

Methods for

Quantitative analysis of fibre mixtures

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- Coopted members
 - Department of Trade and Industry (Laboratory of the Government Chemist)
 - International Wool Secretariat
 - Man-made Fibres Producers' Committee
 - Ministry of Defence
 - Textile Research Council

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Foreword

This revision of this British Standard has been prepared under the direction of the Textiles and Clothing Standards Committee. It is anticipated that a revision of the present UK regulations concerning the determination of the composition of certain textile products will be made to bring them into line with Directive 87/184/EEC amending Directive 72/276/EEC. Until that occurs, the Textile Products (Determination of Composition) Regulations 1976 (SI 202/1976) as amended by SI 767/1982 apply, when establishing and testing fibre percentages of certain textile products. These regulations refer to BS 4407:1975, which will be withdrawn and superseded by this standard on 1 September 1988 when the expected changes to the regulations come into operation.

The principal changes that have been made in this revision in the light of experience gained by national laboratories and the results of inter-laboratory trials carried out under the auspices of the EEC are as follows:

- a) Method 2 has been redrafted to incorporate the use of a stable and easily prepared additional reagent;
- b) certain points in Method 8 have been amended to simplify the test procedures and ensure uniform results;
- c) Method 12 has been deleted because it has proved to be insufficiently accurate;
- d) a new Method 18 has been introduced which utilizes cyclohexanone.

Whenever possible, the fibre components of the textile being analysed should be separated manually: for example, fabric in which the fibre in the warp is different from that in the weft, or yarn consisting of elements which are composed of fibres differing in type. When the different fibres form an intimate mixture, one of the methods based on the selective solution of the individual components (method 13 is an exception) should be used.

Semi-manufactured textiles and, to a lesser extent, finished textiles, may contain fats, waxes or dressings, either occurring naturally or added to facilitate processing. Salts and other water-soluble matter may also be present. Some or all of these substances would be removed during analysis by a chemical method and hence would be included in the soluble fibre component. Non-fibrous matter of this type should therefore be removed before analysis and an appropriate method of pretreatment for this purpose is given.

In addition, textiles may contain resins or other matter added to bond the fibres together or to confer special properties, such as water-repellency or crease-resistance. 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 is also removed before the sample is analysed¹⁾. Dye in dyed fibres is considered to be an integral part of the fibre and is not removed.

Most textile fibres contain water, the amount depending on the type of fibre and on the relative humidity of the surrounding air. Analyses are conducted on the basis of dry mass, and a procedure for determining the dry mass of test specimens and residues is given. The result is therefore obtained on the basis of clean dry fibres.

Provision is also made for recalculating the result after applying:

- a) agreed allowances for moisture and other matter (see Appendix B), and also
- b) allowances, where appropriate, for non-fibrous matter removed in pretreatment.

For many fibres b) can usually be disregarded in calculation.

¹⁾ Recommended procedures for removal of non-fibrous matter are given in ISO/TR 5090.

In some methods the insoluble component or components of a mixture may be partially dissolved in the reagent used to dissolve the soluble component. Where possible, reagents have been chosen that have little or no effect on the insoluble fibres. If loss in mass is known to occur during the analysis, it shall be allowed for and correction factors for this purpose are given. These correction factors have been determined in several laboratories by treating in the appropriate reagent, as specified in the method of analysis, fibres cleaned by the pretreatment. These correction factors apply only to undegraded fibres; different correction factors may be necessary if the fibres have been degraded during processing.

The Directive 73/44/EEC, relating to the quantitative analysis of ternary fibre mixtures allows a choice of methods based on procedures for binary mixtures. The analyst is allowed to choose the variants used for analysis of any ternary fibre mixture, but trials have shown that this is not entirely satisfactory. The UK committee is of the opinion that it is preferable to limit the choice of variants to those listed in Annex 3 of 73/44/EEC, and these are given in Table 2 of this standard. Appendix A details procedures to be adopted for ternary fibre mixtures, and refers to Table 2.

Parts of this standard include references, directly or indirectly, to the handling of asbestos materials. Although the most hazardous types of this fibre (crocidolite) have for some time been very carefully controlled, the potential dangers of asbestos cannot be too strongly emphasized. Any person intending to use any of the asbestos, involving parts of this standard listed below, should follow the approved working practices with respect to asbestos as laid down by the Health and Safety Executive. Details of relevant publications may be obtained from the Health and Safety Executive's Library and Information Services at:

St Hugh's House
Stanley Precinct
Trinity Road
Bootle
Merseyside L20 3QY

The following clauses give references, directly or indirectly, to asbestos sheet:

17.3.2.1 and **25.3.2.1** (cross-reference to BS 509-1)

22.3.2.1 (cross-reference to BS 1994) **29.3.2.1**

Clause **33** and Appendix B give references, directly or indirectly, to asbestos fibre.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 26, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Section 1. General

1 Scope

This British Standard describes methods for the quantitative analysis of textile fibre mixtures after identification of the fibre components of the mixture, including the composition of the individual yarns. Any non-fibrous matter is removed by a suitable pretreatment. If the fibre components do not form an intimate mixture and can be readily separated by hand, the analysis is carried out by the procedure given in section 3.

If part or the whole of the textile consists of an intimate mixture of two fibre types (binary mixture) one of the components is removed using a suitable method²⁾ following the appropriate procedure given in section 4, and the proportion of this component is calculated from the loss in mass. Wherever possible it is preferable to remove the fibre present in greater proportion, thus obtaining the fibre present in the smaller proportion as residue.

If part or the whole of the textile consists of an intimate mixture of three fibre types (ternary mixture) the above process is repeated using a second reagent. Suitable methods for the analysis of specific ternary mixtures are given in Appendix A.

NOTE 1 To obtain a reliable result, at least two determinations should be made, either by manual separation or by chemical separation, if the results of duplicate tests differ by more than 2 %, two repeat determinations should be made.

NOTE 2 The titles of the publications referred to in this standard are listed on the inside back cover.

2 Definitions

For the purposes of this British Standard, the definition given in BS 4815 apply.

²⁾ Method 13 is an exception. It is based on a determination of the content of a constituent element of one of the two components.

Section 2. Information common to the methods given for the quantitative analysis of fibre mixtures

3 Apparatus

3.1 *Glass-filter crucibles*, with sealed-in sintered disc filters of porosity grade P160 complying with BS 1752 and provided with either ground-glass stoppers or watch glass covers.

3.2 *Soxhlet extractor*, complying with BS 2071.

3.3 *Weighing bottles*.

3.4 *Ventilated oven* for drying specimens at 105 ± 3 °C.

3.5 *Desiccator* containing self-indicating silica gel.

3.6 *Analytical balance*, accurate to ± 0.0002 g.

3.7 *Vacuum flask*.

3.8 *Additional apparatus* as specified in the appropriate clauses of this standard.

4 Reagents

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

4.2 *Water* complying with grade 3 of BS 3978.

4.3 *Other reagents* as specified in the appropriate clauses of this standard.

5 Conditioning and testing atmosphere

Dry masses are determined and therefore it is unnecessary to condition the specimen. The analysis is carried out under ordinary room conditions.

6 Sample

Take a sample that is representative of the bulk and sufficient to provide all the specimens, each of at least 1 g, that are required. Follow the sampling procedure as described in BS 4658.

7 Pretreatment of sample

Extract the air-dry sample in a soxhlet apparatus (**3.2**) with light petroleum for 1 h at a minimum rate of 6 cycles/hour. Allow the light petroleum (**4.1**) to evaporate from the sample. Then extract it by immersing the sample in water at room temperature for 1 h and then immersing it in water at 65 ± 5 °C for a further hour, agitating the liquor from time to time. Use a liquor to sample ratio of 100 : 1.

Where non-fibrous matter, e.g. size or special finish, cannot be extracted with light petroleum and water, remove it by substituting for the water treatment 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) note that normal pretreatment with light petroleum and water does not remove all the natural non-fibrous substances. Do not apply additional pretreatment unless the sample does contain finishes insoluble in both light petroleum and water. Include full details of the methods of pretreatment used in the analysis report.

8 Test procedure

8.1 General instructions

8.1.1 *Drying*. Conduct all drying operations for not less than 4 h and not more than 16 h at 105 ± 3 °C in a ventilated oven with the oven door closed throughout. If the drying period is less than 14 h, weigh the specimen or residue to check that its mass has become constant. The mass may be considered to have become constant if, after a further drying of 60 min, its variation is less than 0.05 %. Where apparatus other than a filter crucible is used, conduct drying operations in the oven in such a way as to enable the dry mass of the fibres to be determined without loss of fibre.

8.1.2 *Drying of specimen*. Dry the specimen in a weighing bottle with its stopper beside it. After drying, stopper the weighing bottle before removing it from the oven and transfer it quickly to a desiccator.

8.1.3 *Drying residue*. Dry the residue in the filter crucible with its stopper or cover beside it. After drying, close the crucible and transfer it quickly to a desiccator.

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

8.1.5 *Weighing*. After cooling, complete the weighing of the weighing bottle or crucible within 2 min of its removal from the desiccator.

NOTE Do not handle the crucibles, weighing bottles, specimens or residues with bare hands during the drying, cooling and weighing operations.

8.2 Procedure

Take from the pretreated sample a test specimen weighing at least 1 g. Cut yarn or dissected fabric into lengths of about 10 mm. Dry the specimen in a weighing bottle, cool it in a desiccator and weigh it (see 8.1.2, 8.1.4 and 8.1.5). Transfer the specimen to the glass vessel specified in the appropriate method, reweigh the weighing bottle immediately and obtain the dry mass of the specimen by difference.

Except in the case of method 13, complete the test procedure as specified in the appropriate clause of the standard and examine the residue microscopically to check that the treatment has completely removed the soluble fibre.

9 Calculation and expression of results

9.1 Two-component mixtures

9.1.1 Express the mass of the insoluble component as a percentage of the total mass of fibre in the mixture. Calculate the results on a clean dry mass basis as in 9.1.2 or on clean dry mass with agreed allowances for moisture and other matter and also allowances, where appropriate, for non-fibrous matter removed in the pretreatment as in 9.1.3.

Obtain the percentage of the soluble component by difference.

9.1.2 Determine the percentage of clean dry insoluble component as follows:

$$P_1 = \frac{100rd}{m} \quad (\%)$$

where

- P_1 is the percentage of clean dry insoluble component;
- m is the dry mass of the specimen (g);
- r is the dry mass of the residue (g);
- d is the correction factor for loss in mass of the insoluble component in the reagent.
(Suitable values of d are given in the appropriate clauses of this standard.)

9.1.3 Determine the percentage of clean insoluble component in the mixture with agreed allowances for moisture and other matter and also allowances for non-fibrous matter removed in pretreatment, as follows:

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

where

- P_1A is the percentage of clean insoluble component in the mixture with agreed allowances for moisture and other matter and also, where appropriate, allowances for non-fibrous matter removed in the pretreatment;
- P_1 is the percentage of clean dry insoluble component as calculated in 9.1.2;
- a_1 is the agreed allowance added to the insoluble component for moisture and other matter [see Appendix B (%)];
- a_2 is the agreed allowance added to the soluble component for moisture and other matter [see Appendix B (%)];
- b_1 is the percentage loss of insoluble component caused by the pretreatment (%);
- b_2 is the percentage loss of soluble component caused by the pretreatment (%);

The percentage of the second component (P_2A is equal to $(100 - P_1A)$.

Where a special pretreatment has been used the values b_1 and b_2 should be determined, if possible, by submitting each of the pure fibre constituents to the pretreatment 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 and b_2 , as obtained from tests performed on clean fibres similar to those in the mixture under examination, shall be adopted.

If normal pretreatment by extraction with light petroleum and water is applied, b_1 and b_2 may generally be ignored except in the case of unbleached cotton, unbleached flax and unbleached hemp where the loss due to the pretreatment is usually accepted as 4 %, and in the case of polypropylene as 1 %.

In the case of other fibres, losses in the pretreatment are usually disregarded in calculation.

9.2 Three-component mixtures

9.2.1 General. Express the mass of each component as a percentage of the total mass of the fibres in the mixture. Calculate on a clean dry mass basis as given in 9.2.2, 9.2.3, 9.2.4, 9.2.5 (see Appendix A, variants 1 to 4) or on a clean dry mass with agreed allowances for moisture and other matter and also, where appropriate, allowances for non-fibrous matter removed in the pretreatment, as given in 9.3.

9.2.2 Variant 1. Formulae to be applied where a component of the mixture is removed from one specimen and another component from a second specimen.

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

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

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

where

- 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 pretreatment (g);
- m_2 is the dry mass of the second specimen after pretreatment (g);
- r_1 is the dry mass of the residue after removal of the first component from the first specimen in the first reagent (g);
- r_2 is the dry mass of the residue after removal of the second component from the second specimen in the second reagent (g);
- d_1 is the correction factor for loss in mass in the first reagent, of the second component undissolved in the first specimen^a;

- d_2 is the correction factor for loss in mass in the first reagent, of the third component undissolved in the first specimen^a;
- d_3 is the correction factor for loss in mass in the second reagent, of the first component undissolved in the second specimen^a;
- d_4 is the correction factor for loss in mass in the second reagent, of the third component undissolved in the second specimen^a.

^a The values of d are indicated in the corresponding clauses of this standard relating to the various methods of analysing binary mixtures.

9.2.3 Variant 2. Formulae to be applied where a component (a) is removed from the first test specimen leaving as residue the other two components ($b + c$), and two components ($a + b$) are removed from the second test specimen leaving as residue the third component (c).

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

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

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

where

- P_1 is the percentage of the first clean dry component (component of the first specimen soluble 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 insoluble in both specimens);
- m_1 is the dry mass of the first specimen after pretreatment (g);
- m_2 is the dry mass of the second specimen after pretreatment (g);
- r_1 is the dry mass of the residue after removal of the first component from the first specimen in the first reagent (g);
- 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 (g);
- d_1 is the correction factor for loss in mass in the first reagent, of the second component undissolved in the first specimen^a;
- d_2 is the correction factor for loss in mass in the first reagent, of the third component undissolved in the first specimen^a;

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

^a The values of d are indicated in the corresponding clauses of this standard relating to the various methods of analysing binary mixtures.

9.2.4 Variant 3. Formulae to be applied where two components ($a + b$) are removed from a specimen leaving as residue the third component (c), then two components ($b + c$) are removed from another specimen leaving as residue the first component (a).

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

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

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

where

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 second reagent);

m_1 is the dry mass of the first specimen after pretreatment (g);

m_2 is the dry mass of the second specimen after pretreatment (g);

r_1 is the dry mass of the residue after removal of the first and second components from the first specimen with the first reagent (g);

r_2 is the dry mass of the residue after removal of the second and third components from the second specimen with the second reagent (g);

d_2 is the correction factor for loss in mass with the first reagent of the third component undissolved in the first specimen^a;

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

^a The values of d are indicated in the corresponding clauses of this standard relating to the various methods of analysing binary mixtures.

9.2.5 Variant 4. Formulae to be applied where two components are successively removed from the mixture using the same specimen.

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

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

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

where

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 pretreatment (g);

r_1 is the dry mass of the residue after elimination of the first component by the first reagent (g);

r_2 is the dry mass of the residue after elimination of the first and second components by the first and second reagents (g);

d_1 is the correction factor for loss in mass of the second component in the first reagent^a

d_2 is the correction factor for loss in mass of the third component in the first reagent^a

d_3 is the correction factor for loss in mass of the third component in the first and second reagents^a.

^a The values of d are indicated in the corresponding clauses of this standard relating to the various methods of analysing binary mixtures.

9.3 Calculation of the percentage of each component with addition of agreed allowance for moisture and other matter and also allowances, where appropriate, for non-fibrous matter removed in the pretreatment

Given

$$X = 1 + \frac{a_1 + b_1}{100}, \quad Y = 1 + \frac{a_2 + b_2}{100},$$

$$Z = 1 + \frac{a_3 + b_3}{100}$$