legible, visible and accessible and, in the case of a label, securely attached.

Without prejudice to Paragraph 1 of this article, labels or markings may be replaced or supplemented by accompanying commercial documents when the products are being supplied to economic operators within the supply chain, or when they are delivered in accordance with regulations arranging public procurements. In that case, the textile fibre names and descriptions of fibre compositions referred to in Articles 6, 7, 8, and 9 of this rulebook shall be clearly indicated in the accompanying commercial documents.

Abbreviations for textile fibre names and for descriptions of fibre composition shall not be used, except in the case of indicating the composition of textile products in accompanying commercial documents referred to in Paragraph 3 of this article, in accordance with Serbian standard SRPS ISO 2076, provided that the abbreviations are explained in the same commercial document.

### ***Obligation to supply the label or marking***

#### **Article 15**

When placing a textile product on the market, the manufacturer or its representative shall ensure the supply of the label or marking and the accuracy of the information contained therein.

If the manufacturer or its representative is not established in the Republic of Serbia, the importer shall ensure the supply of the label or marking and the accuracy of the information contained therein.

When making a textile product available on the market, the distributor shall ensure that textile products bear the appropriate labelling or marking prescribed by this rulebook.

Any additional information supplied when textile products are made available on the market cannot be confused with the textile fibre names and the descriptions of fibre compositions, as laid down by this rulebook.

### ***Textile fibre composition descriptions***

#### **Article 16**

Textile products referred to in Articles 6, 7, 8, and 9 of this rulebook that are made available on the market include the description of the composition.

The textile fibre composition descriptions shall be indicated in catalogues and trade literature, on packaging, labels and markings in a manner that is easily legible, clear, visible and in print which is uniform as regards its size, style and font.

The textile fibre composition descriptions referred to in Paragraph 2 of this article shall be clearly visible to the consumer before the purchase, including in cases where the purchase is made by electronic means.

The name of the undertaking or trade marks of the textile product may be given immediately before or after textile fibre composition descriptions of textile products referred to in Articles 6, 7, 8 and 9 of this rulebook.



Without prejudice to Paragraph 4 of this article, where the name of an undertaking or a trade mark of a textile product contains, on its own or as a root or as an adjective, one of the textile fibre names listed in Annex 1 or a name liable to be confused therewith, such trade mark or name shall be given immediately before or after the textile fibre composition descriptions referred to in Articles 6, 7, 8 and 9 of this rulebook.

Other information about textile products, such as the information about the country of origin, special characteristic of products and alike, is always displayed separately on the same marking or label or on a separate marking or label.

The information displayed on the marking or label referred to in this article shall be in Serbian.

Exceptionally, if textile products are imported or intended for export, the information referred to in Paragraph 7 of this article may also be displayed in a foreign language.

In the case of bobbins, reels, skeins or other small quantities of thread, the information referred to in Paragraph 7 of this Article shall be displayed in a foreign language only if it applies to inclusive labelling of packagings of those products.

### ***Special requirements for marking or labelling - Derogations***

#### **Article 17**

The indication of textile fibre names or fibre composition on the labels and markings of textile products listed in Annex V - Textile products the marking or labelling of which is not necessary (hereinafter referred to as Annex 5), which is printed with this rulebook and forms the integral part thereof, is not required.

Without prejudice to Paragraph 1 of this article, where a trade mark or name of an undertaking contains, on its own or as a root or as an adjective, one of textile fibre names listed in Annex 1 or a name liable to be confused therewith, Articles 11, 14, 15 and 16 of this rulebook shall apply.

Where textile products are of the same type and fibre composition, as listed in Annex 6 – Textile products with an inclusive labelling (hereinafter referred to as Annex 6), which is printed with this rulebook and forms an integral part thereof, they may be made available on the market together, with an inclusive labelling.

The fibre composition of textile products sold by the metre may be shown on the length or roll made available on the market.

The textile products referred to in Paragraphs 3 and 4 of this article shall be made available on the market in such a way that the fibre composition of those products is made known to each purchaser in the supply chain, including the consumer.

## **III DETERMINING THE COMPOSITION OF TEXTILE PRODUCTS WHICH ARE MARKED OR LABELLED IN ACCORDANCE WITH THIS RULEBOOK**

***Determining the composition of textile products before making them available on the market***

**Article 18**

Before making textile products available on the market, the conformity of the composition of those products with the information displayed on their markings and labels shall be determined by applying the method of quantitative analysis of textile products composed of binary and ternary mixed fibres referred to in Annex 8 – Method of quantitative analysis of textile products composed of binary and ternary mixed fibres (hereinafter referred to as: Annex 8) which is printed with this rulebook and forms an integral part thereof, or by applying some other method or in other way.

***Inspection of textile products after delivery on the market for the purpose of determining the fibre composition***

**Article 19**

After making textile products available on the market and after their inspection, the composition of those products shall be determined exclusively by applying the method of quantitative analysis of textile products composed of binary and ternary mixed textile fibres referred to in Annex 8.

After making available on the market the textile products set out in Articles 7, 8 and 9 of this rulebook, in determination of fibre composition, the parts of textile products listed in Annex 7 – Parts of textile products which are not taken into account when determining the composition of textile products (hereinafter referred to as: Annex 7), which is printed with this rulebook and forms an integral part thereof, shall not be taken into account.

The composition of textile products referred to in Articles 7, 8, and 9 of this rulebook shall be determined by applying to the anhydrous mass of each fibre the appropriate agreed allowance laid down in Annex 9, after having removed the parts of textile products set out in Annex 7.

Without prejudice to Paragraphs 1-3 of this article, the determining of the composition of textile products, for which this rulebook does not prescribe the method of analyzing the composition, is performed by indicating in the analysis the results obtained, the methods used and the degree of accuracy.

***Tolerances in determining the fibre composition of textile products***

**Article 20**

For the purposes of establishing the fibre composition of textile products, the tolerances laid down in paragraphs 2-5 of this article shall apply.

With the exception of pure fleece wool and the mixture of textile fibres containing pure fleece wool, the presence of “extraneous fibres” referred to in Article 9 of this rulebook does not need to be indicated if the percentage of those fibres does not reach the following values:

1) 2% of the total weight of the textile product, provided this quantity is justified as being

technologically unavoidable in good manufacturing practice, or

2) 5% of the total weight in the case of textile products which have undergone a carding process, provided this quantity is justified as being technologically unavoidable in good manufacturing practice.

A manufacturing tolerance of 3 % shall be permitted between the stated fibre composition of a textile product to be provided in accordance with Article 9 of this rulebook and the percentages obtained from analysis carried out in accordance with Article 19 of this rulebook, in relation to the total weight of fibres shown on the label or marking.

The tolerance laid down in Paragraph 3 of this article shall apply to the following:

1) "other fibres" in accordance with Article 9 of this rulebook, and

2) the percentage of wool referred to in Article 8 Paragraph 2 point 2) of this rulebook.

The tolerances shall be calculated separately. The total weight to be taken into account in calculating the tolerance shall be that of the fibres of the finished textile product less the weight of any extraneous fibres found when applying the tolerance referred to in Paragraph 2 of this Article.

The cumulative application of the tolerances referred to in paragraphs 2 and 3 shall be permitted only if any extraneous fibres found by analysis, when applying the tolerance referred to in Paragraph 2, prove to be of the same chemical type as one or more of the fibres shown on the label or marking.

## IV ADDITIONAL MARKING AND LABELLING OF TEXTILE PRODUCTS

### ***Marking and labelling of the procedure and methods relating to care instructions for textile articles of apparel***

#### **Article 21**

Every textile product which is made available on the market of the Republic of Serbia shall be additionally marked or labelled to provide the information about the procedure and the method relating to care instructions, in accordance with requirements of Serbian standard SRPS EN ISO 3758 or in accordance with the information provided by the manufacturer or its representative.

If the imported textile product is not supplied with the information about the procedure and the method relating to care instructions, the importer shall provide such information on the marking or label.

### ***Marking and labelling the shrinkage in care instructions for textile articles of apparel***

#### **Article 22**

Every textile article of apparel which is made available on the market of the Republic of Serbia shall be additionally marked or labelled to provide care instructions, namely the information about the percentage of shrinking of that product, in accordance with requirements of Serbian standards SRPS EN ISO 5077 and SRPS EN ISO 3175-1 or in accordance with the information provided by the manufacturer or its representative.

If the imported textile article of apparel is not supplied with the information about the shrinkage percentage, the importer shall provide such information on the marking or label.

### ***Marking and labelling the size of textile articles of apparel***

#### **Article 23**

Every textile article of apparel which is made available on the market of the Republic of Serbia shall be additionally marked or labelled to provide the size of that textile article of apparel, in accordance with requirements of Serbian standards SRPS EN 13402-2, SRPS EN 13402-3, SRPS ISO 3636, SRPS ISO 3637, SRPS ISO 4415, SRPS ISO 4416 and SRPS ISO/TR 10652 or in accordance with the information provided by the manufacturer or its representative.

If the imported textile article of apparel is not supplied with the information about the size, such information shall be provided on the marking or labelling by the importer.

With the exception of Paragraphs 1 and 2 of this article, the supplier of bandanas, scarves, and similar products shall mark or label the width and the length of those products in centimetres (cm), or it shall mark or label the length of the longest side, provided that the product is not of rectangular shape.

### ***Putting the information on markings and labels***

#### **Article 24**

Information on markings and labels set out in Articles 21, 22, and 23 of this rulebook shall be put in such manner so they can be visible, legible and comprehensible to consumers, without diminishing visibility, legibility and comprehensibility of the information about the composition of the product.

Exceptionally, the information set out in Articles 21, 22 and 23 of this rulebook can be provided in the accompanying commercial documents when textile articles of apparel are delivered to wholesalers or in case those products are delivered in accordance with the regulation that governs public procurements. That information is also clearly indicated in accompanying commercial documents.

## **V SYNCHRONIZATION WITH REGULATIONS OF THE EUROPEAN UNION**

#### **Article 25**

This Rulebook is synchronized with all principles and relevant requirements as laid down in Directive No 1007/11 of the Parliament and the Council of Europe about the names of textile

fibres and the marking and labelling of the composition of textile products, that indicated the termination of Directive 73/44/EEC of the Council and Directive 96/73/EC and 2008/121/EC of the Parliament and the Council of Europe, as well as with the requirements of Directive of the European Commission No 286/2012 to supplement Directive No 1007/11 of the Parliament and the Council of Europe.

## V TRANSITIONAL AND FINAL PROVISIONS

### Article 26

Starting from the day of the accession of Serbia to the European Union, the manufacturer or its representative may submit a request to the European Commission to add the name of a new textile fibre to the list referred to in Annex 1.

The request referred to in Paragraph 1 of this article shall include technical document composed in accordance with Annex 2 – Minimum requirements for contents of the technical document submitted along with the request for the name of a new textile fibre, which is printed with this rulebook and forms an integral part thereof (hereinafter referred to as: Annex 2).

### Article 27

The following regulations shall cease to be valid starting from the day of putting into force this rulebook:

- 1) The Rulebook on mandatory attestation of jute and on requirements that must be met by organizations of associated labour authorized for jute attestation ("The Official Gazette of the SFRY", No 8/91);
- 2) Order on mandatory attestation of cotton ("The Official Gazette of the SFRY ", No. 65/84 and 44/88);
- 3) Order on mandatory attestation of wool ("The Official Gazette of the SFRY ", No. 65/84).

The following regulations shall cease to be valid starting from the day of applying this Rulebook:

- 1) Rulebook on technical and other requirements for marking, labelling and packing of textile products ("The Official Gazette of the RS ", No 56/09);
- 2) Order on textile products which must be supplied with quality certificate when traded ("The Official Gazette of the FRY ", No 14/92).

### Article 28

Provisions set out in Articles 21-24 of this rulebook shall cease to be valid starting from the day of the accession of the Republic of Serbia to the European Union.

### Article 29

This Rulebook shall enter into effect on the eight day following its publication in "The Official Gazette of the Republic of Serbia", and it shall apply from July 1 2015.

## Annex 1

### LIST OF TEXTILE FIBRE NAMES

Table 1

| No. | Name  | Fibre description   |
|-----|---|---|
| 1   | wool  | fibre from sheep's or lamb's fleeces ( <i>Ovis aries</i> ) or a mixture of fibres from sheep's or lamb's fleeces and the hairs of animals listed in number 2  |
| 2   | alpaca, llama, camel, cashmere, mohair, angora, vicuna, yak, guanaco, cashgora, beaver, otter, followed or not by the word 'wool' or 'hair' | Hair of the following animals: alpaca, llama, camel, cashmere goat, angora goat, angora rabbit, vicuna, yak, guanaco, cashgora goat, beaver, otter  |
| 3   | animal or horsehair, with or without an indication of the kind of animal (e.g. cattle hair, common goat hair, horsehair)                    | hair of various animals not mentioned under number 1 or 2   |
| 4   | silk  | fibre obtained exclusively from silk-secreting insects  |
| 5   | cotton  | fibre obtained from the bolls of the cotton plant ( <i>Gossypium</i> )  |
| 6   | kapok   | fibre obtained from the inside of the kapok fruit ( <i>Ceiba pentandra</i> )  |
| 7   | flax (or linen)   | fibre obtained from the bast of flax plant ( <i>Linum usitatissimum</i> )   |
| 8   | true hemp   | fibre obtained from the bast of hemp ( <i>Cannabis sativa</i> )   |
| 9   | Jute  | fibre obtained from the bast of <i>Corchorus olitorius</i> and <i>Corchorus capsularis</i> . For the purposes of this rulebook, bast fibres obtained from the following species shall be treated in the same way as jute: <i>Hibiscus cannabinus</i> , <i>Hibiscus sabdariffa</i> , <i>Abutilon avicennae</i> , <i>Urena lobata</i> , |
| 10  | abaca (Manila hemp)   | fibre obtained from the sheathing leaf of <i>Musa textilis</i>  |
| 11  | Alfa  | fibre obtained from the leaves of <i>Stipa tenacissima</i>  |
| 12  | coir (coconut)  | fibre obtained from the fruit of <i>Cocos nucifera</i>  |
| 13  | broom   | fibre obtained from the bast of <i>Cytisus scoparius</i> and/or <i>Spartium Junceum</i>   |
| 14  | ramie   | fibre obtained from the bast of <i>Boehmeria nivea</i> and <i>Boehmeria tenacissima</i>   |
| 15  | sisal   | fibre obtained from the leaves of plant <i>Agave sisalana</i>   |
| 16  | sunn  | fibre from the bast of <i>Crotalaria juncea</i>   |

|    |          |   |
|----|----------|---|
| 17 | henequen | fibre from the bast of <i>Agave fourcroydes</i> |
| 18 | maguery  | fibre from the bast of <i>Agave cantala</i>     |

Table 2

| No. | Name               | Fibre description  |
|-----|--------------------|--|
| 19  | acetate            | cellulose acetate fibre wherein less than 92 % but at least 74 % of the hydroxyl groups are acetylated   |
| 20  | alginate           | fibre obtained from metallic salts of alginic acid   |
| 21  | cupro              | regenerated cellulose fibre obtained by the cuprammonium process   |
| 22  | modal              | a regenerated cellulose fibre obtained by a modified viscose process having a high breaking force and high wet modulus. The breaking force ( $B_c$ ) in the conditioned state and the force ( $B_M$ ) required to produce an elongation of 5 % in the wet state are:<br>$B_c$ (cN) $\geq 1,3 \sqrt{T} + 2 T$ ;<br>$B_M$ (cN) $\geq 0,5 \sqrt{T}$<br>where T is the mean linear density in decitex (dtex) |
| 23  | protein            | fibre obtained from natural protein substances, regenerated and stabilised through the action of chemical agents   |
| 24  | triacetate         | cellulose acetate fibre wherein at least 92% of the hydroxyl groups are acetylated   |
| 25  | viscose            | regenerated cellulose fibre obtained by the viscose process for filament and discontinuous fibre   |
| 26  | acrylic            | fibre formed of linear macromolecules comprising at least 85% (by mass) in the chain of the acrylonitrilic pattern   |
| 27  | chlorofibre        | fibre formed of linear macromolecules having in their chain more than 50 % by mass of chlorinated vinyl or chlorinated vinylidene monomeric units  |
| 28  | fluorofibre        | fibre formed of linear macromolecules made from fluorocarbon aliphatic monomers  |
| 29  | modacrylic         | fibre formed of linear macromolecules having in the chain more than 50 % and less than 85 % (by mass) of the acrylonitrilic  |
| 30  | polyamide or nylon | fibre formed from linear macromolecules having in the chain recurring amide linkages of which at least 85 % are joined to aliphatic or cycloaliphatic units  |
| 31  | aramid             | fibre formed from synthetic linear macromolecules made up of aromatic groups joined by amide or imide linkages, of which at least 85 % are joined directly to two aromatic rings and with the number of imide linkages, if present, not exceeding the number of amide linkages   |
| 32  | polyimide          | fibre formed from synthetic linear macromolecules having in the chain recurring imide units  |
| 33  | lyocell            | a regenerated cellulose fibre obtained by dissolution, and an organic solvent (mixture of organic chemicals and water) spinning process, without formation of derivatives  |

|    |                  |   |
|----|------------------|---|
| 34 | polylactide      | fibre formed of linear macromolecules having in the chain at least 85 % (by mass) of lactic acid ester units derived from naturally occurring sugars, and which has a melting temperature of at least 135 °C  |
| 35 | polyester        | fibre formed of linear macromolecules comprising at least 85 % (by mass) in the chain of an ester of a diol and terephthalic acid   |
| 36 | polyethylene     | fibre formed of un-substituted aliphatic saturated hydrocarbon linear macromolecules  |
| 37 | polypropylene    | fibre formed of an aliphatic saturated hydrocarbon linear macromolecule where one carbon atom in two carries a methyl side chain in an isotactic disposition and without further substitution   |
| 38 | polycarbamide    | fibre formed of linear macromolecules having in the chain the recurring ureylene (NH-CO-NH) functional group  |
| 39 | polyurethane     | fibre formed of linear macromolecules composed of chains with the recurring urethane functional group   |
| 40 | vinylal          | fibre formed of linear macromolecules whose chain is constituted by poly(vinyl alcohol) with differing levels of acetalisation  |
| 41 | trivinyll        | fibre formed of acrylonitrile terpolymer, a chlorinated vinyl monomer and a third vinyl monomer, none of which represents as much as 50 % of the total mass   |
| 42 | elastodiene      | elastofibre composed of natural or synthetic polyisoprene, or composed of one or more dienes polymerised with or without one or more vinyl monomers, and which, when stretched to three times its original length and released, recovers rapidly and substantially to its initial length  |
| 43 | elastane         | elastofibre composed of at least 85 % (by mass) of a segmented polyurethane, and which, when stretched to three times its original length and released, recovers rapidly and substantially to its initial length  |
| 44 | glass fibre      | fibre made of glass   |
| 45 | elastomultiester | fibre formed by interaction of two or more chemically distinct linear macromolecules in two or more distinct phases (of which none exceeds 85 % by mass) which contains ester groups as the dominant functional unit (at least 85 %) and which, after suitable treatment when stretched to one and half times its original length and released, recovers rapidly and substantially to its initial length. |
| 46 | elastolefin      | fibre composed of at least 95 % (by mass) of macromolecules partially cross-linked, made up from ethylene and at least one other olefin and which, when stretched to one and a half times its original length and released, recovers rapidly and substantially to its initial length  |
| 47 | melamine         | fibre formed of at least 85 % by mass of cross-linked macromolecules made up of melamine derivatives  |



|    |  |   |
|----|--|---|
| 48 | name corresponding to the material of which the fibres are composed, e.g. metal (metallic, metallised), asbestos, paper, followed or not by the word 'yarn' or 'fibre' | fibres obtained from miscellaneous or new materials not listed above  |
| 49 | Binary polypropylene/polyamide fibre   | Binary fibre which contains between 10% and 25% weight of polyamide fibrils incorporated in polypropylene basis |

## **Annex 2**

### **MINIMUM REQUIREMENTS REGARDING A TECHNICAL FILE TO BE INCLUDED IN THE APPLICATION FOR A NEW TEXTILE FIBRE NAME**

A Technical file to be attached to an application for the inclusion of a new textile fibre name in the list set out in Annex 1 shall contain at least the following information:

1) Proposed name of a new textile fibre:

The name proposed shall be related to the chemical composition of a textile fibre and shall provide information about the characteristics of the fibre.

The name proposed shall be free of any intellectual property rights and shall not be linked to the manufacturer, its company name, a trademark or its other properties.

2) Proposed definition of the textile fibre.

The characteristics mentioned in the definition of the new textile fibre, such as elasticity, shall be verifiable via testing methods to be provided with the technical file along with the experimental results of analyses.

3) Identification of the textile fibre.

Chemical formula, differences from existing textile fibres, together with, where relevant, detailed data such as melting point, density, refractive index, burning behaviour and FTIR spectrum shall be submitted along with the proposed name.

4) Proposed agreed allowance to be used in the calculation of fibre composition the name of which is being proposed.

5) Testing methods for the purpose of identification and quantification, including experimental data.

The applicant shall evaluate the possibility to use the methods listed in Annex 8 of this rulebook or other test methods for which it suggests to be introduced in that annex and shall propose at least one of those testing methods.

For those test methods where the textile fibre can be considered as an insoluble component, the applicant shall evaluate the mass correction factors of the new textile fibre and all the data shall be submitted with the application.

If methods listed in this rulebook are not suitable, the applicant shall provide adequate reasoning and propose a new method.

The application shall contain all the experimental data for the methods proposed. Data on the accuracy, reliability and repeatability of the methods shall be provided with the file.

6) Available scientific information concerning possible allergic reactions or other adverse effects of the new textile fibre on human health, including results of tests conducted to that effect in compliance with special regulations.

7) Additional information to support the application, such as production process, consumer relevance regarding the new textile fibre name.

The manufacturer or any person acting on the manufacturer's behalf shall provide representative samples of the new pure textile fibre and the relevant textile fibre mixtures necessary to conduct the validation of the proposed identification and quantification methods. Additional samples of relevant fibre mixtures from the manufacturer or the person acting on the manufacturer's behalf may also be requested.

### **Annex 3**

## **NAMES FOR TEXTILE PRODUCTS MADE OF PURE FLEECE WOOL**

- in Serbian: "čista runska wool"
- in Bulgarian: "neobработена вълна"
- in Spanish: "lana virgen" or "lana de esquilado"
- in Czech: "střížní vlna"
- in Danish: "ren, ny uld"
- In German: "Schurwolle"
- in Estonian: "uus vill"
- in Greek: "παρθένο μαλλί"
- in English: "fleece wool" or "virgin wool"
- in French: "laine vierge" or "laine de tonte"
- in Irish: "olann lomra"
- in Italian: "lana vergine" or "lana di tosa"

- in Latvian: "pirmlietojuma vilna" or "cirptā vilna"
- in Lithuanian: "natūralioji vilna"
- in Hungarian: "élőgyapjú"
- in Maltese: "suf verġni"
- in Dutch: "scheerwol"
- in Polish: "żywa wełna"
- in Portuguese: "lã virgem"
- in Romanian: "lână virgină"
- in Slovak: "strižná vlna"
- in Slovene: "runska volna"
- in Finnish: "uusi villa"
- in Swedish: "ny ull"

## Annex 4

### SPECIAL PROVISIONS FOR THE MARKING AND LABELLING OF CERTAIN TEXTILE PRODUCTS

| Products   | Marking and labelling provisions  |
|--|---|
| <b>1. The following corsetry products:</b>         | The fibre composition shall be indicated on the label or marking by stating the composition of the whole product or, either inclusively or separately, that of the components listed respectively:  |
| <b>Brassières</b>                                  | - the outside and the inside fabric of the surface of the cups<br>- back  |
| <b>Corsets and girdles</b>                         | - the front, the rear<br>- side panels  |
| <b>Corselets</b>                                   | - the outside and inside fabric of the surface of cups,<br>- the front and rear stiffening panels, and<br>- the side panels   |
| <b>2. Other corsetry products not listed above</b> | The fibre composition shall be indicated by stating the composition of the whole product or, either inclusively or separately, the composition of the various components of the products. Such labelling shall not be compulsory for components representing less than 10 % of the total weight of the product. |

|   |  |
|---|--|
| <b>3. Request</b> for all types of corsetry products  | The separate labelling and marking of the various parts of corsetry products shall be carried out in such a way that the consumer can easily understand to which part of the product the information on the label or marking refers.   |
| <b>4. Etch-printed textiles</b>   | The fibre composition shall be given for the product as a whole and may be indicated by stating, separately, the composition of the base fabric and that of the etched parts. Those components shall be mentioned by name.   |
| <b>5. Embroidered textiles</b>  | The fibre composition shall be given for the product as a whole and may be indicated by stating, separately, the composition of the base fabric and that of the embroidery yarn. Those components shall be mentioned by name. Such labelling or marking is compulsory only for the embroidered parts which amount to at least 10 % of the surface area of the product. |
| <b>6. Yarns consisting of a core and a cover made up of different fibres and made available on the market as such to the consumer</b> | The fibre composition shall be given for the product as a whole and may be indicated by stating the composition of the core and the cover separately. Those components shall be mentioned by name.   |
| <b>7. Velvet and plush textiles, or textiles resembling velvet or plush</b>   | The fibre composition shall be given for the whole product and, where the product comprises a distinct backing and a use-surface composed of different fibres, may be stated separately for those components. Those components shall be mentioned by name  |
| <b>8. Floor coverings and carpets of which the backing and the use-surface are composed of different fibres</b>                       | The fibre composition may be stated for the use-surface alone. The use-surface must be mentioned by name.  |

## **Annex 5**

### **TEXTILE PRODUCTS FOR WHICH MARKING OR LABELLING IS NOT MANDATORY**

1. Sleeve-supporting armbands
2. Watch straps of textile materials
3. Labels and badges
4. Stuffed pan-holders of textile materials
5. Coffee cosy covers
6. Tea cosy covers
7. Sleeve protectors

8. Muffs other than in plush fabric
9. Artificial flowers
10. Pin cushions
11. Painted canvas
12. Textile products for base and underlying fabrics and stiffenings
13. Old made-up textile products, where explicitly stated to be such
14. Gaiters
15. Packaging, not new and sold as such
16. Fancy goods and saddlery, of textile materials
17. Travel goods of textile materials
18. Hand embroidered tapestries, finished or unfinished, and materials for their production, including embroidery yarns sold separately from the canvas and specially presented for use in such tapestries
19. Slide fasteners
20. Buttons and buckles covered with textile materials
21. Book covers of textile materials
22. Toys
23. Textile parts of footwear
24. Table mats having several components and a surface area of not more than 500 cm<sup>2</sup>
25. Oven gloves and cloths
26. Egg cosy covers
27. Make-up cases
28. Tobacco pouches of textile fabric
29. Spectacle, cigarette and cigar, lighter and comb cases of textile fabric
30. Covers for mobile telephones and portable media players with a surface of not more than 160 cm<sup>2</sup>

31. Protective requisites for sports with the exception of gloves
32. Toilet cases
33. Shoe-cleaning cases
34. Funeral products
35. Disposable products, with the exception of wadding
36. Textile products which are subject to the rules that refer to medical products and which are covered by a reference to those rules, non-disposable bandages for medical and orthopaedic use and orthopaedic textile products in general
37. Textile products including cordage, ropes and string, subject to Annex 6 item 12 of this rulebook, normally intended:
  - a) for use as equipment components in the manufacture and processing of goods, or
  - b) for incorporation in machines, installations (e.g. for heating, air conditioning or lighting), domestic and other appliances, vehicles and other means of transport, or for their operation, maintenance or equipment, other than tarpaulin covers and textile motor vehicle accessories sold separately from the vehicle.
38. Textile products for protection and safety purposes such as safety belts, parachutes, life-jackets, emergency chutes, fire-fighting devices, bulletproof waistcoats and special protective garments (e.g. protection against fire, chemical substances or other safety hazards)
39. Air-supported structures (e.g. sports halls, exhibition stands or storage facilities), provided that details of the performances and technical specifications of these products are supplied
40. Sails
41. Animal clothing
42. Flags and banners.

## **Annex 6**

### **TEXTILE PRODUCTS FOR WHICH INCLUSIVE LABELLING IS SUFFICIENT**

1. Floorcloths
2. Cleaning cloths

3. Edgings and trimmings
4. Passementerie
5. Belts
6. Braces
7. Suspenders and garters
8. Shoe and boot laces
9. Ribbons
10. Elastic
11. New packagings sold as such
12. Packing string and agricultural twine; string, cordage and ropes other than those falling within Annex 5, Item 37 of this rulebook. The products under this Item which are sold as cuts of certain length are marked with inclusive labelling on the spool. Strings and cordages under this Item comprise strings and cordages used in hiking and water sports
13. Table mats
14. Handkerchiefs
15. Bun nets and hair nets
16. Ties and bow ties for children
17. Bibs, washgloves and face flannels
18. Sewing, mending and embroidery yarns presented for retail sale in small quantities with a net weight of 1 gram or less.
19. Tape for curtains and blinds and shutters

**Annex 7**  
**PARTS OF TEXTILE PRODUCTS NOT TO BE TAKEN INTO**  
**ACCOUNT FOR DETERMINATION OF FIBRE**  
**COMPOSITION**

| Name of textile product | Parts not taken into account for determination of fibre composition |
|-------------------------|---|
|                         |   |

|  |  |
|--|--|
| a) All textile products                                      | (1) Non-textile parts, selvages, labels and badges, edgings and trimmings not forming an integral part of the product, buttons and buckles covered with textile materials, accessories, decorations, non-elastic ribbons, elastic threads and bands added at specific and limited points of the product and, subject to the conditions specified in Article 10 of this rulebook, visible, isolable fibres which are purely decorative and fibres with antistatic effect;<br>(2) Fatty substances, binders, weightings, sizings and dressings, impregnating products, additional dyeing and printing products and other textile processing products   |
| b) Floor coverings and carpets                               | All components other than the use-surface  |
| c) Upholstery fabrics  | Binding and filling warps and wefts which do not form part of the use-surface  |
| d) Hangings and curtains                                     | Binding and filling warps and wefts which do not form part of the right side of the fabric   |
| e) Socks   | Additional elastic yarns used in the cuff and the stiffening and reinforcement   |
| f) Tights  | Additional elastic yarns used in the cuff and the stiffening and reinforcement   |
| g) Textile products other than those under points (b) to (f) | Base or underlying fabrics, stiffenings and reinforcements, inter-linings and canvas backings, stitching and assembly threads unless they replace the warp and/or weft of the fabric, fillings not having an insulating function and linings, subject to Article 11 Paragraph 2<br>For the purposes of this provision:<br>(1) the base or underlying material of textile products which serve as a backing for the use-surface, in particular in blankets and double fabrics, and the backings of velvet or plush fabrics and kindred products shall not be regarded as backings to be removed;<br>(2) 'stiffenings and reinforcements' mean the yarns or materials added at specific and limited points of the textile products to strengthen them or to give them stiffness or thickness |

## **Annex 8**

### **METHOD FOR THE QUANTITATIVE ANALYSIS OF BINARY AND TERNARY TEXTILE FIBRE MIXTURES**

#### Chapter I

#### PREPARATION OF LABORATORY TEST SAMPLES

#### **1. Preparation of laboratory test samples and test specimens to determine the fibre composition of textile products**

##### 1.1. FIELD OF APPLICATION

This chapter 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. In some cases it is necessary to pre-treat individual



test samples.

## 1.2. DEFINITIONS

### 1.2.1. Bulk source

Bulk source is the quantity of material which is assessed 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.

### 1.2.2. Laboratory bulk sample

Laboratory bulk sample is the 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 shall be sufficient to overcome adequately the variability of the bulk source and to facilitate ease of handling in the laboratory. For made-up and finished products, the procedure of taking laboratory samples is described in Item 1.7 of this Chapter.

### 1.2.3. Laboratory test sample

Laboratory test sample is the 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 shall be sufficient to overcome adequately the variability of the laboratory bulk sample (Item 1.1 of this Chapter).

### 1.2.4. Test specimen

Test specimen is the portion of material required to give an individual test result, and selected from the laboratory test sample.

## 1.3. 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 way that each of them is representative of the laboratory test sample.

## 1.4. SAMPLING FROM LOOSE FIBRES

### 1.4.1. Unorientated fibres

Laboratory test sample is obtained by selecting at random tufts from the laboratory bulk sample. The whole of the laboratory test sample is mixed thoroughly by means of a laboratory carder. The laboratory carder may be replaced by a fibre blender, or the fibres may be mixed by the method of "tufts and rejects". It is necessary first to subject the web or mixture, including loose fibres and fibres adhering to the equipment used for mixing, to pre-treatment. Test specimens are then selected, in proportion to the respective masses, from the web or mixture, from the loose fibres and from fibres adhering to the equipment.

If the card web remains intact after pre-treatment, the test specimens are selected in the manner described in Item 1.4.2 of this Chapter. If the card web is disturbed by the pre-treatment, each test specimen is selected by removing at random at least 16 small tufts of

suitable and approximately equal size.

#### 1.4.2. Orientated fibres (cards, webs, slivers, rovings)

From randomly selected parts of the laboratory bulk sample cut not less than 10 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 10 lengths.

### 1.5. SAMPLING YARN

#### 1.5.1. Yarn in packages or in banks

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 (if the packages can be mounted in a convenient creel a number can be wound simultaneously) 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 text of the yarn is  $t$  (*tex*), and the number of packages selected from the laboratory bulk samples is  $n$ , then to obtain a test sample of 10 g, the length of yarn to be withdrawn from each package is  $10^6/nt$  cm.

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

#### 1.5.2. 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  $10^6/Nt$  cm.

### 1.6. SAMPLING FABRIC

#### 1.6.1. Sampling fabric 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  $\times 10^4/G \text{ cm}^2$ , where  $G$  is the mass of the cloth in  $\text{g/m}^2$ .

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.

#### 1.6.2. Sampling fabrics from a laboratory bulk sample consisting of several cuttings

Take each cutting as described in Item 1.6.1. of this Chapter and give each result separately.

### 1.7. SAMPLING MADE-UP AND FINISHED PRODUCTS

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

Where appropriate, determine the percentage of the various parts of the product not having the same fibre content, in order to check compliance with Article 11 of this rulebook.

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

If the product 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 product and to determine the relative proportions of the various parts in relation to the whole product 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.

## **2. Introduction to methods for the quantitative analysis of textile fibre mixtures**

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 shall 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. The first part of this annex gives the information common to the analyses by this method of all fibre mixtures dealt with in this annex, whatever their composition. It shall 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 fats, waxes and water-soluble matter is also given.

In addition, textiles may contain resins or other matter added to confer special properties. It may happen that, under influence of a certain reagent on the fibre component which is to be solved, such matters dissolve too, either partially or fully. In order to avoid such errors, resins or other matter is removed before chemical analysis of the sample. 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 shall 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 agreed allowances listed in Annex 9.

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(s).

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 shall 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 shall 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.

## Chapter II

# METHODS FOR QUANTITATIVE ANALYSIS OF CERTAIN BINARY TEXTILE FIBRE MIXTURES

### **1. General information common to the methods given for the quantitative chemical analysis of binary textile fibre mixtures**

#### 1.1. FIELD OF APPLICATION

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

#### 1.2. 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 is dissolved, usually by selective solution (Method 12 is an exception. It is based on a determination of the content of a constituent substance of one of the two components). 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.

#### 1.3. MATERIALS AND EQUIPMENT

##### 1.3.1. Apparatus

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

1.3.1.2. Vacuum flask

1.3.1.3. Desiccator containing self-indicating silica gel.

1.3.1.4. Ventilated oven for drying specimens at  $105^{\circ} \pm 3^{\circ}\text{C}$

1.3.1.5. Analytical balance, accurate to 0,0002 g.

1.3.1.6. Soxhlet extractor or other apparatus giving identical results.

##### 1.3.2. Reagents

1.3.2.1. Light petroleum, redistilled, boiling range  $40\text{-}60^{\circ}\text{C}$

1.3.2.2. Other reagents are specified in the appropriate sections of each method

1.3.2.3. Distilled or deionised water

1.3.2.4. Acetone

1.3.2.5. Orthophosphoric acid

1.3.2.6. Urea

1.3.2.7. Sodium-bicarbonate.

All reagents used shall be chemically pure.

#### 1.4. CONDITIONING AND TESTING ATMOSPHERE

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

#### 1.5. 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.

#### 1.6. PRE-TREATMENT OF LABORATORY TEST SAMPLE

Where a substance not to be taken into account in the percentage calculations is present (see Article 19 of this rulebook), it shall 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 test sample in a Soxhlet extractor with light petroleum for one hour at a minimum rate of six cycles per hour. Allow the light petroleum to evaporate from the sample, which is then extracted by direct treatment consisting in soaking the laboratory test sample in water at room temperature for one hour and then soaking it in water at  $65 \pm 5$  °C for a further hour, agitating the liquor from time to time. Use a liquor:laboratory test sample 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.

In the case of elastolefin or fibre mixtures containing elastolefin and other fibres (wool, animal hair, silk, cotton, flax (or linen), true hemp, jute, abaca, alfa, coir, broom, ramie, sisal, cupro, modal, protein, viscose, acrylic, polyamide or nylon, polyester, elastomultiester), the described procedure shall be slightly modified, in that light petroleum ether shall be replaced by acetone.

In the case of binary fibre mixtures containing elastolefin and acetate, the following procedure shall apply as pre-treatment. Extract the laboratory test sample for 10 minutes at 80°C with a solution containing 25 g/l of 50% orthophosphoric acid and 50 g/l of urea. Use a liquor:laboratory test sample ratio of 100:1. Wash laboratory test sample in water, then drain and wash it in a 0,1% - sodium-bicarbonate solution, and finally wash it carefully in water.

Where non-fibrous matter cannot be extracted with light petroleum and water, it shall 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 contains finishes insoluble in both light petroleum and water.

Analysis reports shall 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 four hours and not more than 16 hours at  $105 \pm 3$  °C in a ventilated oven with the oven door closed throughout. If the drying period is less than 14 hours, the specimen must be 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 shall 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 which is placed beside the balance, until complete cooling of the weighing bottles is attained, and in any case for not less than two hours.

#### 1.7.1.3. Weighing

After cooling, complete the weighing of the weighing bottle within two 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 specimen to the glass

vessel specified in the appropriate applied method from this Annex, 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.

## 1.8. CALCULATION AND EXPRESSION OF RESULTS

Express the mass of the insoluble component as a percentage of the total mass of fibre in the mixture. The percentage of soluble component is obtained by difference of the mass before the solution and the mass of insoluble residue. Calculate the results on the basis of clean, dry mass, adjusted by (a) the agreed allowances 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 1.8.2.

### 1.8.1. 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 in mass of the insoluble component in the reagent during the analysis. Suitable values of 'd' are given in the relevant section „Calculation and expression of results“ of each method.

Of course, such values of 'd' are the normal values applicable to chemically undegraded fibres.

1.8.2. Percentage of insoluble component on clean dry mass basis, with adjustment by conventional factors and, where appropriate, correction factors for loss of mass during pre-treatment is calculated by applying the following formula:

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

where:

$P_{1A} \%$  is the percentage of insoluble component, adjusted by agreed allowances 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 1.8.1.

$a_1$  is the agreed allowance for the insoluble component (see Annex 9 of this rulebook),

$a_2$  is the agreed allowance for the soluble component (see Annex 9 of this rulebook),

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

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

The percentage of the second component is calculated from the following formula:

$$P_{2A} \% = 100 - P_{1A} \%$$

Where a special pre-treatment has been used, the values of  $b_1$  and  $b_2$  shall 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, shall 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.

## **2. Method of quantitative analysis by manual separation**

### **2.1. 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.

### **2.2. PRINCIPLE**

After identification of the constituents of the mixture, 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.

### **2.3. APPARATUS**

2.3.1. Weighing bottle or any other apparatus giving identical results.

2.3.2. Desiccator containing self-indicating silica gel.

2.3.3. Ventilated oven for drying specimens at  $105^\circ \pm 3^\circ\text{C}$

2.3.4. Analytical balance, accurate to 0,0002 g.

2.3.5. Soxhlet extractor, or other apparatus giving an identical result.

2.3.6. Needle.

2.3.7. Twist tester or similar apparatus

### **2.4. REAGENTS**

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

2.4.2. Distilled or deionised water.

2.4.3. Acetone.

2.4.4. Orthophosphoric acid.

2.4.5. Urea.

2.4.6. Sodium-bicarbonate.

All reagents used should be chemically pure.

## 2.5. CONDITIONING AND TESTING ATMOSPHERE

from Item 1.4. of this Annex

## 2.6. LABORATORY TEST SAMPLE

from Item 1.5. of this Annex

## 2.7. PRE-TREATMENT OF LABORATORY TEST SAMPLE

from Item 1.6. of this Annex

## 2.8. PROCEDURE

### 2.8.1. 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  $105 \pm 3$  °C until a constant mass is obtained, as described in items 1.7.1 and 1.7.2.

### 2.8.2. 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 item 2.8.1.

## 2.9. 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 the agreed allowances and the correction factors necessary to take account of loss of matter during pre-treatment.

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

|                   |                             |                  |  |                   |
|-------------------|-----------------------------|------------------|--|-------------------|
| $P_1\% =$         | $\frac{100 m_1}{m_1 + m_2}$ | $\frac{100}{1 +$ | <table border="1" style="margin-left: auto; margin-right: auto; border-collapse: collapse;"> <tr> <td style="width: 50%; text-align: center; vertical-align: middle;"><math>\frac{m_2}{m_1}</math></td> </tr> </table> | $\frac{m_2}{m_1}$ |
| $\frac{m_2}{m_1}$ |                             |                  |  |                   |

$P_1\%$  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.

2.9.2. For calculation of the percentage of each component with adjustment by agreed allowances and, where appropriate, by correction factors for loss of matter during pre-treatment, see Item 1.8.2 of Section 1 of this Chapter.

### 3. Precision of the methods and test reports

#### 3.1. PRECISION OF THE METHOD

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 repeatability is expressed by confidence limits of the results for a confidence level of 95 %. By this is meant that the difference between two results in a series of analyses made in different laboratories would, given a normal and correct application of the method to an identical and consistent mixture, exceed the confidence limit only in five cases out of a 100.

#### 3.2. TEST REPORT

3.2.1. State that the analysis was conducted in accordance with this method,

3.2.2. Give details of any special pre-treatment (from Item 1.6. of this Annex),

3.2.3. Give the individual results and the arithmetic mean, each to an accuracy of 0,1.

### 4. Special methods – Summary table

| Method | Field of application (*)  |                       | Reagent/Description                      |
|--------|---|-----------------------|--|
|        | Soluble component   | Insoluble component   |  |
| 1      | Acetate   | certain other fibres  | Acetone                                  |
| 2.     | certain protein fibres  | certain other fibres  | hypochlorite <sub>1</sub>                |
| 3.     | viscose, cupro or certain types of modal                                    | certain other fibres  | formic acid and zinc chloride            |
| 4.     | polyamide or nylon  | certain other fibres  | formic acid, 80% m/m                     |
| 5.     | Acetate   | certain other fibres  | benzyl alcohol                           |
| 6.     | triacetate or polylactide   | certain other fibres  | dichloromethane                          |
| 7.     | certain cellulose fibres  | certain other fibres  | sulphuric acid, 75% m/m                  |
| 8.     | acrylics, certain modacrylics or certain chlorofibres                       | certain other fibres  | dimethylformamide                        |
| 9.     | certain chlorofibres  | certain other fibres  | carbon disulphide/acetone 55,5/44,5% v/v |
| 10.    | Acetate   | certain other fibres  | glacial acetic acid                      |
| 11.    | silk, polyamide or nylon  | certain other fibres  | sulphuric acid, 75% m/m                  |
| 12.    | Jute  | certain animal fibres | nitrogen content method                  |
| 13.    | Polypropylene   | certain other fibres  | Xylene                                   |
| 14.    | certain other fibres  | certain other fibres  | concentrated sulphuric acid              |
| 15.    | chlorofibres, certain modacrylics, certain elastanes, acetates, triacetates | certain other fibres  | Cyclohexanone                            |
| 16.    | Melamine  | cotton or aramid      | hot formic acid 90% m/m                  |

(\*) Detailed list of fibres per each of methods

## METHOD No. 1

### ACETATE AND OTHER CERTAIN FIBRES (Acetone method)

#### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

## 1.1. acetate (19)

with

2.1. 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 fibres (23), viscose (25), acrylic (26), polyamide or nylon (30) polyester (35), polypropylene (37), elastomultiester (45), elastolefin (46) and melamine (47) fibres and binary polypropylene/polyamide fibre (49), after removal of non-fibrous matter.

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 gram of test 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. 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; except for melamine, 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  $\pm 1$  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 applies to binary fibre mixtures of:

1.1. certain protein fibres, namely: wool (1), animal hair (2 and 3), silk (4), protein (23)

with

2.1. cotton (5), cupro (21), viscose (25), acrylic (26), chlorofibres (27), polyamide or nylon (30), polyester (35), polypropylene (37), elastane (43), glass fibre (44), elastomultiester (45), elastolefin (46), melamine (47) and binary polypropylene/polyamide fibre (49), after removal of non-fibrous matter.

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 known 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

- a) Erlenmeyer flask with ground-glass stopper, 250 ml
- b) Thermostat, adjustable to  $20 \pm 2^\circ\text{C}$ .

##### 3.2. Reagents

- a) Hypochlorite reagent

#### (1) Lithium hypochlorite solution

This consists of a freshly prepared solution containing 35 ( $\pm 2$ ) g/l of active chlorine (approximately 1 M), to which 5 ( $\pm 0,5$ ) 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 need not be checked iodometrically.

#### (2) Sodium hypochlorite solution

This consists of a freshly prepared solution containing 35 ( $\pm 2$ ) g/l of active chlorine (approximately 1 M) to which  $5 \pm 0,5$  g/l of previously dissolved sodium hydroxide is added. Check the active chlorine content of the solution iodometrically before each analysis.

#### b) 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 test specimen 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 test specimen.

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, cooling is required. 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, modal, and melamine for which 'd' = 1,01, and unbleached cotton, for which 'd' = 1,03

### 6. PRECISION

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



## MEHOD No. 3

### VISCOSE, CUPRO OR CERTAIN TYPES OF MODAL AND CERTAIN OTHER FIBRES (Method using formic acid and zinc chloride)

#### 1. FIELD OF APPLICATION

This method is applicable to binary fibre mixtures of:

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

with

2.1 cotton (5), polypropylene (37), elastolefin (46) and melamine (47), after removal of non-fibrous matter.

If a modal fibre is found to be present, a preliminary test shall 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 mass, corrected if necessary, is expressed as a percentage of the insoluble component related to 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

a) glass-stoppered conical flasks of at least 200 ml capacity.

b) apparatus for maintaining flasks at  $40 \pm 2^\circ\text{C}$  (water bath).

##### 3.2. Reagents

a) 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).

Note:

Attention is drawn, in this respect, which lays down that all reagents used shall be chemically pure; in addition, it is essential to use only fused anhydrous zinc chloride.

b) Ammonium hydroxide solution: dilute 20 ml of a concentrated ammonia solution (relative density at 20 °C: 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 gram 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 pre-heated to 40 °C.

Wash crucible and residue thoroughly with water at 40 °C. Rinse the fibrous residue in approximately 100 ml of cold ammonia solution (3.2. item b of this method) ensuring that this residue remains wholly immersed in the solution for 10 minutes. To ensure that the fibrous residue is immersed in 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. Rinse residue thoroughly with cold water.

Do not apply suction until all 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' is 1,00, except for cotton for which the value of 'd' =1,02 and for melamine 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  $\pm 2$  for a confidence level of 95 %

#### METHOD No. 4

#### POLYAMIDE OR NYLON, AND CERTAIN OTHER FIBRES (Method using 80 % m/m formic acid)

#### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

1.1 polyamide or nylon (30)

with

2.1 wool (1), animal hair (2 and 3), cotton (5), cupro (21), modal (22), viscose (25), acrylic (26), chlorofibre (27), polyester (35), polypropylene (37), glass fibre (44), elastomultiester (45), elastolefin (46) and melamine (47), after removal of non-fibrous matter.

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

## 2. PRINCIPLE

The polyamide or nylon 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 insoluble component related to the dry mass of the mixture. The percentage of dry polyamide or nylon fibre 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

a) Formic acid (80% m/m, relative density at 20°C : 1,186). Dilute 880 ml formic acid of 90% m/m (relative density at 20°C : 1,204) to 1 litre with water. Alternatively, dilute 780 ml of 98 to 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.

b) 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 gram 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. Drain the crucible with suction and wash out the residue with formic acid reagent first, and then with hot water, ammonia dilute solution and finally 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 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 melamine, for which '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  $\pm 1$  for a confidence level of 95 %.

### METHOD No. 5

#### ACETATES AND CERTAIN OTHER FIBRES (Method using benzyl alcohol)

##### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

1.1. acetate (19)

with

2.2. triacetate (24), polypropylene (37), elastolefin (46), melamine (47) and binary polypropylene/polyamide fibre (49), after removal of non-fibrous matter.

##### 2. PRINCIPLE

The acetate fibre is dissolved out from a known dry mass of the mixture, with benzyl alcohol at  $52 \pm 2^\circ\text{C}$ .

The residue is collected, washed, dried and weighed; its mass is expressed, if necessary, as a percentage of the insoluble component related to the total mass of fibres in 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

- a) Glass-stoppered conical flask of at least 200 ml capacity
- b) Mechanical shaker.
- c) Water bath with thermostat or some other apparatus for keeping the flask at a temperature of  $52 \pm 2^\circ\text{C}$ .

###### 3.2. Reagents

- a) Benzyl alcohol.

b) 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 gram of specimen. Insert the stopper, secure the flask to the shaker so that it is immersed in the water-bath, kept at  $52 \pm 2$  °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  $52 \pm 2$  °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  $52 \pm 2$  °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; except for melamine for which '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  $\pm 1$  for a confidence level of 95 %.

#### METHOD No. 6

### TRIACETATES OR POLYLACTIDES AND CERTAIN OTHER FIBRES (Method using dichloromethane)

#### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

1.1. triacetate (24) or polylactide (34)

with

2.1. wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (21), modal (22), viscose (25), acrylic (26), polyamide or nylon (30), polyester (35), polypropylene (37), glass fibre (44), elastomultiester (45), elastolefin (46), melamine (47) and binary polypropylene/polyamide fibres (49), after removal of non-fibrous matter.

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 or polylactide fibres are 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 gram of the test specimen, insert the stopper, shake the flask every 10 minutes to wet out the specimen and stand for 30 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.

## 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, elastomultiester, elastolefin and melamine 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  $\pm 1$  for a confidence level of 95 %.

### METHOD No. 7

#### CERTAIN CELLULOSE FIBRES AND CERTAIN OTHER FIBRES (Method using 75 % m/m sulphuric acid)

##### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

1. cotton (5), flax (7), true hemp (8), ramie (14), cupro (21), modal (22), viscose (25)

with

2. polyester (35), polypropylene (37), elastomultiester (45) and elastolefin (46) and binary polypropylene/polyamide fibre (49), after removal of non-fibrous matter.

##### 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 insoluble component related to the total mass of fibres in 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

- a) Glass-stoppered conical flask of at least 500 ml capacity.
- b) Water bath with thermostat or other apparatus for maintaining the flask at  $50 \pm 5^\circ\text{C}$ .

###### 3.2. Reagents

a) Sulphuric acid  $75 \pm 2\%$  m/m

Prepare by adding carefully, while cooling, 700 ml of sulphuric acid (relative density at  $20^\circ\text{C}$ : 1,84) to 350 ml of distilled water.

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

. b) Ammonia, dilute solution

Dilute 80 ml of ammonia solution (relative density at  $20^\circ\text{C}$ : 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 glass-stoppered conical flask of at least 500 ml capacity, add 200 ml of 75 % sulphuric acid per gram of specimen, insert the stopper and carefully shake the flask to wet out the specimen. Maintain the flask at  $50 \pm 5$  °C for one hour, shaking it at regular intervals of approximately 10 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 except for binary polypropylene/polyamide fibre for which '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  $\pm 1$  for a confidence level of 95 %.

#### METHOD No. 8

#### ACRYLIC, CERTAIN MODACRYLIC OR CERTAIN CHLOROFIBRES AND CERTAIN OTHER FIBRES (Method using dimethylformamide)

#### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

1.1. acrylics (26), certain modacrylics (29), or certain chlorofibres (27),  
Solubility of these modacrylics and chlorofibres in reagent is checked before analyzing

with

2.2. wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (21), modal (22), viscose (25), polyamide or nylon (30), polyester (35), polypropylene (37), elastomultiester (45), elastolefin (46), melamine (47) and binary polypropylene/polyamide fibre (49), after removal of non-fibrous matter.



This method is equally applicable to acrylics, and certain modacrylics, treated with pre-metallised 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

- a) Glass-stoppered conical flask of at least 200 ml capacity.
- b) Water bath with thermostat for maintaining temperature at 90-95°C

### 3.2. Reagent

Dimethylformamide (boiling point  $153 \pm 1$  °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 gram 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 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.

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 except in the following cases: wool, cotton, copper fibre, modal, polyester, elastomultiester and melamine, 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  $\pm 1$  for a confidence level of 95 %.

### METHOD No. 9

#### CERTAIN CHLOROFIBRES AND CERTAIN OTHER FIBRES (Method using mixture of carbon disulphide and acetone in the proportion 55,5/44,5 v/v)

##### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

1. certain chlorofibres (27), namely certain polyvinyl chloride fibres, whether after-chlorinated or not (solubility of polyvinyl chloride fibres in reagent are checked before analysis).

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 (35), polypropylene (37), glass fibres (44), elastomultiester (45), melamine (47), binary polypropylene/polyamide fibre (49), after removal of non-fibrous matter.

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

When the polyamide or nylon content of the mixture exceeds 25 %, method No 4 shall 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.

##### 3. APPARATUS AND REAGENTS

(other than those specified in the general instructions)

###### 3.1. Apparatus

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

b) Mechanical shaker.

### 3.2. Reagents

a) 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

b) 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 gram of specimen. Seal the flask securely, and shake the flask on a mechanical shaker, or vigorously by hand, for 20 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; except for melamine, for which 'd' is 1,01.

### 6. PRECISION

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

#### METHOD No. 10

#### ACETATE AND CERTAIN OTHER FIBRES (Method using glacial acetic acid)

##### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

### 1.1. acetate (19)

with

2.1. certain chlorofibres (27), namely polyvinyl chloride fibres, whether afterchlorinated or not, polypropylene (37), elastolefin (46), melamine (47) and binary polypropylene/polyamide fibre (49), after removal of non-fibrous matter.

## 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

- a) Glass-stoppered conical flask of at least 200 ml capacity
- b) Mechanical shaker.

### 3.2. Reagent

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

## 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 gram of specimen. Seal the flask securely and shake on the mechanical shaker, or vigorously by hand, for 20 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 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  $\pm 1$  for a confidence level of 95 %.

## METHOD No. 11

### SILK OR POLYAMIDE AND CERTAIN OTHER FIBRES (Method using 75 % m/m sulphuric acid)

#### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

1.1. silk (4), polyamide or nylon (30)

with

2.1. wool (1), animal hair (2 and 3), polypropylene (37), elastolefin (46), melamine (47) and binary polypropylene/polyamide fibre (49), after removal of non-fibrous matter.

#### 2. PRINCIPLE

The silk fibre, or polyamide or nylon fibre is dissolved out from a known dry mass of the mixture, with 75 % m/m sulphuric acid. Wild silks such as "tussah" silk, are not completely soluble in 75 % m/m sulphuric 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 silk, polyamide or nylon 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

a) Sulphuric acid (75  $\pm$  2% m/m)

Prepare by adding carefully, while cooling, 700 ml sulphuric acid (relative density at 20 °C: 1,84) to 350 ml distilled water.

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

b) Sulphuric acid, dilute solution:

add 100 ml sulphuric acid (relative density at 20 °C: 1,84) slowly to 1 900 ml distilled water.

c) Ammonia, dilute solution:

dilute 200 ml concentrated ammonia (relative density at 20 °C: 0,880) to 1 000 ml with water.

#### 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 75 % m/m sulphuric acid per gram of specimen and insert the stopper. Shake vigorously and stand for 30 minutes at room temperature.

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.

In case of binary mixtures of polyamides with binary polypropylene/polyamide fibre, after filtering fibres through measured filter crucibles and before applying the described washing procedure, rinse the residue in filter crucible twice, using each time 50ml of 75 % sulphuric acid.

#### 5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of 'd' is 1,00, except for wool for which the value of 'd' is 0,985; for binary polypropylene/polyamide fibre 'd' is 1,005 and for melamine '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  $\pm 1$  for a confidence level of 95 %, except for binary mixtures of polyamides with binary polypropylene/polyamide fibre for which the confidence level is not higher than  $\pm 2$ .

#### METHOD No. 12

#### JUTE AND CERTAIN ANIMAL FIBRES (Method by determining nitrogen content)

#### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

1.1. jute (9)

with

2.1. certain animal fibres, after removal of non-fibrous matter.

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

- a) Kjeldahl digestion flask, 200 - 300 ml capacity.
- b) Kjeldahl distillation apparatus with steam injection.
- c) Titration apparatus, allowing precision of 0,05 ml.

### 3.2. Reagents

- a) Toluene.
- b) Methanol.
- c) Sulphuric acid, relative density at 20 °C: 1,84. This reagent and reagents that follow should be nitrogen-free
- d) Potassium sulphate
- e) Selenium dioxide
- f) 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.

- g) 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.

- h) Boric acid solution.

Dissolve 20 g of boric acid in 1 litre of water.

i) 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 laboratory test sample in a Soxhlet apparatus with a mixture of 1 volume of toluene and 3 volumes of methanol for four 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  $105 \pm 3$  °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.

Note:

The toxic effects of toluene and methanol shall 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 at 20°C). 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 15 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, use 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% | = | $\frac{28 (V - b) n n}{W}$ |
|----|---|----------------------------|

with

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.

|     |   |   |
|-----|---|---|
| PA% | = | $\frac{A - 0,22}{16,2 - 0,22} \times 100$ |
|-----|---|---|

where

PA% = 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  $\pm 1$  for a confidence level of 95 %.

## METHOD No. 13

### POLYPROPYLENE FIBRES AND CERTAIN OTHER FIBRES (Xylene method)

#### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

##### 1.1. polypropylene fibres (37)

with

2.1. 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 (35), glass fibre (44), elastomultiester (45) and melamine (47), after removal of non-fibrous matter.

#### 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

- a) Glass-stoppered conical flask of at least 200 ml capacity
- b) Reflux condenser (suitable for liquids of high boiling point), fitting the conical flask given in subitem a) of this method.
- c) Water bath at boiling point of xylene

##### 3.2. Reagent

Xylene distilling between 137 and 142 °C.

Note:

Xylene 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 (Item 3.1, subitem a. of this method), add 100 ml of xylene (Item 3.2. of this method) per gram of specimen. Attach the condenser (Item 3.1, subitem b.) bring the contents to the boil and maintain at boiling point for three minutes. Immediately decant the hot liquid through the weighed filter crucible. 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 (1.3.2.1 of general instructions from Chapter 2 of this Annex). 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.

Note:

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. (e.g. apparatus described in *Melliand Textilberichte* 56 (1975) pages 643-645).

#### 5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of 'd' is 1,00, except for melamine, for which '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  $\pm 1$  for a confidence level of 95 %.

#### METHOD No. 14

#### CERTAIN FIBRES AND CERTAIN OTHER FIBRES (Method with concentrated sulphuric acid method)

##### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

1.1. cotton (5), acetate (19), cupro (21), modal (22), triacetate (24), viscose (25), certain acrylics (26), certain modacrylics (29), polyamide or nylon (30), polyester (35) and elastomultiester (45).

with

2.2. chlorofibres (27), based on vinyl chloride homopolymers, whether after-chlorinated or not, polypropylene (37), elastolefin (46), melamine (47) and binary polypropylene/polyamide fibre (49), after removal of non-fibrous matter.

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

This method can be used in place of methods No 8 and No 9.

## 2. PRINCIPLE

The constituent other than the chlorofibre, polypropylene, elastolefin, melamine or binary polypropylene/polyamide fibre ( i.e. the fibres mentioned under point 1 of paragraph 2) is dissolved out from a known dry mass of the mixture with concentrated sulphuric acid (relative density 1,84 at 20 °C).

The residue, consisting of the chlorofibre, polypropylene, elastolefin, melamine or binary polypropylene/polyamide fibre 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

- a) Glass-stoppered conical flask of at least 200 ml capacity.
- b) Glass rod with flattened end.

### 3.2. Reagents

- a) Sulphuric acid, concentrated (relative density 1,84 at 20 °C).
- b) 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 deionised water. After cooling to room temperature, dilute the solution to one litre with water.

- c) 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 test specimen contained in the flask (3.1.a of this method), add 100 ml of sulphuric acid (3.2. a of this method) 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.a of this method) 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.a of this method) 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.b of this method), distilled or deionised water (1.3.2.3 of the general instructions from Chapter II of this Annex), ammonia solution (3.2.c of this method), and finally wash thoroughly with distilled or deionised 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.

#### 5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of 'd' is 1,00, except for melamine and binary polypropylene/polyamide fibre for which '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  $\pm 1$  for a confidence level of 95 %.

#### METHOD No. 15

#### CHLOROFIBRES, CERTAIN MODACRYLICS, CERTAIN ELASTANES, ACETATES, TRIACETATES AND CERTAIN OTHER FIBRES (Method using cyclohexanone)

#### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

1.1. acetate (19), triacetate (24), chlorofibre (27), certain modacrylics (29), certain elastanes (43)

with

2.2. wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (21), modal (22), viscose (25),

polyamide or nylon (30), acrylic (26), glass fibre (44) and melamine (47), after removal of non-fibrous matter.

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.

Methods Nos 9 or 14 can be used for mixtures containing chlorofibres.

## 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, dried 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 specified in the general instructions)

### 3.1. Apparatus

- a) Hot extraction apparatus suitable for use in the test procedure in section 4 of this method (see figure in the continuation of this method), this is a variant of the apparatus described in "Melliand Textilberichte" 56 (1975) p. 643 - 645).
- b) Filter crucible to contain the specimen.
- c) Porous baffle (porosity grade 1).
- d) Reflux condenser that can be adapted to the distillation flask.
- e) Heating device.

### 3.2. Reagents

- a) Cyclohexanone, boiling point 156°C. b)

Ethyl alcohol, 50 % by volume.

Note:

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

## 4. TEST PROCEDURE

Follow the procedure described in the general instructions of Chapter II of this Annex 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.

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 exceptions of silk and melamine, for which 'd' is 1,01, and acryl, for which 'd' is 0.,98.

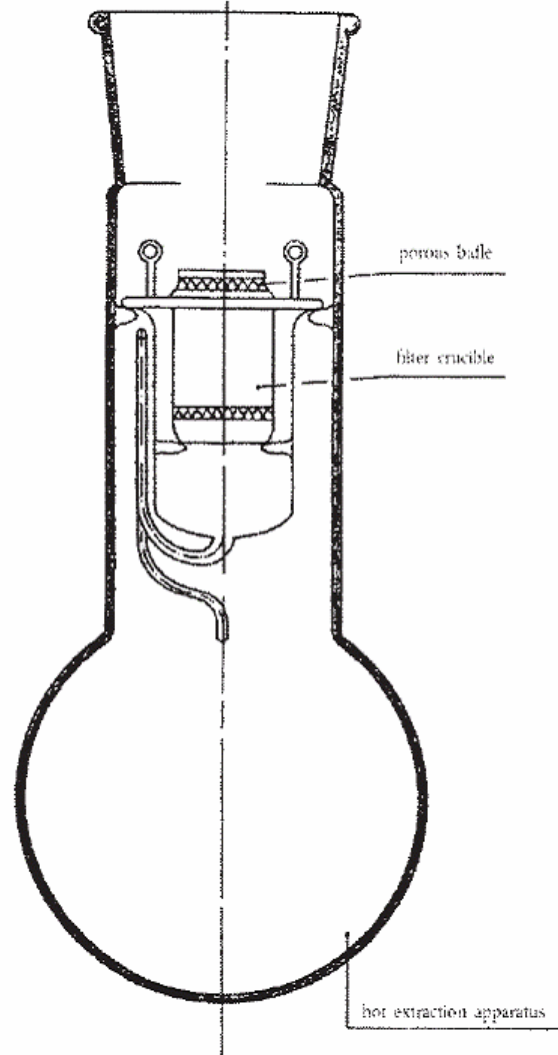
## 6. PRECISION

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

Figure referred to in point 3.1 (a) of method No 15

(Figure refers to: - porous baffle, - filter crucible, - hot extraction apparatus)

Figure referred to in point 3.1(a) of method No 15



## METHOD No. 16

### MELAMINE AND OTHER CERTAIN FIBRES (Method with hot formic acid)

#### 1. FIELD OF APPLICATION

This method is applicable to binary mixtures of:

1.1. melamine (47)

with

2.1. cotton (5), aramid (31) and polypropylene (37), after removal of non-fibrous matter.

#### 2. PRINCIPLE

Melamine, starting from a known dry mass of the mixture, is dissolved out from a



known dry mass of the mixture with hot formic acid (concentration 90% m/m).

The residue is collected, washed, dried and weighed, and its mass, corrected if necessary, is expressed as a percentage of the residue component from the total fibre mass in the mixture. The percentage of other components is found by difference.

Note:

The solubility of melamine depends to great extent on the temperature and the recommended temperature is strictly advised.

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

#### 3.1. Apparatus

- a) Glass-stoppered conical flask of at least 200 ml capacity.
- b) Shaking water bath or other apparatus to shake and maintain the flask at  $90 \pm 2^\circ\text{C}$

#### 3.2. Reagents

- a) Formic acid (90% m/m, relative density 1,204 at  $20^\circ\text{C}$ ).

Dilute 890 ml of 98 to 100% concentrated formic acid (relative density 1,220 at  $20^\circ\text{C}$ ) to 1 litre with water.

Hot formic acid is very corrosive and must be handled with care.

- b) Ammonia, dilute solution.

Dilute 80 ml of concentrated ammonia solution (relative density 0,880 at  $20^\circ\text{C}$ ) to 1 litre with water.

### 4. TEST PROCEDURE

Follow the procedure described in the general instructions, Chapter II of this Annex, and proceed as follows:

To the test specimen contained in the glass-stoppered conical flask of at least 200 ml capacity, add 100 ml of formic acid per gram of specimen. Insert the stopper, shake the flask to wet out the specimen. Maintain the flask in a shaking water bath at  $90 \pm 2^\circ\text{C}$  for one hour and shake it vigorously. Cool the flask to room temperature. Decant the liquid through weighed filter crucible. Add 50 ml of formic acid to the flask containing the residue, shake manually and filter the contents of the flask through the filter crucible. Transfer any residual fibres to the crucible by washing out the flask with a little more formic acid used as reagent. Drain the crucible with suction and wash the residue with reagent consisting of formic acid, hot water, dilute ammonia solution and finally with cold water, draining the crucible with suction after each addition. Do not apply suction until all 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 1,02.

## 6. PRECISION

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

# Chapter III QUANTITATIVE ANALYSIS OF TERNARY TEXTILE FIBRE MIXTURES

## INTRODUCTION

Methods for the quantitative chemical analyses are based on selective dissolving of certain components. There are four variations of this method:

1. Using two different test specimens, dissolve the component a) from the first test specimen, and dissolve the component b) from the second test specimen. Weigh the residues from each specimen and the percentage of each of the two soluble components is calculated from the respective losses in mass. The percentage of the third component c) is calculated by difference.
2. Using two different test specimens, dissolve the component a) from the first test specimen, and two components (a and b) from the second test specimen. Weigh the residue from the first test specimen and the percentage of the first component a) is calculated from the loss in mass. Weigh the residue of the second test specimen; it corresponds to the component c). The percentage of the third component b) is calculated by difference.
3. Using two different test specimens, dissolve two components (a and b) from the first test specimen, and two components (b and c) from the second test specimen. The residue corresponds to two components c) and a). The percentage of the third component b) is calculated by difference.
4. Using only one test specimen, after removing one of the components, weigh the residue formed by other two fibres and the percentage of the soluble component is measured from the loss in mass. Dissolve one of the two fibres of the residue, weigh the insoluble component and the percentage of the second soluble component is calculated from the loss in mass.

If there are any other options, it is advisable to use one of first three variants. Where chemical analysis is used, the expert responsible for the analysis must take care to select methods employing solvents which dissolve only the correct fibres a), leaving the other fibre(s) intact.

By way of example in Chapter 5 of this Annex, a table is given and it contains a certain number of ternary fibre mixtures, together with methods for analysing binary fibre mixtures which can, in principle, be used for analyzing these ternary fibre mixtures.

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

Before proceeding with any analysis, all the fibres present in the mixture must be identified. In some methods the insoluble component of the mixture may be partially dissolved in the reagent used to dissolve the soluble component(s). Wherever possible, reagents have been chosen that have little or no effect on the insoluble fibres. If a loss in mass is known to occur during the analysis, the result shall be corrected; correction factors are given for this purpose. These factors have been determined in several laboratories by treating, with the appropriate reagent as specified in the method of analysis, fibres cleaned by the pre-treatment. These correction factors apply only to undegraded fibres and different correction factors may be necessary if the fibres have been degraded before or during processing. If the fourth variant, in which a textile fibre is subjected to the successive action of two different solvents, must be used, correction factors must be applied for possible losses in mass undergone by the fibre in the two treatments. At least two determinations shall be made, both in the case of manual separation and in the case of chemical separation.

## **1. General information on methods for the quantitative chemical analysis of ternary fibre mixtures**

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

### 1.1. FIELD OF APPLICATION

The field of application of each method for analyzing binary fibre mixture specifies to which fibres the method is applicable (see Chapter II referring to methods for quantitative analysis of certain binary textile fibre mixtures).

### 1.2. PRINCIPLE

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

### 1.3. MATERIALS AND EQUIPMENT

#### 1.3.1. Apparatus

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

1.3.1.2. Vacuum flask

1.3.1.3. Desiccator containing self-indicating silica gel

1.3.1.4. Ventilated oven for drying specimens at  $150 \text{ }^\circ \pm 3 \text{ }^\circ\text{C}$

1.3.1.5. Analytical balance, accurate to 0,0002 g

1.3.1.6. Soxhlet extractor or other apparatus giving identical results.

#### 1.3.2. Reagents

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

1.3.2.2. Other reagents are specified in the appropriate sections of each method

1.3.2.3. Distilled or deionised water

1.3.2.4. Acetone

1.3.2.5. Orthophosphoric acid

1.3.2.6. Urea

1.3.2.7. Sodium-bicarbonate

All reagents used shall be chemically pure.

### 1.3. CONDITIONING AND TESTING ATMOSPHERE

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

### 1.5. 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.

### 1.6. PRE-TREATMENT OF LABORATORY TEST SAMPLE

Where a substance not to be taken into account in the percentage calculations (see Article 19 of the Rulebook) is present, it shall 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 test sample in a Soxhlet extractor with light petroleum for one hour at a minimum rate of six cycles per hour. Allow the light petroleum to evaporate from the sample, which is then extracted by direct treatment consisting in soaking the laboratory test sample in water at room temperature for one hour and then soaking it in water at  $65 \pm 5$  °C for a further hour, agitating the liquor from time to time. Use a liquor: laboratory test sample 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. In case of elastolefin or fibre mixtures containing elastolefin and other fibres (wool, animal hair, silk, cotton, flax, true hemp, jute, abaca, alfa, coir, broom, ramie, sisal, cupro, modal, protein, viscose, acrylic, polyamide or nylon, polyester, elastomultiester), the described procedure shall be slightly modified, in fact light petroleum ether shall be replaced by acetone.

Where non-fibrous matter cannot be extracted with light petroleum and water, it shall 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 contains finishes insoluble in both light petroleum and water. Analysis reports shall 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 four hours and not more than 16 hours at  $105 \pm 3$  °C in a ventilated oven with the oven door closed throughout. If the drying period is less than 14 hours, the specimen must be checkweighed to determine whether its mass is constant. The mass may be considered as constant if, after a further drying period of 60 minutes, its variation is less than 0,05 %.

Avoid handling crucibles and weighing bottles, specimens or residues with bare hands during the drying, cooling and weighing operations. Dry specimens in a weighing bottle with its cover beside it. After drying, stopper the weighing bottle before removing it from the oven, and transfer it quickly to the desiccator.

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

#### 1.7.1.2. Cooling

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

#### 1.7.1.3. Weighing

After cooling, complete the weighing of the weighing bottle within two 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 specimen to the glass vessel specified in the appropriate method of this annex which is applied, 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(s).

## 1.8. CALCULATION AND EXPRESSION OF RESULTS

Express the mass of the insoluble component as a percentage of the total mass of fibre in the

mixture. Calculate the results on the basis of clean, dry mass, adjusted by a) the agreed allowances and b) the correction factors necessary to take account of loss of matter during pre-treatment and analysis.

1.8.1. Calculation of percentage of mass of clean dry fibres, disregarding loss of fibre mass during pre-treatment.

#### 1.8.1.1. VARIANT 1

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

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

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

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

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

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

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

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

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

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

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

$d_1$  is the correction factor for loss in mass in the first reagent, of the second component undissolved in the first specimen, 'd' values being stated in Chapter II with this Annex, referring to various methods for the analysis of binary fibre mixtures;

$d_2$  is the correction factor for loss in mass in the first reagent, of the third component undissolved in the first specimen,

$d_3$  is the correction factor for loss in mass in the second reagent, of the first component undissolved in the second specimen,

$d_4$  is the correction factor for loss in mass in the second reagent of the third component undissolved in the second specimen.

### 1.8.1.2. VARIANT 2

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

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

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

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

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

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

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

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

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

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

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

$d_1$  is the correction factor for loss in mass in the first reagent, of the second component undissolved in the first specimen,

$d_2$  is the correction factor for loss in mass in the first reagent, of the third component undissolved in the first specimen,

$d_4$  is the correction factor for loss in mass in the second reagent, of the third component undissolved in the second specimen.

### 1.8.1.3. VARIANT 3

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

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

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

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

$P_1\%$  is the percentage of the first clean dry component (component dissolved by the reagent),

$P_2\%$  is the percentage of the second clean dry component (component dissolved by the



reagent),

$P_3\%$  is the percentage of the third clean dry component (component dissolved by the reagent in the second specimen),

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

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

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

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

$d_2$  is the correction factor for loss in mass in the first reagent of the third component undissolved in the first specimen,

$d_3$  is the correction factor for loss in mass in the second reagent of the first component undissolved in the second specimen.

#### 1.8.1.4. VARIANT 4

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

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

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

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

$P_1\%$  is the percentage of the first clean dry component (first soluble component),

$P_2\%$  is the percentage of the second clean dry component (second soluble component),

$P_3\%$  is the percentage of the third clean dry component (insoluble component),

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

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

$r_2$  is the dry mass of the residue after elimination of the first and second component by the first and second reagents,

$d_1$  is the correction factor for the loss in mass of the second component in the first reagent,

$d_2$  is the correction factor for the loss in mass of the third component in the first reagent,

$d_3$  is the correction factor for the loss in mass of the third component in the first and second reagents. " $d_3$ " should be specified in advance by means of experimental methods, whenever possible

1.8.2. Calculation of the percentage of each component with adjustment by agreed allowances, wherever appropriate, correction factors for losses in mass during pre-treatment operations:

If:

$$A = 1 + \frac{a_1 + b_1}{100} ;$$

$$B = 1 + \frac{a_2 + b_2}{100} ;$$

$$C = 1 + \frac{a_3 + b_3}{100} ;$$

then

|   |
|---|
| $P_1A\% = \frac{P_1A}{P_1A + P_2B + P_3C} \times 100$ |
| $P_2A\% = \frac{P_2B}{P_1A + P_2B + P_3C} \times 100$ |
| $P_3A\% = \frac{P_3C}{P_1A + P_2B + P_3C} \times 100$ |

$P_1A\%$  is the percentage of the first clean dry component, including moisture content and loss in mass during pre-treatment,

$P_2A\%$  is the percentage of the second clean dry component, including moisture content and loss in mass during pre-treatment,

$P_3A\%$  is the percentage of the third clean dry component, including moisture content and loss in mass during pre-treatment,

$P_1$  is the percentage of the first clean dry component obtained by one of the formulae given in 1.8.1,

$P_2$  is the percentage of the second clean dry component obtained by one of the formulae given in 1.8.1,

$P_3$  is the percentage of the third clean dry component obtained by one of the formulae given in 1.8.1.

$a_1$  is the agreed allowance of the first component,

$a_2$  is the agreed allowance of the second component,

$a_3$  is the agreed allowance of the third component,

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

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

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

Where a special pre-treatment has been used, the values of  $b_1$ ,  $b_2$  and  $b_3$  shall be determined, if possible, by submitting each of the pure fibre constituents to the pre-treatment applied in the analysis. Pure fibres are those free from all non-fibrous material except that which they normally contain (either naturally or because of the manufacturing process), in the state (unbleached, bleached) in which they are found in the material to be analysed.

Where no clean separate constituent fibres used in the manufacture of the material to be analysed are available, average values of  $b_1$ ,  $b_2$  and  $b_3$  as obtained from tests performed on clean fibres similar to those in the mixture under examination, must be used.

If normal pre-treatment by extraction with light petroleum and water is applied, correction factors  $b_1$ ,  $b_2$  and  $b_3$  may generally be ignored, except in the case of unbleached cotton, unbleached flax and unbleached hemp, where the loss due to the pre-treatment is usually accepted as 4 %, and in the case of polypropylene as 1 %. In the case of other fibres, losses due to the pre-treatment are usually disregarded in calculations.

#### 1.8.3. Note:

Examples of calculations are given in Section 4 of this chapter.

## **2. Method of quantitative analysis by manual separation of ternary fibre mixtures**

### 2.1. FIELD OF APPLICATION

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

### 2.2. PRINCIPLE

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

### 2.3. APPARATUS

- 2.3.1. Weighing bottle or other apparatus giving identical results.
- 2.3.2. Desiccator containing self-indicating silica gel
- 2.3.3. Ventilated oven for drying specimens at  $105 \pm 3$  °C
- 2.3.4. Analytical balance, accurate to 0,0002 g
- 2.3.5. Soxhlet extractor, or other apparatus giving an identical result
- 2.3.6. Needle
- 2.3.7. Twist tester or similar apparatus

### 2.4. REAGENTS

- 2.4.1. Light petroleum, redistilled, boiling range 40 to 60 °C.
- 2.4.2. Distilled or deionised water.

### 2.5. CONDITIONING AND TESTING ATMOSPHERE

from item 1.4. of this Annex

## 2.6. LABORATORY TEST SAMPLE

from item 1.5. of this Annex

## 2.7. PRE-TREATMENT OF LABORATORY TEST SAMPLE

from item 1.6. of this Annex

## 2.8.

### PROCEDURE

#### 2.8.1. Analysis of yarn

Select from the pre-treated laboratory test sample a specimen of mass not less than 1 g. For a very fine yarn, the analysis may be made on a minimum length of 30 m, whatever its mass.

Cut the yarn into pieces of a suitable length and separate the fibre types by means of a needle and, if necessary, a twist tester. The fibre types so obtained are placed in pre-weighed weighing bottles and dried at  $105 \pm 3$  °C until obtaining a constant mass, as described in 1.7.1 and 1.7.2.

#### 2.8.2. Analysis of cloth and knitted fabrics

Take 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 types of fibres, collect them in pre-weighed weighing bottles and proceed as described in 2.8.1. of this Section.

## 2.9. CALCULATION AND EXPRESSION OF RESULTS

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

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

|   |
|---|
| $P_1\% = \frac{100 m_1}{m_1 + m_2 + m_3} = \frac{100}{1 + \frac{m_2 + m_3}{m_1}}$ |
| $P_2\% = \frac{100 m_2}{m_1 + m_2 + m_3} = \frac{100}{1 + \frac{m_1 + m_3}{m_2}}$ |

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

P<sub>1</sub>% is the percentage of the first clean dry component

P<sub>2</sub>% is the percentage of the second clean dry component

P<sub>3</sub>% is the percentage of the third clean dry component

m<sub>1</sub> is the clean dry mass of the first component

m<sub>2</sub> is the clean dry mass of the second component

m<sub>3</sub> is the clean dry mass of the third component

2.9.2. For calculation of the percentage of each component with adjustment by agreed allowances and, where appropriate, by correction factors for losses in mass during pre-treatment, see 1.8.2.

### **3. Method of quantitative analysis of ternary fibre mixtures by combining of manual separation and chemical separation**

The method of manual separation shall be used whenever possible, taking account of the proportions of components before proceeding to any chemical treatment of each of the separate components.

#### **3.1. PRECISION OF THE METHODS**

The precision indicated in each method of analysis of binary fibre mixtures relates to the reproducibility (see Chapter 2 of this Annex relating to methods for quantitative analysis of certain binary textile fibre mixtures).

Reproducibility refers to the reliability, i.e. the closeness of agreement between experimental values obtained by operators in different laboratories or at different times using the same method and obtaining individual results on specimens of an identical homogenous mixture. Reproducibility is expressed by confidence limits of the results for a confidence level of 95 %.

By this is meant that the difference between two results in a series of analyses made in different laboratories would, given a normal and correct application of the method to an identical and homogenous mixture, exceed only in five cases out of 100.

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

Given that in the four variants of the quantitative chemical analysis of ternary fibre mixtures, provision is made for two dissolutions (using two separate specimens for the first three variants and a single specimen for the fourth variant) and, assuming that E1 and E2 denote the precision

of the two methods for analysing binary fibre mixtures, the precision of the results for each component is shown in the following table:

| Component fibre | Variants |         |       |
|-----------------|----------|---------|-------|
|                 | 1        | 2 and 3 | 4     |
| A               | E        | E1      | E     |
| B               | E        | E1+E2   | E1+E2 |
| C               | E1+E2    | E2      | E1+E2 |

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

### 3.2. TEST REPORT

3.2.1. Indicate the variant(s) used to carry out the analysis, the methods, reagents and correction factors.

3.2.2. Give details of any special pre-treatments (see 1.6).

3.2.3. Give the individual results and the arithmetic mean, each to the first decimal place

3.2.4. Whenever possible, state the precision of the method for each component, calculated in accordance with the table in Item 3.1 Section 3

### **4. Examples of the calculation of percentages of the components of certain ternary fibre mixtures using some of the variants described in 1.8.1. Chapter III of this Annex**

Considers the case of a fibre mixture which gave the following components when qualitatively analysed for composition of the material:

1. carded wool; 2. nylon (polyamide); 3. unbleached cotton.

VARIANT No. 1

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

1. Dry mass of the first specimen after pre-treatment is  $(m_1) = 1,6000$  g

2. Dry mass of the residue after treatment with alkaline sodium hypochlorite (polyamide + cotton)  $(r_1) = 1,4166$  g

3. Dry mass of the second specimen after the pre-treatment  $(m_2) = 1,8000$  g

4. Dry mass of the residue after treatment with formic acid (wool+cotton)  $(r_2) = 0,9000$  g

Treatment with alkaline sodium hypochlorite does not entail any loss in mass of polyamide, while unbleached cotton loses 3%, therefore  $d_1$  is 1,00 and  $d_2$  is 1,03.

Treatment with formic acid does not entail any loss in mass for wool or unbleached cotton, therefore  $d_3$  and  $d_4 = 1,00$

If the values obtained by chemical analysis and the correction factors are substituted in the formula under item 1.8.1.1., the following result is obtained:

$$P_1\% (\text{wool}) = [1,03/1,00 - 1,03 \times 1,4166/1,6000 + (0,9000/1,8000) \times (1 - 1,03/1,00)] \times 100 = 10,30$$

$$P_2\% (\text{polyamide}) = [1,00/1,00 - 1,00 \times 0,9000/1,8000 + (1,4166/1,6000) \times (1 - 1,00/1,00)] \times 100 = 50,00$$

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

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

|           |        |
|-----------|--------|
| Wool      | 10,30% |
| Polyamide | 50,00% |
| Cotton    | 39,70% |

These percentages must be corrected according to the formula under item 1.8.2 in order to take account of the agreed allowances and correction factors for any losses in mass after pre-treatment.

As indicated in Annex 9, the agreed allowance is as follows: carded wool 17,00%, polyamide 6,25%, cotton 8,50%, unbleached cotton also shows a loss in mass of 4%, after pre-treatment with light petroleum and water.

Therefore:

$$P_{1A}\% (\text{wool}) = 10,30 \times [1 + (17,00 + 0,0)/100] / [10,30 \times (1 + (17,00 + 0,0)/100) + 50,00 \times (1 + (6,25 + 0,0)/100) + 39,70 \times (1 + (8,50 + 4,0)/100)] \times 100 = 10,97$$

$$P_{2A}\% (\text{polyamide}) = 50,0 \times [(1 + (6,25 + 0,0)/100)/109,8385] \times 100 = 48,37$$

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

The raw material composition of the yarn is therefore as follows:

|           |        |
|-----------|--------|
| Polyamide | 48,4%  |
| Cotton    | 40,6%  |
| Wool      | 11,0%  |
|           | 100,0% |



#### VARIANT No 4

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

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

1. dry mass of the specimen after pre-treatment (m) = 1,6000 g.
2. dry mass of the residue after treatment with alkaline sodium hypochlorite (viscose+cotton)(r<sub>1</sub>) = 1,4166 g
3. dry mass of the residue after the second treatment of the residue r<sub>1</sub> with zinc chloride/formic acid (cotton) (r<sub>2</sub>) = 0,6630 g

Treatment with alkaline sodium hypochlorite does not entail any loss in mass of viscose, while unbleached cotton loses 3%, therefore d<sub>1</sub> = 1,00 and d<sub>2</sub> = 1,03.

As a result of treatment with formic acid – zinc chloride, the mass of cotton is reduced by 2%, so d<sub>3</sub> = (1,03 × 1,02) = 1,0506, rounded at 1,05 (with d<sub>3</sub> as the correction factor for the corresponding loss or the increase in the mass of the third component in the first and the second reagent).

If the values obtained by chemical analysis and correction factors are substituted in the formula given in item 1.8.1.4., the following results are obtained:

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

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

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

As has already been indicated for Variant 1, these percentages must be corrected by the formula indicated in point 1.8.2.

$$P_{1A}\% (\text{wool}) = 10,17 \times [1 + (17,0 + 0,0)/100] / [10,17 \times (1 + (17,00 + 0,0)/100) + 46,32 \times (1 + (13 + 0,0)/100) + 43,51 \times (1 + (8,5 + 4,0)/100)] \times 100 = 10,51\%$$

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

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

The raw material composition of the mixture is therefore as follows:

|         |       |
|---------|-------|
| Viscose | 46,2% |
| Cotton  | 43,3% |
| Wool    | 10,5% |
|         | 100   |

**5. Table of typical ternary fibre mixtures which may be analysed by the selected method from this Annex which is applied for the analysis of binary fibre mixtures (for illustration purposes)**

| Mixture No. | Component fibres     |  |   | Variant    | Number of method used and reagent for binary fibre mixtures   |
|-------------|----------------------|--|---|------------|---|
|             | Component 1          | Component 2                                | Component 3   |            |   |
| 1           | wool or hair         | viscose, cupro or certain types of modal   | cotton  | 1 and/or 4 | 2. (hypochlorite) and 3.(zinc chloride/formic acid)   |
| 2           | wool or hair         | polyamide or nylon                         | cotton, viscose, cupro or modal                       | 1 and/or 4 | 2. (hypochlorite) and 4. (formic acid 80% m/m)  |
| 3           | wool, hair or silk   | certain other fibres                       | viscose, cupro, modal or cotton                       | 1 and/or 4 | 2. (hypochlorite) and 9. (carbon disulphide/acetone 55,5 /44,5% v/v)  |
| 4           | wool or hair         | polyamide or nylon                         | polyester, polypropylene, acrylic or glass fibre      | 1 and/or 4 | 2. (hypochlorite) and 4. (formic acid 80% m/m)  |
| 5           | wool, hair or silk   | certain other fibres                       | polyester, acrylic, polyamide or nylon or glass fibre | 1 and/or 4 | 2. (hypochlorite) and 9. (carbon disulphide/acetone 55,5 /44,5% v/v)  |
| 6           | silk                 | wool or hair                               | polyester   | 2          | 11. (sulphuric acid 75% m/m) and 2. (hypochlorite)  |
| 7           | polyamide or nylon   | acrylic or certain other fibres            | cotton, viscose, cupro or modal                       | 1 and/or 4 | 4. (formic acid 80% m/m) and 8. (dimethylformamide)   |
| 8           | certain chlorofibres | polyamide or nylon                         | cotton, viscose, cupro or modal                       | 1 and/or 4 | 8. (dimethylformamide) and 4. (formic acid,80% m/m) or 9. (carbon - disulphide/acetone, 55,5 /44,5% v/v) and 4. (formic acid 80% m/m) |
| 9           | acrylic              | polyamide or nylon                         | polyester   | 1 and/or 4 | 8. (dimethylformamide) and 4. (formic acid 80% m/m)   |
| 10          | acetate              | polyamide or nylon or certain other fibres | viscose, cotton, cupro or modal                       | 4          | 1. (acetone) and 4. (formic acid 80% m/m)   |

|    |  |  |  |            |  |
|----|--|--|--|------------|--|
| 11 | certain chlorofibres                     | acrylic or certain other fibres          | polyamide or nylon   | 2 and/or 4 | 9. (carbondisulphide/acetone 55,5 /44,5% v/v) and 8. (dimethylformamide)     |
| 12 | certain chlorofibres                     | polyamide or nylon                       | acrylic  | 1 and/or 4 | 9.(carbon-disulfide/acetone 55,5 /44,5% v/v) and 4.<br>(formic acid 80% m/m) |
| 13 | polyamide or nylon                       | viscose, cupro, modal or cotton          | polyester  | 4          | 4.(formic acid), 80% m/m and 7. (sulphuric acid 75% m/m)                     |
| 14 | acetate                                  | viscose, cupro, modal or cotton          | polyester  | 4          | 1. (acetone) and 7. (sulphuric acid 75% m/m)                                 |
| 15 | acrylic                                  | viscose, cupro, modal or cotton          | polyester  | 4          | 8. (dimethylformamide) and 7. (sulphuric acid 75% m/m)                       |
| 16 | acetate                                  | wool, hair or silk                       | cotton, viscose, cupro, modal, polyamide or nylon, polyester, acrylic  | 4          | 1. (acetone) and 2. (hypochlorite)   |
| 17 | triacetate                               | wool, hair or silk                       | {cotton, viscose, cupro, modal, polyamide or nylon, polyester, acrylic | 4          | 6. (dichloromethane) and 2. (hypochlorite)                                   |
| 18 | acrylic                                  | wool, hair or silk                       | polyester  | 1 and/or 4 | 8. (dimethylformamide) and 2. (hypochlorite)                                 |
| 19 | acrylic                                  | silk                                     | wool or hair   | 4          | 8. (dimethylformamide) and 11. (sulphuric acid 75% m/m)                      |
| 20 | acrylic                                  | wool, hair or silk                       | cotton, viscose, cupro or modal  | 1 and/or 4 | 8. (dimethylformamide) and 2. (hypochlorite)                                 |
| 21 | wool, hair or silk                       | cotton, viscose, cupro or modal          | polyester  | 4          | 2. (hypochlorite) and 7. (sulphuric acid 75% m/m)                            |
| 22 | viscose, cupro or certain types of modal | cotton                                   | polyester  | 2 and/or 4 | 3. (zinc-chloride/formic acid) and 7. (sulphuric acid 75% m/m)               |
| 23 | acrylic                                  | viscose, cupro or certain types of modal | cotton   | 4          | 8. (dimethylformamide) and 3.(zinc-chloride/formic acid)<0}                  |

|    |                      |  |                                 |            |   |
|----|----------------------|--|---------------------------------|------------|---|
| 24 | certain chlorofibres | viscose, cupro or certain types of modal | cotton                          | 1 and/or 4 | 9. (carbon-disulphide/acetone, 55,5/44,5% v/v) and 3. (zinc-chloride/formic acid) or 8.(dimethylformamide) and 3.(zinc-chloride/formic acid)<0} |
| 25 | acetate              | viscose, cupro or certain types of modal | cotton                          | 4          | 1. (acetone) and 3.(zinc-chloride/formic acid)  |
| 26 | triacetate           | viscose, cupro or certain types of modal | cotton                          | 4          | 6. (dichloromethane) and 3.(zinc-chloride/formic acid)  |
| 27 | acetate              | silk                                     | wool or hair                    | 4          | 1. (acetone) and 11. (sulphuric acid 75% m/m)   |
| 28 | triacetate           | silk                                     | wool or hair                    | 4          | 6. (dichloromethane) and 11. (sulphuric acid 75% m/m)   |
| 29 | acetate              | acrylic                                  | cotton, viscose, cupro or modal | 4          | 1.(acetone) and 8. (dimethylformamide)  |
| 30 | triacetate           | acrylic                                  | cotton, viscose, cupro or modal | 4          | 6. (dichloromethane) and 8. (dimethylformamide)   |
| 31 | triacetate           | polyamide or nylon                       | cotton, viscose, cupro or modal | 4          | 6. (dichloromethane) and 4. (formic acid 80% m/m)   |
| 32 | triacetate           | cotton, viscose, cupro or modal          | polyester                       | 4          | 6. (dichloromethane) i 7. (sulphuric acid 75% m/m)  |
| 33 | acetate              | polyamide or nylon                       | polyester or acrylic            | 4          | 1. (acetone) and 4. (formic acid 80% m/m)   |
| 34 | acetate              | acrylic                                  | polyester                       | 4          | 1. (acetone) and 8. (dimethylformamide)   |
| 35 | certain chlorofibres | cotton, viscose, cupro or modal          | polyester                       | 4          | 8. (dimethylformamide) and 7. (sulphuric acid, 75% m/m) or 9. (carbon-disulphide/acetone, 55,5 /44,5% v/v) and 7. (sulphuric acid 75% m/m)      |
| 36 | cotton               | polyester                                | elastolefin                     | 2 and/or 4 | 7.(sulphuric acid 75% m/m) and 14. (concentrated sulphuric acid)  |

|    |                     |           |          |            |   |
|----|---------------------|-----------|----------|------------|---|
| 37 | certain modacrylics | polyester | melamine | 2 and/or 4 | 8. (dimethylformamide) and 14. (concentrated sulphuric acid)<0} |
|----|---------------------|-----------|----------|------------|---|

## Annex 9

### AGREED ALLOWANCE APPLIED ON CALCULATION OF FIBRE MASS CONTAINED IN TEXTILE PRODUCT

| Fibre No | Fibres                 | Percentage  |
|----------|------------------------|-------------|
| 1-2      | Wool and animal hair   |             |
|          | combed fibres          | 18,25%      |
|          | carded fibres          | 17,00%<br>* |
| 3        | Animal hair            |             |
|          | combed fibres          | 18,25%      |
|          | carded fibres          | 17,00%<br>* |
|          | Horsehair              |             |
|          | combed fibres          | 16,00%      |
|          | carded fibres          | 15,00%      |
| 4        | Silk                   | 11,00%      |
| 5        | Cotton                 |             |
|          | normal fibres          | 8,50%       |
|          | mercerised fibres      | 10,50%      |
| 6        | Kapok                  | 10,90%      |
| 7        | Flax                   | 12,00%      |
| 8        | True hemp              | 12,00%      |
| 9        | Jute                   | 17,00%      |
| 10       | Abaca                  | 14,00%      |
| 11       | Alfa                   | 14,00%      |
| 12       | Coir                   | 13,00%      |
| 13       | Broom                  | 14,00%      |
| 14       | Ramie (bleached fibre) | 8,50%       |
| 15       | Sisal                  | 14,00%      |
| 16       | Sunn                   | 12,00%      |
| 17       | Henequen               | 14,00%      |
| 18       | Maguey                 | 14,00%      |
| 19       | Acetate                | 9,00%       |
| 20       | Alginate               | 20,00%      |
| 21       | Cupro                  | 13,00%      |
| 22       | Modal                  | 13,00%      |

|    |  |        |
|----|--|--------|
| 23 | Protein                                  | 17,00% |
| 24 | Triacetate                               | 7,00%  |
| 25 | Viscose                                  | 13,00% |
| 26 | Acrylic                                  | 2,00%  |
| 27 | Chlorofibre                              | 2,00%  |
| 28 | Fluorofibre                              | 0,00%  |
| 29 | Modacrylic                               | 2,00%  |
| 30 | Polyamide or nylon                       |        |
|    | discontinuous fibre                      | 6,25%  |
|    | filament                                 | 5,75%  |
| 31 | Aramid                                   | 8,00%  |
| 32 | Polyimide                                | 3,50%  |
| 33 | Lyocell                                  | 13,00% |
| 34 | Polilactide                              | 1,50%  |
| 35 | Polyester                                | 1,50%  |
| 36 | Polyethylene                             | 1,50%  |
| 37 | Polypropylene                            | 2,00%  |
| 38 | Polycarbamide                            | 2,00%  |
| 39 | Polyurethane                             |        |
|    | discontinuous fibre                      | 3,50%  |
|    | filament                                 | 3,00%  |
| 40 | Vinyal                                   | 5,00%  |
| 41 | Trivinyal                                | 3,00%  |
| 42 | Elastodiene                              | 1,00%  |
| 43 | Elastane                                 | 1,50%  |
| 44 | Glass fibre                              |        |
|    | with an average diameter of over 5 µm    | 2,00%  |
|    | with an average diameter of 5 µm or less | 3,00%  |
| 45 | Elastomultiester                         | 1,50%  |
| 46 | elastolefin                              | 1,50%  |
| 47 | Melamine                                 | 7,00%  |
| 48 | Metal fibre                              | 2,00%  |
|    | Metallised fibre                         | 2,00%  |
|    | Asbestos                                 | 2,00%  |
|    | Paper yarn                               | 13,75% |
| 49 | binary polypropylene/polyamide fibre     | 1,00%  |

\*Agreed allowance of 17,00% is also applied when it is not possible to define if a textile product containing wool and/or animal hair, combed or carded.