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## ANNEX VIII

### **Methods for the quantitative analysis of binary and ternary textile fibre mixtures** (referred to in Article 19(1))

#### CHAPTER 1

#### **I. Preparation of laboratory test 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<sup>(1)</sup>.

##### 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<sup>(2)</sup>.

###### 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<sup>(3)</sup>.

###### 2.4. Test specimen

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

##### 3. PRINCIPLE

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

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

##### 4. SAMPLING FROM LOOSE FIBRES

###### 4.1. Unorientated fibres

Obtain the laboratory test sample by selecting tufts at random from the laboratory bulk sample. Mix thoroughly the whole of the laboratory test sample by means of a laboratory carder<sup>(4)</sup>. 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.

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

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

From randomly selected parts of the laboratory bulk sample cut not less than 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<sup>(5)</sup>, 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<sup>2</sup>, where  $G$  is the mass of the cloth in g/m<sup>2</sup>.

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

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material by cutting through all the layers so that each specimen contains an equal length of each layer.

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

6.2. From a laboratory bulk sample consisting of several cuttings

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

## 7. SAMPLING MADE-UP AND FINISHED 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.

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

## 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 section.

Mixtures of fibres during processing and, to a lesser extent, finished textiles may contain non-fibrous matter, such as fats, waxes or dressings, or water-soluble matter, either occurring

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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.

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- (1) In some cases it is necessary to pre-treat the individual test specimen.
- (2) For made-up and finished articles see point 7.
- (3) See point 1.
- (4) The laboratory carder may be replaced by a fibre blender, or the fibres may be mixed by the method of 'tufts and rejects'.
- (5) If the packages can be mounted in a convenient creel a number can be wound simultaneously.

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