Pursuant to Article 6 paragraph 1 of the Law on Technical Requirements for Products and Conformity Assessment (Official Gazette of Montenegro 53/11), the Ministry of Economy adopted the following

R U L E B O O K

ON LABELLING AND MARKING OF TEXTILE PRODUCTS*

Subject

Article 1

This Rulebook shall lay down the method of labelling and marking of textile products by means of the use of textile fibre names, affixing a label (hereinafter referred to as: labelling), marking of the fibre composition of textile products and textile products containing non-textile parts of animal origin, determining fibre composition of textile products by quantitative analysis of two-component and three-component mixtures of textile fibres.

Definitions

Article 2

Terms used in this Rulebook shall have the following meanings:

1) textile product shall mean any product in raw condition, semi-finished product and finished product of industrial or manual manufacture composed of textile fibres, irrespective of the manufacturing process applied;
2) textile fibre shall mean:

- a fibre featured by flexibility, fineness and bigger ratio of length and maximum cross section, due to which it is suitable for textile processing, or
- a flexible strip or a tube-shape product whose apparent width does not exceed 5 mm, including also strips cut from wider strips or films made from the substances suitable for textile processing and used for production of fibres.

3) apparent width shall mean the width of the strip or tube-shape product which is bended, flattened or twisted, or the average width if the width is not uniform;
4) textile component shall mean a part of a textile product with a fibre content that can be identified;
5) extraneous fibres shall mean fibres which are not indicated on the label or marking of a textile product;
6) lining shall mean a special component used to make up garments and other textile products, which consists of one or more layers of textile material, attached along one or more edges;
7) affixing a label shall mean providing of data related to textile fibre names on a label attached to a textile product;
8) marking shall mean providing of data related to textile fibre names on a textile product by means of embossing, sewing, embroidering, printing or application of any other similar technology;
9) inclusive affixing of a label shall mean the use of one label for several textile products or components;
10) product for single use shall mean a textile product intended to be used only once or within a limited period of time and which is, under normal conditions of use, not intended to be used again for the same or similar purpose;
11) agreed allowance shall mean the value of moisture content which is added in the procedure of calculation related to the mass of dry, clean fibres taking into account the agreed factors.

Application

Article 3

This Regulation shall apply to all textile products which are made available on the market, as well as to:

1) products containing minimum 80% by weight of textile fibres;
2) material for furniture, umbrella and sunshade coverings consisting of minimum 80% by weight of textile fibres in components of those products which contain textile fibres;
3) textile components which are composed of at least 80% by weight of upper layers or coverings, of: upper layers of multi-layer floor coverings,

- mattress coverings,
- coverings of camping goods,

4) textile products incorporated in other products making their component parts, where their composition is indicated.

Exception from application

Article 4

This Rulebook shall not apply to textile products which are given, on the contractual basis, to physical persons who work in their own homes, or to legal persons who manufacture textile products using the supplied material, which is not their raw material and customised textile products made up at a self-employed tailors.

General requirement for placing on the market of textile products

Article 5

Textile products shall be placed on the market only if they are labelled, and/or marked, by the textile fibre names and accompanied by the documentation providing information as laid down in this Rulebook (hereinafter referred to as: accompanying documentation).

Textile fibre names

Article 6

Only textile fibre names provided in Annex I of this Rulebook can be used for a description of fibre compositions on labels and markings of textile products.

The names listed in Annex I of this Rulebook shall be used exclusively as names for textile fibres whose features correspond to the description of fibres given in Table 1.

The names provided in Annex I shall not be used for other fibres.

The term „silk“ shall not be used as a marking for fibre shape or fibre description which has the form of filament yarn if it does not correspond to the fibre composition provided in Annex I of this Rulebook.

Application for new textile fibre names

Article 7

A manufacturer or his representative can submit a request to the EU Commission for adding a name for a new textile fibre to the list provided in Annex I of this Rulebook.

The request referred to in paragraph 1 of this Article must contain a technical document drawn up in accordance with Annex II which forms an integral part of this Rulebook.

Pure textile products

Article 8

A textile product composed solely of a single type of textile fibre can be labelled or marked as: „100%“, „pure“ or „all“.

The terms referred to in paragraph 1 of this Article, or similar terms, shall not be used for textile products which are not composed solely of a single type of fibre.

It can be considered that a textile product is composed solely of a single type of fibre also if it is contains maximum 2% by weight of extraneous fibres, if the quantity of other fibres is technically justified by means of a good manufacturing practice and it is not added as a matter of routine.

The provision of paragraph 3 of this Article shall not apply to textile products made up of mixtures of textile fibres incorporating virgin wool as one of their components.

By way of exception from paragraph 3 of this Article, a textile product which has been a subject of a carding process can be considered as composed of solely a single fibre if it does not contain more than 5% by weight of other fibres, if this quantity is technically justified by means of a good manufacturing practice and it is not added as a matter of routine.

Virgin wool products

Article 9

A textile product can be labelled or marked by the name „virgin wool“ if it consists solely of wool fibres which have not been previously incorporated in a finished product, which has not undergone any spinning or felting processes except the processes necessary for manufacture of that product and which have not been damaged by the processing or use.

By way of exception from paragraph 1 of this Article, the name „virgin wool“ can be used to indicate the content of wool in a textile product composed of textile fibre mixture, if:

1) all the wool contained in the textile product concerned fulfils requirements for wool fibre set out in paragraph 1 of this Article,
2) virgin wool contained in that textile product account for minimum 25% of total weight of the textile product concerned, and
3) that textile product incorporates only virgin wool and maximum one more type of some other fibre obtained as a result of scribbling procedure.

The total composition of the textile product referred to in paragraph 2 of this Article shall be declared in percentages.

The extraneous fibres in textile products referred to in paragraph 1 and 2 of this Article, including wool products which were a subject of a carding process, shall not exceed 0,3% by weight of the textile product, must be technically justified by means of a good manufacturing practice and they must not be added as a matter of routine.

Textile products made of several types of fibres

Article 10

Textile products containing several types of fibres shall be labelled and/or marked by declaring the name and percentage by weight of all textile fibres incorporated in the textile product concerned, in descending order.

By way of exception from paragraph 1 of this Article,for a textile fibre accounting for maximum 5% by weight of the textile product consisting of several types of fibres, or for several types of textile fibres which jointly account for maximum 15% by weight of the textile product, the term „other fibres“ can be used provided that the content expressed in percentage of those (that) other fibre cannot be determined at the time of manufacture.

The total percentage of those fibres in relation to the total weight of the textile product shall be also given together with the term “other fibres.”

For textile products with pure cotton warp and pure flax weft, in which a percentage of flax is minimum 40% of the total weight of the unsized fabric the name „pure cotton fabric“ can be indicated, and which shall be accompanied by the fibre composition by means of the term „pure cotton warp – pure flax“.

By way of exception from Article 6 paragraph 1 of this Rulebook, for textile products whose fibre composition was not possible to be determined at the time of manufacture, the term „mixed fibres“ or „unspecified fibre composition” may be disclosed on the label or marking.

As an exception from paragraph 1 of this Article, for textile fibres whose names are not listed in Annex I of this Rulebook a term „other fibres“ may be used, while the total percentage of all such fibres in relation to the total weight of a textile product concerned shall be disclosed together with this term.

Decorative fibres and fibres with antistatic effect

Article 11

Visible separate fibres which are exclusively decorative and do not exceed 7% of the weight of the finished product, shall not be considered when defining the fibre composition of the textile products referred to in Articles 8 and 10 of this Rulebook.

Concerning metal and other fibres which are incorporated with the aim to obtain an antistatic effect and which do not exceed 2 % of the weight of the finished textile product shall not be considered when defining the fibre composition of textile products referred to in Article 8 and 10 of this Rulebook.

With regards to textile products provided for in Article 10 paragraph 5 of this Rulebook, for which it was not possible to define the fibre composition at the time of manufacture, the percentage set out in paragraphs 1 and 2 of this Article shall be calculated on the weight of the warp and on the weight of the weft separately.

Multi-component textile products

Article 12

Any textile product consisting of two or more textile components with different fibre composition shall bear a label or marking providing the fibre composition of each textile component.

The labelling or marking referred to in paragraph 1 of this Article shall not be obligatory for textile components which:

- are not main linings, and
- represent less than 30% of the total weight of the textile product.

In case two or more textile products have the same fibre composition and form one unit, they may bear only one label or marking.

Textile products containing non-textile parts of animal origin

Article 13

The presence of non-textile parts of animal origin in textile products shall be stated by the term „Contains non-textile parts of animal origin“ on a label or marking of a product containing non-textile parts, in the event when textile products containing those non-textile parts are made available on the market.

The labelling and/or marking of the fibre composition of textile products shall be conducted in such a way so as not to mislead the consumer and to be easy for him to understand them.

Labelling and marking of specific textile products

Article 14

Labelling and/or marking of the fibre composition of specific textile products is provided for in Annex III which forms a constituent part of this Rulebook.

Labels and markings

Article 15

Textile products which are made available on the market shall bear a label or a marking with indicated fibre composition.

Labels and markings on textile products must be durable, easily legible, visible, accessible and firmly attached.

As an exception from paragraph 1 of this Article, it is possible ro replace or supplement labels and markings by accompanying documentation in the event when textile products are supplied to economic operators within the supply chain or when textile products are supplied in accordance with regulations on public procurements.

Names of textile fibres and descriptions of fibre compositions provided for in Article 6 and Articles 8 to 10 of this Rulebook must be clearly stated in the documentation referred to in paragraph 3 of this Article.

Acronyms shall not be given on labels and markings, except in cases of mechanised processing code or acronyms as indicated in accordance with the international standards, if those are explained in the accompanying documentation.

Obligation to affix the label or marking

Article 16

When a textile product is placed on the market, the manufacturer shall provide the label or marking for the concerned textile product and shall be responsible for the accuracy of the given information.

If the manufacturer does not have a head office in Montenegro, the importer shall affix the label or marking on the textile product and shall be held responsible for the accuracy of the information given on the label, and/or on the marking.

A distributer shall be considered a manufacturer if he places the product on the market under his name or trademark, or if he himself attaches the label or changes its content.

When making the product available on the market, the distributer shall ensure that the textile product bears an appropriate label or marking as set out in this Rulebook.

In the event when making the textile product available on the market, the suppliers referred to in paragraph 1 to 4 of this Article shall ensure that any information provided is not in disrepancy with names of textile fibres and descriptions of textile fibres composition laid down in this Rulebook.

The use of textile fibres names and descriptions of fibre compositions

Article 17

When making a textile product available on the market, descriptions of the fibre composition referred to in Article 6 and Article 8 to 10 of this Rulebook shall be stated in catalogues and other advertising materials, on packaging, labels and markings in such a way that they are easily legible, visible, clear and printed in the identical font, style and size.

Descriptions referred to in paragraph 1 of this Article, must be easily visible for the costumer before the purchase, as well as when the purchase is made by electronic means.

A trademark or a name of the economic operator can be given directly before or after the description of the textile fibre composition referred to in Article 6 and Article 8 to 10 of this Rulebook.

If a trademark or a name of the economic operator contains as an independent word, a root of the word or an adjective, one of the textile fibres names listed in Annex I of this Rulebook or a name that can be wrongly interpreted as a fibre name, such trademark or a name shall obligatory be stated before or after the description of the textile fibre referred to in Article 6 and Article 8 to 10 of this Rulebook. Other information are always shown separately.

Labels and markings must be given in Montenegrin language, unless otherwise stipulated by a special regulation.

As an exception from paragraph 5 of this Article, in the case of small quantities of threads on bobbins, reels, skeins and similar, affixing of a label and marking in Montenegrin language shall be done only in case of the inclusive labelling during packaging.

If the products referred to in paragraph 6 of this Article are sold individually, they shall be labelled or marked in any of the official languages of the European Union if they are inclusively labelled in Montenegrin language.

Special examples of labelling and marking

Article 18

Stating the names of textile fibres and textile products fibre composition on labels and markings of textile products, provided for in Annex IV which forms an integral part of this Rulebook, is not obligatory.

If a trademark or a name of the economic operator contains, as an independent word, as a root of the word or as an adjective, one of the textile fibre names listed in Annex I of this Rulebook or a name which is not listed in that Annex, Article 12 and Article 15 to 17 of this Rulebook shall be applicable.

In case when the products provided for in Annex V, which forms an integral part of this Rulebook, are of the same type and the fibre component, they can be made available on the market with an inclusive label.

Regarding textile products which are sold by the metre, the fibre composition shall be indicated on the length or at the end of each roll which is 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 their fibre composition shall be shown to each purchaser within the supply chain, including the final costumer.

Determining fibre composition

Article 19

Determining fibre composition of textile products shall be done by means of checks on the conformity of the fibre composition of textile products in relation to the information on the fibre composition incorporated in those products, implemented through:

- methods of analysis of textile products composed of two-components and three-components mixtures of textile fibres provided for in Annex VII which form an integral part of this Rulebook, or
- harmonized standards referred to in the above mentioned Annex.

When determining a fibre composition referred to in Articles 8 to 10 of this Rulebook, the components of textile products listed in Annex VI, which forms an integral part of this Rulebook, shall not be taken into account.

The fibre composition of textile products, in accordance with Articles 8 to 10 hereto, shall be determined by adding the appropriate agreed allowance provided for in Annex VIII, which forms an integral part of this Rulebook, to the dry mass of each fibre, not taking into account parts of textile products provided for in Annex VI hereto.

The laboratories which perform testing of textile mixtures for which there is not a unique method of analysis in the European Union shall determine the fibre composition of such mixtures by stating the results obtained, methods used and the accuracy degree in their testing report.

Allowed tolerances

Article 20

Besides for the virgin wool and mixtures of textile fibres containing virgin wool, the presence of „extraneous fibres“ pursuant to Article 9 paragraph 4 of this Rulebook shall not be necessarily disclosed if the percentage of those fibres does not exceed the following values:

1) 2% of the total weight of the textile product, if this quantity is technically justified by means of a good manufacturing practice and is not added as a matter of routine; or
2) 5% of the total weight in case of the textile product produced by applying a carding process, if this process is unavoidable in good manufacturing practice, and is not added as a matter of routine.

Concerning a textile product for which the fibre composition is expressed in line with Article 9 of this Rulebook, a tolerance of 3% shall be allowed, when the percentage of fibre content disclosed on the marking or label is compared to the percentage of that fibre received by means of testing in accordance with Article 19 of this Rulebook.

The tolerances referred to in paragraph 2 of this Article shall also apply to:

1) fibres that can be designated by the term „other fibres“, in accordance with Article 10 paragraph 2 of this Rulebook;
2) percentage of virgin wool referred to in Article 9 paragraph 2 point 2 hereto.

The allowed tolerances provided for in paragraphs 1 and 2 of this Article shall be calculated separately and the total weight taken into account when calculating the allowed tolerances referred to in this paragraph shall be the total weight of textile fibres of the finished product reduced for the weight of „extraneous fibres“ detected when applying the tolerances referred to in paragraph 2 of this Article.

Calculation of a cumulative sum of allowed tolerances laid down in paragraphs 2 and 3 of this Article shall be permitted only if the testings show that any detected textile fibres, by applying the allowed tolerances referred to in paragraph 2 of this Article, are of the same chemical content, as well as one or several types of fibres disclosed on the label or marking.

Approval for higher tolerances

Article 21

The European Commission can approve tolerances higher than those tolerances set out in Article 20 paragraphs 2 to 4 of this Rulebook in the case of special textile products whose manufacturing process requires higher tolerances than those set out in Article 20 paragraph 2 and 3 of this Rulebook.

Before placing the textile product on the market, the manufacturer shall submit to the European Commission a request for issuing of the approval for tolerances set out in paragraph 1 of this Article, which contains reasons and evidences of exceptional manufacturing circumstances.

The approval shall be issued on the basis of the prescribed documentation.

Delayed application

Article 22

Provisions of Article 7, Article 17 paragraph 6 and Article 21 of this Rulebook shall be applied as from the day of accession of Montenegro to the European Union.

Textile products placed on the market

Article 23

The manufacturer and/or his representative, or the importer, may, at the latest until 1 January 2016, place on the market and/or put it into service a product which was, until the enforcement of this Rulebook, manufactured in accordance with requirements set out in regulations listed in Article 24 of this Rulebook.

Cessation of application

Article 24

As from the date on which this Rulebook is applied, the application of the following rulebooks shall cease: Rulebook on the Mandatory Certification of Jute and Conditions for Cooperative Organizations Authorized to Certify Jute Products (Official Gazette of SFRY 8/91), Decree on the Mandatory Certification of Cotton (Official Gazette of SFRY 65/84 and 44/88), Decree on the Mandatory Certification of Wool (Official Gazette of SFRY 65/84) and Decree on Textile Products with Mandatory Quality Certificates for Market Placement (Official Gazette of FRY 14/92).

Entering into force

Article 25

This Rulebook shall enter into force on the eighth day following that of its publication in the Official Gazette of Montenegro and shall be applied as from 1 July 2015.

Number: 01-2322/2
Podgorica, 4 November 2014.

Minister,
Vladimir Kavarić, *hand-signature*

*This Rulebook transposes the Regulation number 1007/11 of the European Parliament and of the Council on textile fibres names and related labelling and marking of the fibre composition of textile products.

ANNEX I

List of textile fibre names

(Article 6 of this Rulebook)

Table 1

Number	Name	Fibre description
1	Wool	fibre from sheep's or lambs' fleeces (Ovis aries) or a mixture of fibres from sheep's or lambs' 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, kashmir 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 the 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 (Gossypium)
6	Kapok	fibre obtained from the inside of the kapok fruit (Ceiba pentandra)
7	Flax (or linen)	fibre obtained from the bast of the flax plant (Linum usitatissimum)
8	True hemp	fibre obtained from the bast of hemp (Cannabis sativa)
9	Jute	fibre obtained from the bast of Corchorus olitorius and Corchorus capsularis. For the purposes of this Rulebook, bast fibres obtained from the following species shall be treated in the same way as jute: Hibiscus cannabinus, Hibiscus sabdariffa, Abutilon avicennae, Urena lobata, Urena sinuata
10	Abaca (manila hemp)	fibre obtained from the sheathing leaf of Musa textilis
11	Alfa	fibre obtained from the leaves of Stipa tenacissima
12	Coir (coconut)	fibre obtained from the fruit of Cocos nucifera
13	Broom	fibre obtained from the bast of Cytisus scoparius and/or Spartium Junceum
14	Ramie	fibre obtained from the bast of Boehmeria nivea and Boehmeria tenacissima
15	Sisal	fibre obtained from the leaves of Agave sisalana
16	Sunn	fibre from the bast of Crotalaria juncea
17	Henequen	fibre from the bast of Agave fourcroydes
18	Maguey	fibre from the bast of Agave cantala

Table 2

Number	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	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: B C (cN) ≥ 1,3 ffiffiffi T p þ 2 T B M (cN) ≥ 0,5 ffiffiffi T p where T is the mean linear density in decitex
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 pattern
30	Polyamide or nylon	fibre formed from synthetic 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	Trivinyl	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	Polypropylene / polyamide two-component fibre	Two-component fibre consisting of between 10% and 25% per weight of polyamide fibres incorporated in polypropylene matrix

ANNEX II

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

(Article 7 of this Rulebook)

A technical document to be attached to an application for the inclusion of a new textile fibre name in the list set out in Annex I of this Rulebook, as provided for in Article 6 of this Rulebook, shall contain the following information:

1) Proposed name of the textile fibre:
   The name proposed shall be related to the chemical composition and shall provide information about the characteristics of the fibre, if appropriate. The name proposed shall be free of any intellectual property rights and shall not be linked to the manufacturer.
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 documentation 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.
4) Proposed agreed allowance to be used in the calculation of fibre composition.
5) Sufficiently developed identification and quantification methods, including experimental data:

The applicant shall evaluate the possibility to use the methods listed in Annex VII of this Rulebook or the harmonised standards to be introduced in that Annex to analyse the most expected commercial mixtures of the new textile fibre with other textile fibres and shall propose at least one of those methods. For those methods or harmonised standards where the textile fibre can be considered as an insoluble component, the applicant shall evaluate the mass correction factors of the new textile fibre. All the experimental 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, robustness and repeatability of the methods shall be provided with the documentation.

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 accordance with appropriate regulations.
7) Additional information to support the application: production process, consumer relevance.

The manufacturer or his representative 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 his representative can also be requested.

ANNEX III

LABELLING AND MARKING OF SPECIFIC TEXTILE PRODUCTS

(Article 14 of this Rulebook)

	Products	**Labelling and marking provisions**
1	The following corsetry products: a) Brassières b) Corsets and girdles c) Corselets	The fibre composition shall be indicated on the label and marking by stating the composition of the whole product or separately of the following components: -the outside and the inside fabric of the cups -back -the front and the rear side and -side panels -the outside and inside fabric and surface of cups -the front and rear stiffening panels and -the side panels
2	Other corsetry products not listed above	The fibre composition of the textile product 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.
3	All 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.
4	Etch-printed textiles	The fibre composition of the textile product 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.
5	Embroidered textiles	The fibre composition of the textile product 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
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	The fibre composition of the textile product 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.
7	Velvet and plush textiles, or textiles resembling velvet or plush	The fibre composition of the textile product 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.
8	Floor coverings and carpets of which the backing and the use-surface are composed of different fibres	The fibre composition may be stated for the use-surface alone. The use-surface must be mentioned by name.

ANNEX IV

TEXTILE PRODUCTS FOR WHICH LABELLING OR MARKING IS NOT MANDATORY

(Article 18 paragraph 2 of this Rulebook)

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 pile 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 2
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 2
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 subject to the regulations on medicinal products and covered by a reference to those regulations, disposable bandages for medical and orthopaedic use and orthopaedic textile products in general
37. Textile products including cordage, ropes and string referred to in Annex V point 12 of this Rulebook normally intended:
    a) for use as equipment components in the manufacture and processing of goods;
    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 V

PRODUCTS FOR WHICH INCLUSIVE LABELLING IS POSSIBLE

(Article 18 paragraph 3 of this Rulebook)

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 packaging sold as such
12. Packing string and agricultural twine; string, cordage and ropes other than those falling within point 37 Annex IV of this Rulebook. For the products falling within this item and sold in cut lengths, the inclusive labelling shall be that of the reel. The cordage and ropes falling within this item include those used in mountaineering 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 VI
COMPONENTS OF TEXTILE PRODUCTS NOT TO BE TAKEN INTO ACCOUNT FOR THE DETERMINATION OF FIBRE COMPOSITION OF TEXTILE PRODUCTS

(Article 19 paragraph 2 of this Rulebook)

Name of textile product	Components not taken into account when determining fibre composition of textile products
a) All textile products	(1) Non-textile parts, selvedges, labels and badges, edgings and trimmings, 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. (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 reinforcement yarns.
f) Tights	Additional elastic yarns used in the cuff and reinforcement yarns.
g) All textile products other than those under points b) to f) of this Annex	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, subject to Article 11(2) of this Rulebook, linings For the purposes of this provision: (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; (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 VII

METHODS OF QUANTITATIVE ANALYSIS OF TEXTILE PRODUCTS COMPOSED OF TWO-COMPONENT AND THREE-COMPONENT MIXTURES OF TEXTILE FIBRES

(Article 19 paragraph 1 of this Rulebook)

CHAPTER I

Section I

Preparation of laboratory samples and test specimens to determine the fibre composition of textile products

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 the individual test specimen.

2. DEFINITIONS

2.1. Bulk source

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.

2.2. Laboratory bulk sample

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 adequately overcome the variability of the bulk source and to facilitate ease of handling in the laboratory. For made-up and finished articles the procedure of taking laboratories samples is described in point 7 of this Chapter.

2.3. Laboratory test sample

That portion of the laboratory bulk sample that is subjected to pre-treatment to remove non-fibrous matter, and from which test specimens are taken. The size and nature of the laboratory test sample shall be sufficient to overcome adequately the variability of the laboratory bulk sample (point 1 of this Chapter).

2.4. Test specimen

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

3. PRINCIPLE

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

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

4. SAMPLES FROM LOOSE FIBRES

4.1. Unorientated fibres

Obtain the laboratory test sample by selecting tufts at random from the laboratory bulk sample. Mix thoroughly the whole of the laboratory test sample 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'. Subject the web or mixture, including loose fibres and fibres adhering to the equipment used for mixing, to pre-treatment. Then select test specimens, in proportion to the respective masses, from the web or mixture, from the loose fibres and from the fibres adhering to the equipment.
If the card web remains intact after pre-treatment, select the test specimens in the manner described in point 4.2. of this Chapter. If the card web is disturbed by the pre-treatment, select each test specimen by removing at random at least 16 small tufts of suitable and approximately equal size and then combine them.

4.2. Orientated fibres (cards, webs, slivers, rovings)

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

5. SAMPLING YARN

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 tex of the yarn is "t" and the number of packages selected from the laboratory bulk sample is „n", then to obtain a test sample of 10 g, the length of yarn to be withdrawn from each package is 10^6/nt cm.

If nt is high, i.e. more than 2 000, wind a heavier skein and cut it across in two places to make a tow of suitable mass. The ends of any sample in the form of a tow shall be securely tied before pre-treatment and test specimens taken from a place remote from the tie bands.

5.2. Yarn on warp

Take the laboratory test sample by cutting a length from the end of the warp, not less than 20 cm long and comprising all the yarns in the warp except the selvedge yarns, which are rejected. Tie the bunch of threads together near one end. If the sample is too large for pre-treatment as a whole divide it into two or more portions, each tied together for pre-treatment, and reunite the portions after each has been pre-treated separately. Take a test specimen by cutting a suitable length from the laboratory test sample from the end remote from the tie band, and comprising all the threads in the warp.
For warp of „N" threads of tex „t", the length of a specimen of mass 1 g is 10 5 /Nt cm.

6. SAMPLING FABRIC

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

Cut a diagonal strip from one corner to the other and remove the selvedges. This strip is the laboratory test sample. To obtain a laboratory test sample of x g, the strip area shall be x10 4 /G cm 2 , where G is the mass of the cloth in 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.

6.2. From a laboratory bulk sample consisting of several cuttings

Treat each cutting as described in 6.1. of this Chapter, and give each result separately

7. SAMPLING MADE-UP AND FINISHED PRODUCTS

The bulk laboratory 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.
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.

II Introduction to the 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. This first part of the Annex gives the information common to the analyses by this method of all fibre mixtures dealt with in the Annex, whatever their composition. It 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 parts of this Rulebook.
Mixtures of fibres during processing and, to a lesser extent, finished textiles may contain non-fibrous matter, such as fats, waxes or dressings, or water-soluble matter, either occurring naturally or added to facilitate processing. Non- fibrous matter must be removed before analysis. For this reason a method for removing oils, fats, waxes and water- soluble matter is also given.

In addition, textiles may contain resins or other matter added to confer special properties. Such matter, including dyestuffs in exceptional cases, may interfere with the action of the reagent on the soluble component and/or it may be partially or completely removed by the reagent. This type of added matter may thus cause errors and 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 IX.
Before proceeding with any analysis, all the fibres present in the mixture shall 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 TWO-COMPONENT TEXTILE FIBRE MIXTURES

I General information common to the methods given for the quantitative chemical analysis of textile two-component 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, usually by selective solution. (Method No. 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
I.3.1.1. Filter crucibles and weighing bottles large enough to contain such crucibles, or any other apparatus giving identical results.
I.3.1.2. Vacuum flask.
I.3.1.3. Desiccator containing self-indicating silica gel.
I.3.1.4. Ventilated oven for drying specimens at 105 ± 3 °C.
I.3.1.5. Analytical balance, accurate to 0,0002 g.

I.3.1.6. Soxhlet extractor or other apparatus giving identical results.
I.3.2. Reagents.
I.3.2.1. Light petroleum, redistilled, boiling range 40 to 60 °C.
I.3.2.2. Other reagents are specified in the appropriate section of each method.
I.3.2.3. Distilled or deionised water.
I.3.2.4. Acetone.
I.3.2.5. Orthophosphoric acid.
I.3.2.6. Urea.
I.3.2.7. Sodium bicarbonate.
All reagents used shall be chemically pure.

1.4. STANDARD 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 (see Chapter 1.1.)

Where a substance not to be taken into account in the percentage calculations (see Article 19 of this 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 laboratory test sample in a Soxhlet extractor with light petroleum for 1 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 1 hour and then soaking it in water at 65 ± 5 °C for a further hour, agitating the liquor from time to time. Use a liquor-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 procedure just described shall be slightly modified, in that light petroleum ether shall be replaced by acetone.

In the case of two-component 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, 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 4 hours and not more than 16 hours at 105 ± 3 °C in a ventilated oven with the oven door closed throughout. If the drying period is less than 14 hours, the specimen must be weighed to check that its mass has become constant. The mass may be considered to have become constant if, after a further drying period of 60 minutes, its variation is less than 0,05 %.
Avoid handling crucibles and weighing bottles, specimens or residues with bare hands during the drying, cooling and weighing operations.
Dry specimens in a weighing bottle with its cover beside it. After drying, stopper the weighing bottle before removing it from the oven, and transfer it quickly to the desiccator.
Dry the filter crucible in a weighing bottle with its cover beside it in the oven. After drying, close the weighing bottle and transfer it quickly to the desiccator.
Where apparatus other than a filter crucible is used, drying operations in the oven 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, the latter placed beside the balance, until complete cooling of the weighing bottles is attained, and in any case for not less than 2 hours.

1.7.1.3. Weighing

After cooling, complete the weighing of the weighing bottle within 2 minutes of its removal from the desiccator. Weigh to an accuracy of 0,0002 g.

1.7.2. Procedure

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

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. 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 shall be made by applying the formula given in point 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 test 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 for 'd' are given in the relevant section of each method.
Of course, these values for 'd' are the normal values applicable to chemically undegraded fibres.

1.8.2. Calculation of 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:

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

where:
P_{1A}%- is the percentage of insoluble component adjusted by agreed allowances and for loss in mass during pre- treatment,
P_1- is the percentage of clean dry insoluble component as calculated from the formula shown in point 1.8.1,
a_1 is the agreed allowance for the insoluble component (see Annex VIII of this Rulebook),
a_2 is the agreed allowance for the soluble component (see Annex VIII 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 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 b1 and b2 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 b1 and b2 may generally be ignored, except in the case of unbleached cotton, unbleached flax (or linen) and unbleached hemp, where the loss due to the pre-treatment is conventionally taken as 4 %, and in the case of polypropylene, where it is taken as 1 %.
In the case of other fibres, losses due to the pre-treatment are conventionally disregarded in calculations.

II Method of quantitative analysis by manual separation

II.1 FIELD OF APPLICATION

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

II.2. PRINCIPLE

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

II.3. APPARATUS

II.3.1. Weighing bottle or any other apparatus giving identical results.

II.3.2. Desiccator containing self-indicating silica gel.
II.3.3. Ventilated oven for drying specimens at 105 ± 3 °C.
II.3.4. Analytical balance, accurate to 0,0002 g.
II.3.5. Soxhlet extractor, or other apparatus giving an identical result.
II.3.6. Needle.
II.3.7. Twist tester or similar apparatus.

II.4. REAGENTS

II.4.1. Petroleum, boiling range 40-60°C.
II.4.2. Distilled or deionised water.
II.4.3. Acetone.
II.4.4. Orthophosphoric acid.
II.4.5. Urea.
II.4.6. Sodium bicarbonate.

All reagents used shall be chemically pure.

II.5. STANDARD TESTING ATMOSPHERE

See 1.4.

II.6. LABORATORY TEST SAMPLE

See 1.5.

II.7. PRE-TREATMENT OF LABORATORY TEST SAMPLE

See 1.6.

II.8. PROCEDURE

II.8.1. Analysis of yarn

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

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

II.8.2. Analysis of cloth

Select from the pre-treated laboratory test sample, well away from all selvedges, a specimen of mass not less than 1 g, with edges carefully trimmed to avoid fraying and running parallel with weft or warp yarns, or in the case of knitted fabrics in the line of wales and courses. Separate the different fibre types, collect them in pre-weighed weighing bottles and proceed as described in point 2.8.1.

II.9. CALCULATION AND EXPRESSION OF RESULTS

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

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

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

P_1% - is the percentage of the first clean, dry component,
m_1 - is the clean, dry mass of the first component,
m_2 . is the clean, dry mass of the second component.

II.9.2. For calculation of the percentage of each component with adjustment by agreed allowances and, where appropriate, by correction factors for loss of matter during pre-treatment, see point 1.8.2. Section I of this Chapter.

III.1. PRECISION OF THE METHODS

The precision indicated in individual methods relates to the reproducibility.

The reproducibility refers to the reliability, i.e. the closeness of agreement between experimental values obtained by operators in different laboratories or at different times using the same method and obtaining individual results on specimens of an identical consistent mixture.

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

Therefore, 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 100.

III.2. TEST REPORT

III.2.1. State that the analysis was conducted in accordance with this method.
III.2.2. Give details of any special pre-treatment (see 1.6).
III.2.3. Give the individual results and the arithmetic mean, each to an accuracy of 0,1.

IV Special methods – Summary table

	Summary table		
Method	Soluble component	Insoluble component	**Reagent/description**
1	Acetate	Certain other fibres	Acetone
2	Certain protein fibres	Certain other fibres	Hypochlorite
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, polyamides and 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	Certain other fibres	Hot formic acid, 90 % m/m

METHOD NO.1

ACETATE AND CERTAIN OTHER FIBRES

(Acetone method)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to two-component fibre mixtures of:

1. acetate (19)

with

2. wool (1), animal hair, (2 and 3), silk (4), cotton (5), flax or linen (7), true hemp (8), jute (9), abaca (10), alfa (11), coir (12), broom (13), ramie (14), sisal (15), cupro (21), modal (22), protein (23), viscose (25), acrylic (26), polyamide or nylon (30) polyester (35), elastomultiester (45), elastolefin (46) and melamine (47) fibres and polypropylene / polyamide two-component fibres (49).

This method shall not be 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 total mass of fibres in 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

A test procedure, according to the general instrictions, shall be the following:

To the test 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 1 hour. Transfer the residue to the filter crucible. Wash the residue in the filter crucible with acetone and drain with suction. Refill the crucible with acetone and allow to drain under gravity.

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

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of 'd' is 1,00, except for melamine, for which 'd' = 1,01.

6. PRECISION

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

METHOD No. 2

CERTAIN PROTEIN FIBRES AND CERTAIN OTHER FIBRES
(Method using hypochlorite)

1. Field of application

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

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

with

2. cotton (5), cupro (21), modal (22), viscose (25), acrylic (26), chlorofibres (27), polyamide or nylon (30), polyester (35), polypropylene (37), elastane (43), glass fibre (44), elastomultiester (45), elastolefin (46) and melamine (47) and olypropylene / polyamide two-component fibres (49).

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 ± 2 °C.

3.2 Reagents

a) Hypochlorite reagent

1) Lithium hypochlorite solution

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

2) Sodium hypochlorite solution

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

(b) Acetic acid, dilute solution
Dilute 5 ml of glacial acetic acid to 1 litre with water.

4. TEST PROCEDURE

A test procedure, according to the general instructions, shall be the following: 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, the reaction heat of this method must be distributed and removed. Otherwise, considerable errors may be caused by the incipient dissolution of the non-soluble fibres.

After 40 minutes, filter the flask contents through a weighed glass-filter crucible and transfer any residual fibres into the filter crucible by rinsing the flask with a little hypochlorite reagent. Drain the crucible with suction and wash the residue successively with water, dilute acetic acid, and finally water, draining the crucible with suction after each addition. Do not apply suction until each washing liquor has drained under gravity.
Finally, drain the crucible with suction, dry the crucible with the residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of ‘d’ is 1,00, except for cotton, viscose, 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 ± 1 for a confidence level of 95 %.

METHOD 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, after removal of non-fibrous matter, to two-component fibre mixtures of:

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

with

2. cotton (5), elastolefin (46) and melamine (47).

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 corrected mass is expressed as a percentage of the dry mass of the mixture. The percentage of dry viscose, cupro or modal fibre is found by difference.

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

3.1. Apparatus

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

b) Apparatus for maintaining flasks at 40 ± 2 °C.

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:

It is necessary that all reagents used shall be chemically pure and 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) 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 2,5 hours, 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, point 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 the ammonia solution for 10 minutes, one may, for example, use a filter crucible adaptor fitted with a tap by which the flow of the ammonia solution can be regulated).
Do not apply suction until each washing liquor has drained under gravity.
Finally, drain the remaining liquid with suction, dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Results shall be calculated as described in the general instructions. The value of ‘d’ is 1,00, except for cotton for which "d" is 1,02; and 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 ± 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 applied, after removal of non-fibrous matter, to two-component mixtures:

1. polyamide or nylon (30)

   and

2. 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).

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

A test procedure, according to general procedures, is the following: 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, 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’ = 1,01.

6. PRECISION

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

METHOD No. 5

ACETATE AND CERTAIN OTHER FIBRES

(Method using benzyl alcohol)

1. FIELD OF APPLICATION

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

1. acetate (19)

   with

2. triacetate (24), polypropylene (37), elastolefin (46) melamin (47) and polypropylene / poliamide two-component fibres (49).

2. PRINCIPLE

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

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

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

3.1. Apparatus

a) Glass-stoppered conical flask of at least 200 ml capacity.
b) Mechanical shaker.
c) Thermostat or other apparatus for keeping the flask at a temperature of 52 ± 2 °C.

3.2. Reagents

a) Benzyl alcohol.
b) Ethanol.

4. TEST PROCEDURE

The test procedure, according to the general instructions, is the following: 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 ± 2 °C, and shake for 20 minutes at this temperature.
Instead of using a mechanical shaker, the flask may be shaken vigorously by hand.
Decant the liquid through the weighed filter crucible. Add a further dose of benzyl alcohol in the flask and shake as before at 52 ± 2 °C for 20 minutes.
Decant the liquid through the crucible. Repeat the cycle of operations a third time.
Finally pour the liquid and the residue into the crucible; wash any remaining fibres from the flask into the crucible with an extra quantity of benzyl alcohol at 52 ± 2 °C. Drain the crucible thoroughly.
Transfer the fibres into a flask, rinse with ethanol and after shaking manually decant through the filter crucible.
Repeat this rinsing operation two or three times. Transfer the residue into the crucible and drain thoroughly. Dry the crucible and the residue and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of ‘d’ is 1,00, except for melamine, for which ‘d’ = 1,01.

6. PRECISION

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

METHOD No.6

TRIACETATE OR POLYLACTIDE AND CERTAIN OTHER FIBRES

(Method using dichloromethane)

1. FIELD OF APPLICATION

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

1. triacetate (24) or polylactide (34)

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 fibre (44), elastomultiester (45), elastolefin (46) and melamine (47) and polypropylene/polyamide two-component fibres (49).

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 or polylactide 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

To the test 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 to wet out the test specimen and stand for 30 minutes at room temperature, shaking the flask every 10 minutes. 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

The results are calculated 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 ± 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, after removal of non-fibrous matter, to binary fibre mixtures of:

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

with

2. polyester (35), polypropylen (37), elastomultiester (45) and elastolefin (46) and polypropylen/polyamide two-component fibres (49).

2. PRINCIPLE

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

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

3.1. Apparatus

a) Glass-stoppered conical flask of at least 500 ml capacity.

b) Thermostat or other apparatus for maintaining the flask at 50 ± 5 °C.

3.2. Reagents

a) Sulphuric acid 75 ± 2 % m/m

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

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

b) Ammonia, dilute solution

Dilute 80 ml of ammonia solution (relative density at 20 °C: 0,880) to 1 litre with water.

4. TEST PROCEDURE

The test procedure, according to the general instructions, shall be the following:
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 ± 5 °C for 1 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

The results shall be calculated as described in the general instructions. The value of "d" is 1,00 except in the case of polypropylene/polyamine two-component fibres, for which "d" = 1,01.

6. PRECISION

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

METHOD No. 8

ACRYLICS, CERTAIN MODACRYLICS OR CERTAIN CHLOROFIBRES AND CERTAIN OTHER FIBRES

(Method using dimethylformamide)

1. FIELD OF APPLICATION

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

1. acrylics (26), certain modacrylics (29), or certain chlorofibres (27), the solubility of such modacrylics or chlorofibres in the reagent shall be checked before carrying out the analysis,

   with
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) and melamine (47) and polypropylene/polyamide two-component fibres (49).

It is equally applicable to acrylics, and certain modacrylics, treated with premetallised 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 at 90-95°C

3.2. Reagent

Dimethylformamide (boiling point 153 ± 1°C) not containing more than 0,1 % water.

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

4. TEST PROCEDURE

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 1 hour. Shake the flask and its contents gently by hand five times during this period.

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

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

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

5. CALCULATION AND EXPRESSION OF RESULTS

The results shall be calculated as described in the general conditions. The value of "d" is 1,00; except in the case of wool, cotton, cupro, modal, polyester, elastomultiester and melamine, for which the value of "d" = 1,01.

6. PRECISION

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

Method No. 9

CERTAIN CHLOROFIBRES AND CERTAIN OTHER FIBRES

(Method using 55,5/44,5 % v/v mixture of carbon disulphide and acetone)

FIELD OF APPLICATION

1. This method is applicable, after removal of non-fibrous matter, to binary fibre mixtures of: certain chlorofibres (27), i.e. certain polyvinyl chloride fibres, whether after-chlorinated (before carrying out the analysis, the solubility of the polyvinyl chloride fibres in the reagent shall be checked.)

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 fibre (44), elastomultiester (45) and melamine (47) and polypropylene/polyamide two-component fibres (49).

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

The test procedure, according to the general instructions, shall be the following:

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 reagents, 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’ = 1,01.

7. PRECISION

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

METHOD No. 10

ACETATE AND CERTAIN OTHER FIBRES

(Method using glacial acetic acid)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to two-component fibre mixtures of:
1. acetate (19)
with
a. 2. certain chlorofibres (27) namely polyvinyl chloride fibres, polipropilenom (37), elastolefinom (46), melamine (47) and polypropylene/polyamide two-component fibres (49)

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

The test procedure, according to the general instructions, shall be the following: 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

The results are calculated as described in the general instructions. The value of "d" is 1,00.

6. PRECISION

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

METHOD No. 11

SILK, POLYAMIDE AND CERTAIN OTHER FIBRES

(Method using 75 % m/m sulphuric acid)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to two-component fibre mixtures of:

1. silk (4) or polyamide or nailon (30)

   with

2. wool (1), animal hair (2 and 3), polypropylen (37), elastolefin (46) and melamine (47) and polypropylene/polyamide two-component fibre (49)

2. PRINCIPLE

The silk, polyamide or nailon fibre is dissolved out from a known dry mass of the mixture, with 75 % m/m sulphuric acid. Wild silk, 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 nailon 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 ± 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 litre with water.

4. TEST PROCEDURE

The test procedure, according to the general conditions, shall be the following: 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 again and stand for 30 minutes.
Shake a last time and filter the contents of the flask through the weighed filter crucible. Wash any remaining fibres from the flask with the 75 % sulphuric acid reagent. Wash the residue on the crucible successively with 50 ml of the dilute sulphuric acid reagent, 50 ml water and 50 ml of the dilute ammonia solution. Each time allow the fibres to remain in contact with the liquid for about 10 minutes before applying suction. Finally rinse with water, leaving the fibres in contact with the water for about 30 minutes.

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

5. CALCULATION AND EXPRESSION OF RESULTS

The results shall be calculated as described in the general results. The value of "d" is 1,00, except for wool; for which "d" = 0,985, for polypropylene/polyamide two-component fibres, for which "d" = 1,005 and for melamine, for which "d"= 1,01.

6. PRECISION

On a homogeneous mixture of textile materials, the confidence limits of results obtained by this method are not greater than ± 1 for a confidence level of 95 %, except for binary mixtures of polyamide with polypropylene/polyamide two-component fibres for which a confidence limits of results are not greater than ±2.

METHOD No. 12

JUTE AND CERTAIN ANIMAL FIBRES

(Method by determining nitrogen content)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to two-component fibre mixtures of:

1. jute (9)

with

2. certain animal fibres.

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

3. 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) Sulphur acid, relative density 1,84 at 20°C. This reagent and other reagents stated hereafter 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 4 hours at a minimum rate of 5 cycles per hour. Allow the solvent to evaporate from the sample in air, and remove the last traces in an oven at 105 ± 3 °C. Then extract the sample in water (50 ml per g of sample) by boiling under reflux for 30 minutes. Filter, return the sample to the flask, and repeat the extraction with an identical volume of water.

Filter, remove excess water from the sample by squeezing, suction, or centrifuging and then allow the sample to become air-dry.

Note:

The toxic effects of toluene and methanol shall be borne in mind and full precautions shall 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 at 20 °C: 1,84). Heat the flask, gently at first, until the whole of the fibre is destroyed, and then heat it more vigorously until the solution becomes clear and almost colourless. Heat it for a further 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 0,02 N 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

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

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:

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

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 ± 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, after removal of non-fibrous matter, to binary fibre mixtures of:

1. polypropylene fibres (37)

with

2. wool (1), animal hair (2 and 3), silk (4), cotton (5), acetate (19), cupro (21), modal (22), triacetate (24), viscose (25), acrylic (26), polyamide or nylon (30), polyester (35), glass fibre (44), elastomultiester (45) and melamine (47).

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 (osim onih navedenih u opštim uputstvima)

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 referred to in indent a) of this method.
c) Heating mantle 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

The test procedure, according to the general conditions, shall be the following: to the specimen contained in the conical flask (point 3.1, indent a. of this method), add 100 ml of xylene (point 3.2 of this method) per gram of specimen. Attach the condenser (point 3.1, indent b.) bring the contents to the boil and maintain at boiling point for 3 minutes.

Immediately decant the hot liquid through the weighed filter crucible (see Note 1). Repeat this treatment twice more, each time using a fresh 50 ml portion of solvent. Wash the residue remaining in the flask successively with 30 ml of boiling xylene (twice), then with 75 ml of light petroleum (1.3.2.1 general instructions from Chapter II 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.

Notes:

1. The filter crucible through which the xylene is to be decanted must be pre-heated.
2. After the treatment with boiling xylene, ensure that the flask containing the residue is cooled sufficiently before the light petroleum is introduced.
3. In order to reduce the fire and toxicity hazards to the operator, a hot extraction apparatus using the appropriate procedures, giving identical results, may be used. (f. e. the apparatus described in Melliand Textilberichte 56 (1975), pp. 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’ = 1,01.

6. PRECISION

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

METHOD No. 14

CERTAIN FIBRES AND CHLOROFIBRES (HOMOPOLYMERS OF VINYL CHLORIDE), ELASTOLEFIN OR MELAMINE

(Concentrated sulphuric acid method)

1. FIELD OF APPLICATION

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

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. chlorofibres (27) based on homopolymers of vinyl chloride, whether after-chlorinated or not, elastolefin (46) and melamine (47 and polypropylene / polyamide two-component fibres (49).

The modacrylics concerned are those which give a limpid solution when immersed in concentrated sulphuric acid (relative density at 20 °C: 1,84).
This method can be used in place of methods No 8 and No 9.

2. PRINCIPLE

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

The residue, consisting of the chlorofibre, the elastolefin or the melamine, 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 constituents 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 at 20 °C: 1,84).
(b) Sulphuric acid, approximately 50 % (m/m) aqueous solution.

Prepare by adding carefully, while cooling, 400 ml of sulphuric acid (relative density at 20 °C: 1,84) 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 at 20 °C: 0,880) to one litre with distilled water

4. TEST PROCEDURE

The test procedure, according to the general instructions, shall be the following: 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 set out in 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

The results shall be calculated as described in the general instructions. The value of "d" is 1,00, except for melamine, and polypropylene / polyamide two-component fibre, for which "d" = 1,01.

6. PRECISION

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

METHOD No.15

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

(Method using cyclohexanone)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to two-component fibre mixtures of:

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

with

2. wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (21), modal (22), viscose (25), polyamide or nylon (30), acrylic (26), glass fibre (44) and melamine (47).

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

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

2. PRINCIPLE

The acetate and triacetate fibres, chlorofibres, certain modacrylics, and certain elastanes are dissolved out from a known dry mass with cyclohexanone at a temperature close to boiling point. The residue is collected, washed, 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 point 4 (see figure: this is a variant of the apparatus described in Melliand Textilberichte 56 (1975.) pp. 643-645).
b) Filter crucible to contain the test specimen.
c) Porous baffle (porosity grade 1).
d) Reflux condenser that can be adapted to the distillation flask.
e) Heating device.

3.2. Reagen

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

The test procedure, according to the general instructions, shall be the following:

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.

5. CALCULATION AND EXPRESSION OF RESULTS

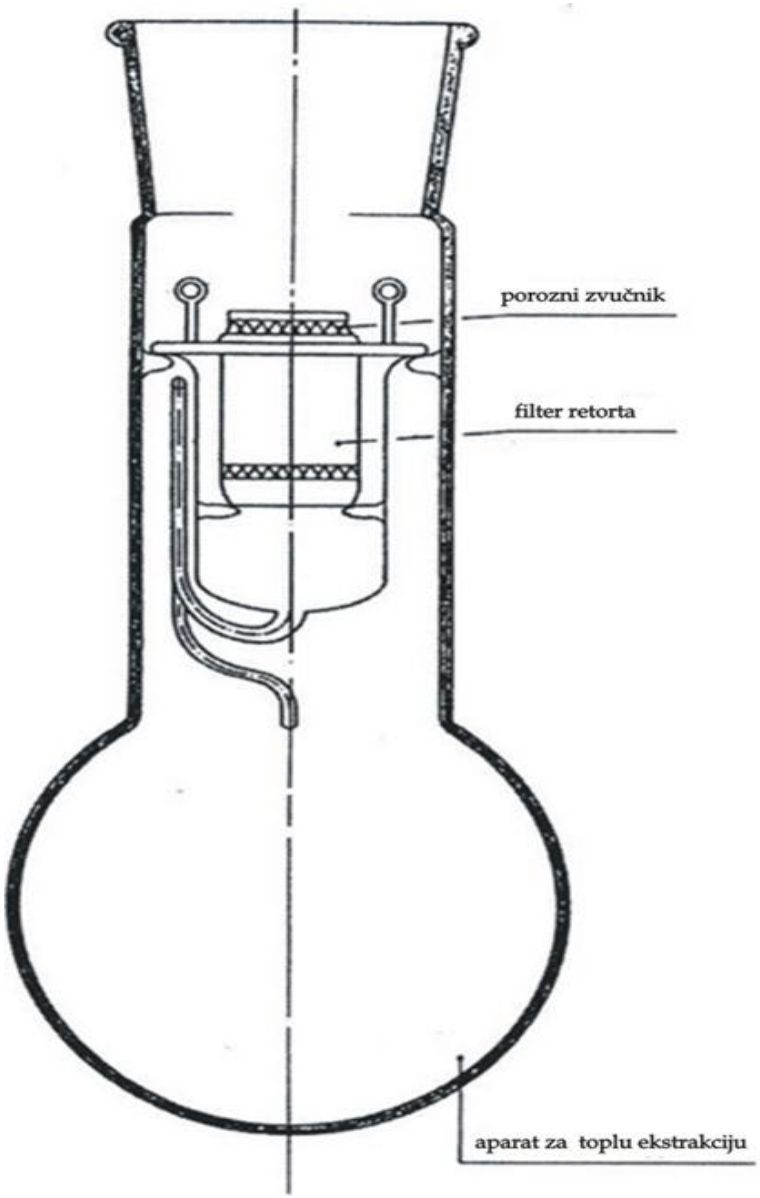
Calculate the results as described in the general instructions. The value of ‘d’ is 1,00 except in the case of silk and melamine for which ‘d’ = 1,01, and acrylic, for which ‘d’ = 0,98.

6. PRECISION

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

The picture related to this method is No. 15, point 3.1.a

(It depicts the following: - porous bafle – filter crucible – hot extraction apparatus)


porozni zvučnik
filter retorta
aparat za toplu ekstrakciju

METHOD No.16

MELAMINE AND CERTAIN OTHER FIBRES

(Method using hot formic acid)

1. FIELD OF APPLICATION

This method is applicable, after removal of non-fibrous matter, to two-component fibre mixtures of:

1. melamine (47)

with

2. cotton (5) and aramid (31).

2. PRINCIPLE

The melamine is dissolved out from a known dry mass of the mixture with hot formic acid (90 % m/m).

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 the second constituents is obtained by difference.

Note:

Keep strictly the recommended temperature range because the solubility of melamine is very much dependent on temperature.

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 ± 2 °C.

3.2. Reagents

a) Formic acid (90 % m/m, relative density at 20 °C: 1,204). Dilute 890 ml of 98 to 100 % m/m formic acid (relative density at 20 °C: 1,220) 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 at 20 °C: 0,880) to 1 litre with water.

4. TEST PROCEDURE

The test procedure, according to the general instructions, shall be the following: 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 and shake the flask to wet out the specimen. Maintain the flask in a shaking water bath at 90 ± 2 °C for 1 hour, shaking it vigorously. Cool the flask to room temperature. Decant the liquid through the 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 reagent. Drain the crucible with suction and wash the residue with formic acid reagent, hot water, dilute ammonia solution, and finally cold water, draining the crucible with suction after each addition. Do not apply suction until each washing liquor has drained under gravity. Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

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

6. PRECISION

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

CHAPTER III

QUANTITATIVE ANALYSIS OF THREE-COMPONENT TEXTILE FIBRE MIXTURES

INTRODUCTION

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

1. Using two different test specimens, a component (a) is dissolved from the first test specimen, and another component (b) from the second test specimen. The insoluble residues of each specimen are weighed and the percentage of each of the two soluble components is calculated from the respective losses in mass. The percentage of the third component (c) is calculated by difference.
2. Using two different test specimens, a component (a) is dissolved from the first test specimen and two components (a and b) from the second test specimen. The insoluble residue of the first test specimen is weighed and the percentage of the component (a) is calculated from the loss in mass. The insoluble residue of the second test specimen is weighed; it corresponds to component (c). The percentage of the third component (b) is calculated by difference.
3. Using two different test specimens, two components (a and b) are dissolved from the first test specimen and two components (b and c) from the second test specimen. The insoluble residues correspond to the two components (c) and (a) respectively. The percentage of the third component (b) is calculated by difference.
4. Using only one test specimen, after removal of one of the components, the insoluble residue formed by the two other fibres is weighed and the percentage of the soluble component is calculated from the loss in mass. One of the two fibres of the residue is dissolved, the insoluble component is weighed and the percentage of the second soluble component is calculated from the loss in mass.

Where a choice is possible, it is advisable to use one of the first three variants.
Where chemical analysis is used, the expert responsible for the analysis must take care to select methods employing solvents which dissolve only the correct fibre(s), leaving the other fibre(s) intact.
By way of example, a table is given in Section V of this Annex which contains a certain number of three-component fibre mixtures, together with methods for analysing two-component fibre mixtures which can, in principle, be used for analysing these three-component 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 chemical methods, the insoluble component of a mixture may be partially dissolved in the reagent used to dissolve the soluble component(s). Wherever possible, reagents have been chosen that have little or no effect on the insoluble fibres. If a loss in mass is known to occur during

the analysis, the result 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 undergraded fibres and different correction factors may be necessary if the fibres have been degraded before or during processing. If the fourth variant, in which a textile fibre is subjected to the successive action of two different solvents, must be used, correction factors must be applied for possible losses in mass undergone by the fibre in the two treatments. At least two determinations shall be made, both in the case of manual separation and in the case of chemical separation.

I. General information on methods for the quantitative chemical analysis of three-component fibre mixtures

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

1.1. FIELD OF APPLICATION

The field of application of each method for analysing two-component fibre mixtures specifies to which fibres the method is applicable (see Chapter II relating to methods for quantitative analysis of certain two-component 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 more of the four variants of the process of selective solution described in the introduction is applied. Except where this presents technical difficulties, it is preferable to dissolve the major fibre component so as to obtain the minor fibre component as final residue.

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 ± 3 °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.

1.4. STANDARD 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 this 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 laboratory test sample in a Soxhlet extractor with light petroleum for 1 hour at a minimum rate of six cycles per hour. Allow the light petroleum to evaporate from the laboratory test sample, which is then extracted by direct treatment consisting in soaking the laboratory test sample in water at room temperature for 1 hour and then soaking it in water at 65 ± 5 °C for a further hour, agitating the liquor from time to time. Use a liquor: laboratory test sample ratio of 100:1. Remove the excess water from the laboratory test sample by squeezing, suction or centrifuging and then allow the laboratory test 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 procedure just described 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 4 hours and not more than 16 hours at 105 ± 3 °C in a ventilated oven with the oven door closed throughout. If the drying period is less than 14 hours, the specimen must be checkweighed to determine whether its mass is constant. The mass may be considered as constant if, after a further drying period of 60 minutes, its variation is less than 0,05 %.

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

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

Dry the filter crucible in a weighing bottle with its cover beside it in the oven. After drying, close the weighing bottle and transfer it quickly to the desiccator.

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

1.7.1.2. Cooling

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

1.7.1.3. Weighing

After cooling, complete the weighing of the weighing bottle within 2 minutes of its removal from the desiccator; weigh to an accuracy of 0,0002 g.

1.7.2. Procedure

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

1.8. CALCULATION AND EXPRESSION

Express the mass of each component as a percentage of the total mass of fibre in the mixture. Calculate the results on the basis of dean dry mass, adjusted by:

a) agreed allowances and
b) the correction factors necessary to take account of loss of non-fibrous matter during pre-treatment and analysis.

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

1.8.1.1. VARIANT 1

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

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

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

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

P_1% is the percentage of the first clean dry component (component in the first specimen dissolved in the first reagent),
P_2% is the percentage of the second clean dry component (component in the second specimen dissolved in the second reagent),
P_3% is the percentage of the third clean dry component (component undissolved in both specimens),
m_1 is the dry mass of the first specimen after pre-treatment,
m_2 is the dry mass of the second specimen after pre-treatment,
r_1 is the dry mass of the residue after removal of the first component from the first specimen in the first reagent,
r_2 is the dry mass of the residue after removal of the second component from the second specimen in the second reagent,

d_1 is the correction factor for loss in mass in the first reagent, of the second component undissolved in the first specimen. The values of d are indicated in Chapter II of this Annex relating to the various methods of analysing binary 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 the other two components (b + v), and two components (a + b) are removed from the second test specimen, leaving as residue the third component v).

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

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

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

P_1% is the percentage of the first clean dry component (component 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 second components from the second specimen in the second reagent,
d_1 is the correction factor for loss in mass in the first reagent, of the second component undissolved in the first specimen,
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 (v), then two components (b + v) are removed from another specimen, leaving as residue the first component a).

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

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

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

P_1% is the percentage of the first clean dry component (component dissolved by the reagent),
P_2% is the percentage of the second clean dry component (component dissolved by the reagent),
P_3% is the percentage of the third clean dry component (component dissolved in the second specimen by the reagent),
m_1 is the dry mass of the first specimen after pre-treatment,
m_2 is the dry mass of the second specimen after pre-treatment,
r_1 is the dry mass of the residue after the removal of the first and second components from the first specimen with the first reagent,
r_2 is the dry mass of the residue after 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 where two components are successively removed from the mixture using the same specimen:

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

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

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

P_1% is the percentage of the first clean dry component (first soluble component),
P_2% is the percentage of the second clean dry component (second soluble component),
P_3% is the percentage of the third clean dry component (insoluble component),
m is the dry mass of the specimen after pre-treatment,
r_1 is the dry mass of the residue after elimination of the first component by the first reagent,
r_2 is the dry mass of the residue after elimination of the first and second component by the first and second reagents,
d1 is the correction factor for loss in mass of the second component in the first reagent,
d_2 is the correction factor for loss in mass of the third component in the first reagent,
d_3 is the correction factor for loss in mass of the third component in the first and second reagents. Wherever possible d 3 should be determined in advance by experimental methods.

1.8.2. Calculation of the percentage of each component with adjustment by agreed allowances and, where 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

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

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

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

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 loss in mass of the third component during pre-treatment

Where a special pre-treatment is used the values b 1 , b 2 and b 3 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 those which they normally contain (either naturally or because of the manufacturing process), in the state (unbleached, bleached) in which they are found in the material to be analysed.
Where no clean separate constituent fibres used in the manufacture of the material to be analysed are available, average values of b 1 , b 2 and b 3 as obtained from tests performed on clean fibres similar to those in the mixture under examination, must be used.
If normal pre-treatment by extraction with light petroleum and water is applied, correction factors b 1 , b 2 and b 3 may generally be ignored, except in the case of unbleached cotton, unbleached flax (or linen) and unbleached hemp where the loss due to pre-treatment is usually accepted as 4 % and in the case of polypropylene as 1 %.
In the case of other fibres, losses due to pre-treatment are usually disregarded in calculations.

1.8.3. Note:

Calculation examples are given in Section IV of this Chapter.

II Method of quantitative analysis by manual separation of three-component fibre mixtures

2.1. FIELD OF APPLICATION

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

2.2 PRINCIPLE

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

2.3. APPARATUS

2.3.1. Weighing bottles 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 ± 3 °C.
2.3.4. Analytical balance accurate to 0,0002 g.
2.3.5. Soxhlet extractor, or other apparatus giving identical results.
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. STANDARD TESTING ATMOSPHERE

See 1.4.

2.6. LABORATORY TEST SAMPLE

See point 1.5. of this Section

2.7. PRE-TREATMENT OF LABORATORY TEST SAMPLES

See point 1.6. of this Section

2.8. PROCEDURE

2.8.1. Analysis of yarn

Take from the pre-treated laboratory test sample a specimen of mass not less than 1 g. For a very fine yarn, the analysis may be made on a minimum length of 30 m, whatever its mass.
Cut the yarn into pieces of a suitable length and separate the fibre types by means of a needle and, if necessary, a twist tester. The fibre types so obtained are placed in pre-weighed weighing bottles and dried at 105 ± 3 °C to constant mass, as described in 1.7.1 and 1.7.2.

2.8.2. Analysis of cloth and knitted cloth

Take from the pre-treated laboratory test sample a specimen of mass not less than 1 g, not including a selvedge with edges carefully trimmed to avoid fraying and running parallel with weft or warp yarns, or in the case of knitted fabrics in the line of the wales and courses. Separate the

different types of fibres and collect them in pre-weighed weighing bottles and proceed as described in 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 in fibre mass during pre-treatment:

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

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

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

P_1% is the percentage of the first clean dry component,
P_2% is the percentage of the second clean dry component,
P_3% is the percentage of the third clean dry component,
m_1 is the clean dry mass of the first component,
m_2 is the clean dry mass of the second component,
m_3 is the clean dry mass of the third component.

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.

III Method of quantitative analysis of third-component fibre mixtures by a combination of manual separation and chemical separation

Wherever possible, manual separation shall be used, taking account of the proportions of components separated 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 two-component fibre mixtures relates to the reproducibility (see Chapter II of this Annex relating to methods for quantitative analysis of certain two-component textile fibre mixtures).
Reproducibility refers to the reliability, i.e. the closeness of agreement between experimental values obtained by operators in different laboratories or at different times using the same method and obtaining individual results on specimens of an identical homogeneous mixture.
Reproducibility is expressed by confidence limits of the results for a confidence level of 95 %.
By this is meant that the difference between two results in a series of analyses made in different laboratories would, given a normal and correct application of the method to an identical and homogeneous mixture, exceed the confidence limit only in five cases out of 100.

To determine the precision of the analysis of a three-component fibre mixture the values indicated in the methods for the analysis of two-component fibre mixtures which have been used to analyse the three-component fibre mixture are applied in the usual way.
Given that in the four variants of the quantitative chemical analysis of three-component 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 E 1 and E 2 denote the precision of the two methods for analysing two-component fibre mixtures, the precision of the results for each component is shown in the following table:

Component fibre	Variant		
	1	2 and 3	4
A	E_1	E_1	E_1
B	E_2	E_1+E_2	E_1+E_2
C	E_1+E_2	E_2	E_1+E_2

If the fourth variant is used, the 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 v, 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. Wherever possible, state the precision of the method for each component, calculated according to the table provided for in point I. Section III.

4. Examples of the calculation of percentages of the components of certain three-component fibre mixtures using some of the variants described in point 1.8.1 Chapter III of this Annex

We shall consider the case of a fibre mixture which gave the following components when qualitatively analysed for raw material composition:

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

VARIANT No.1

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

1. mass of the first dry specimen after pre-treatment is (m1) = 1,6000 g.
2. mass of the dry residue after treatment with alkaline sodium hypochlorite (polyamide + cotton) (r1) = 1,4166g.
3. mass of the second dry specimen after pre-treatment (m2) = 1,8000 g.
4. mass of the dry residue after treatment with formic acid (wool + cotton) r2) = 0,9000 g.

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

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

P_2% (polyamide) = [1,00/1,00 – 1,00 × 0,9000/1,8000 + (1,4166/1,6000) × (1 – 1,00/1,00)] × 100 = 50,00
P_3% (cotton) = 100 – (10,30 + 50,00) = 39,70

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

Wool	10.30%
Polyamide	50.00%
Cotton	39.70%

These percentages must be corrected according to the formulae under 1.8.2, in order to take account of the agreed allowances and the correction factors for any losses in mass after pre-treatment.
As indicated in Annex IX, the agreed allowances are as follows: carded wool 17,00 %, polyamide 6,25 %, cotton 8,50 %, also unbleached cotton shows a loss in mass of 4 %, after pre-treatment with light petroleum and water.

Therefore:

P_1A% (wool) = 10,30 × [1 + (17,00 + 0,0)/100] / [10,30 × (1 + (17,00 + 0,0)/100) + 50,00 × (1 + (6,25 + 0,0)/100) + 39,70 × (1 + (8,50 + 4,0)/100)] × 100 = 10,97

P_2A% (polyamide) = 50,0 × [(1 + (6,25 + 0,0)/100)/109,8385] × 100 = 48,37

P_3A% (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

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

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

1. mass of the dry specimen after pre-treatment (m) = 1,6000 g.
2. mass of the dry residue after treatment with alkaline sodium hypochlorite (viscose + cotton) (r1) = 1,4166 g
3. mass of the dry residue after the second treatment of the residue r1 with zinc chloride/formic acid (cotton) (r2) = 0,6630g.

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

As a result of treatment with formic acid-zinc chloride, the mass of cotton increases by 2%, so that d3 = (1,03 × 1,02) = 1,0506, rounded to 1,05, (d3 being the correction factor for the respective loss or increase in mass of the third component in the first and second reagents).

If the values obtained by chemical analysis and the correction factors are substituted in the formulae given in point 1.8.1.4, the following result is obtained:

P_2% (viscose)=1,00 × (1,4166/1,6000) × 100 – (1,00/1,03) × 43,51 = 46,32%

P_3% (cotton)=1,05 × (0,6630/1,6000) × 100 = 43,51%
P_1% (wool)=100 – (46,32 + 43,51) = 10,17%

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

P_1A% (wool) = 10,17 × [1 + (17,0 + 0,0)/100)]/[10,17 × (1 + (17,00 + 0,0)/100) +46,32 × (1 + (13 + 0,0)/100) + 43,51 × (1 + (8,5 + 4,0)/100)] × 100 = 10,51%

P_2A% (viscose) = 46,32 × [1 + (13 + 0,0)/100] / 113,21 × 100 = 46,24%

P_3A% (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%
	100.0%

IV Table of typical three-component mixtures which may be analysed using a selected method provided for in this Annex applied for the analysis of two-component fibre mixtures (for illustration purposes)

Mixture No.	Component fibres			Variant	Number of method used and reagent for two-component 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 9. (carbon disulphide/acetone 55,5 /44,5 %v/v)
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	Polyamid or nylon	Acryl 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	Acryl or certain other fibres	Polyamide or nylon	2 and/or 4	9. (carbon disulphide/acetone 55,5 /44,5 %v/v) and 8. (dimethylformamide)
12	Certain chlorofibres	Polyamide or nylon	Acryl	1 and/or 4	9.(carbon disulphide/acetone 55,5 /44,5 %v/v) and 4. (formid acid 80%m/m)
13	Polyamide or nylon	Viscose, cupro, modal or cotton	Polyester	4	4.(formic acid, 80%m/m and 7. (sulphur acid 75%m/m)
14	Acetate	Viscose, cupro, modal or cotton	Polyester	4	1.(acetone) and 7. (sulphur acid 75 %m/m)
15	Acrylic	Viscose, cupro, modal or cotton	Polyester	4	8.(dimethylformamid) and 7. (sulphur 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)
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)
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) and 7. (formic 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	Polyester	Melamine	2	8.(dimethylformamide

	modacrylics			and/or 4	) and 14. (concentrated sulphuric acid)

ANNEX VIII

AGREED ALLOWANCES USED TO CALCULATE THE MASS OF FIBRES CONTAINED IN A TEXTILE PRODUCT

(Article 19 paragraph 3 of this Rulebook)

Fibre No	Fibres	Percentages
1-2	Wool and animal hair	
	Čombed fibres	18,25
	Carded fibres	17,00[1]
3	Animal hair	
	Čombed fibres	18,25
	Carded fibres	17,00[1]
	Horsehair	
	Čombed fibres	15,00
	Carded fibres	15,00
4	Silk	11,00
5	Cotton	
	Normal fibres	8,50
	Mercerised fibres	10,50
6	Kapok	10,90
7	Flax (or linen)	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	Polylactide	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	vinylal	5,00
41	Trivinyl	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	Polypropilene/polyamide two-component fibres	1,00

[1] The agreed allowances of 17,00% shall also be applied where it is impossible to ascertain whether the textile product containing wool and/or animal hair is combed or carded.