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Methods for

Quantitative analysis of fibre mixtures

ICS 59.060.01

Committees responsible for this British Standard

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British Carpet Manufacturers' Association Ltd.
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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Contents

Section 1. General

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

Section 3. Method of quantitative analysis by manual separation

Section 4. Methods of quantitative analysis by chemical procedures

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 These regulations refer to BS 4407:1975, which will be withdrawn and superseded by this standard on 1 September 1988 when the expected changes to the regulations come into operation.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 analysed[1]. 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.

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[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.

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 method[2] 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 determination 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 stand are listed on the inside back cover.

2 Definitions

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

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[2] Method 13 is an exception. It is based on a determination of the content of a constituent element of one of the two component

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:

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

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:

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

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

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

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

then

$$P_1A = \frac{P_1X}{P_1X + P_2Y + P_3Z} \times 100\ (\%)$$

$$P_2A = \frac{P_2Y}{P_1X + P_2Y + P_3Z} \times 100\ (\%)$$

$$P_3A = \frac{P_3Z}{P_1X + P_2Y + P_3Z} \times 100\ (\%)$$

where

P_1A	is the percentage of the first clean dry component, including agreed allowance for moisture and loss in mass during pretreatment;
P_2A	is the percentage of the second clean dry component, including agreed allowance for moisture and loss in mass during pretreatment;
P_3A	is the percentage of the third clean dry component, including agreed allowance for moisture and loss in mass during pretreatment;
P_1	is the percentage of the first clean dry component obtained by one of the formulae given in **9.2**;
P_2	is the percentage of the second clean dry component obtained by one of the formulae given in **9.2**;
P_3	is the percentage of the third clean dry component obtained by one of the formulae given in **9.2**;
a_1	is the agreed allowance for moisture of the first component (see Appendix B) (%);
a_2	is the agreed allowance for moisture of the second component (see Appendix B) (%);
a_3	is the agreed allowance for moisture of the third component (see Appendix B) (%);
b_1	is the percentage of loss in mass of the first component during pretreatment;
b_2	is the percentage of loss in mass of the second component during pretreatment;
b_3	is the percentage of loss in mass of the third component during pretreatment;

X, Y, Z are correction factors.

Where a special pretreatment is used the values b_1, b_2 and b_3 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 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, should be used.

If normal pretreatment by extraction with light petroleum and water is applied b_1, b_2 and b_3 may generally be ignored, except in the case of unbleached cotton, unbleached flax and unbleached hemp where the loss due to 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 calculations.

10 Report

10.1 State that the analysis was conducted in accordance with the appropriate method(s) of this standard.

10.2 State whether the result relates to the overall composition of the assembly or to an individual component of the assembly.

10.3 Give details of any special treatment for the removal of size or finish given in substitution for or addition to the specified pretreatment.

10.4 State the number of individual determinations made. Give the individual results and the arithmetic mean, each to an accuracy of 0.1 %.

10.5 State whether the result is based on:

a) clean dry mass;

b) clean dry mass with agreed allowances for moisture and other matter and also, where appropriate, for non-fibrous matter removed in the pretreatment, and state the values of the allowances used.

10.6 Whenever possible, state the precision of the method for each component for ternary mixtures. Calculate the precision according to Table 1 (see clause **16**).

Section 3. Method of quantitative analysis by manual separation

11 General

This method is applicable to textile fibre mixtures of all types, provided that they do not contain an intimate mixture of two or more different fibres, which can be separated by hand.

12 Principle

After identification of the constituents of the fibre mixture including the composition of the individual yarns, the non-fibrous material is removed by suitable pretreatment and then the constituents are separated by hand, dried and weighed in order to calculate the proportion of each fibre in the mixture.

13 Apparatus and reagents

13.1 Apparatus

The following apparatus, together with apparatus specified in clause **3**, is required.

13.1.1 *Needle.*

13.1.2 *Twist tester*, or similar apparatus.

13.2 Reagents

No additional reagents are required.

14 Test procedure

14.1 Analysis of yarn

Select from the pretreated sample a specimen weighing 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 weighing bottles and dried at a temperature of 105 ± 3 °C until a constant mass is obtained, as described in **8.1**. Remove the fibre, re-weigh the weighing bottle immediately and determine the mass of fibre by difference.

14.2 Analysis of fabric

Select from the pretreated sample, well away from all selvedges, a specimen weighing not less than 1 g, with edges carefully trimmed and running parallel with weft or warp yarns, or in the case of knitted fabrics in the line of wales and courses. In the case of knitted fabrics, ensure that all cut loops are removed before selection of the specimen. Separate the different fibre types, collect them in weighing bottles and proceed as described in **14.1**.

15 Calculation and expression of results

15.1 General

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

15.2 Method based on clean dry mass

15.2.1 ***Binary mixtures***

$$P_1 = \frac{100m_1}{m_1 + m_2} = \frac{100}{1 + m_2/m_1} \quad (\%)$$

$$P_2 = 100 - P_1$$

where

P_1 is the percentage of the first clean, dry component;

P_2 is the percentage of the second clean, dry component;

m_1 is the clean, dry mass of the first component (g);

m_2 is the clean, dry mass of the second component (g).

15.2.2 ***Ternary mixtures***

$$P_1 = \frac{100\,m_1}{m_1 + m_2 + m_3} = \frac{100}{1 + \dfrac{m_2 + m_3}{m_1}} \quad (\%)$$

$$P_2 = \frac{100\,m_2}{m_1 + m_2 + m_3} = \frac{100}{1 + \dfrac{m_1 + m_3}{m_2}} \quad (\%)$$

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

where

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 (g);

m_2 is the clean, dry mass of the second component (g);

m_3 is the clean, dry mass of the third component (g).

15.3 For calculation of the percentage of each component with adjustment for agreed allowance for moisture and other matter and also, where appropriate, for non-fibrous matter removed in the pretreatment, see **9.1.3** in the case of binary mixtures and **9.2** and **9.3** in the case of ternary mixtures.

Section 4. Methods of quantitative analysis by chemical procedures

16 Precision of the methods

The precision indicated in individual methods relates to the reproducibility. 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.

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

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

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

Given that in the four variants (see Appendix A) of the quantitative chemical analysis of ternary 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 E_1 and E_2 denote the precision of the two methods for analysing binary mixtures, the precision of the results for each component is shown in Table 1.

Table 1 — Precision of methods for ternary mixtures

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 (*c*), which would be difficult to evaluate.

17 Method 1. Binary mixtures of acetate and certain other fibres (method using acetone)

17.1 General

This method is applicable, after removal of non-fibrous matter, to binary mixtures of acetate with cotton, wool, animal hair, silk, protein, polyamide, polyester, acrylic, flax, true hemp, jute, abaca, alfa, coir, broom, ramie, sisal, modal, cupro and viscose. It is not applicable to mixtures containing modacrylic. In no circumstances is the method applicable to acetate which has been deacetylated on the surface.

17.2 Principle

The acetate is dissolved from a known dry mass of the mixture using acetone. 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 acetate is found by difference.

17.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

17.3.1 ***Apparatus***

17.3.1.1 *Conical flask*, capacity 250 mL, with ground glass stopper.

17.3.2 ***Reagent***

17.3.2.1 *Acetone* complying with BS 509-1.

17.4 Test procedure

Follow the procedure described in **8.2** and proceed as follows.

To the specimen contained in the stoppered conical flask (**17.3.1.1**) add 100 mL of acetone (**17.3.2.1**) per gram of specimen, insert the stopper, shake the flask, and stand it for 30 min at room temperature shaking it occasionally. Decant the liquid through the weighed filter crucible retaining the fibres in the flask. Repeat the treatment twice more (making three extractions in all) but for periods of 15 min only, so that the total time of treatment in acetone is 1 h. Wash the residue into the filter crucible with acetone, and drain with suction. Refill the crucible with acetone and allow it to drain under gravity. Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them (see **8.1.3**, **8.1.4** and **8.1.5**).

17.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of *d* is 1.00.

17.6 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 %.

18 Method 2. Binary mixtures of certain protein fibres (wool, animal hair, silk or protein) and certain other fibres (method using hypochlorite)

18.1 General

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

a) wool, animal hair, silk and protein based on casein; with

b) cotton, cupro, elastane, modal, viscose, acrylic, chlorofibres, polyamide, polyester or nylon, polypropylene and glass.

If different protein fibres are present, the method gives the total of their amounts, but not their individual quantities.

18.2 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.

18.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

18.3.1 ***Apparatus***

18.3.1.1 *Erlenmeyer flask*, capacity 250 mL, with ventilated, loose fitting stopper.

18.3.1.2 *Means of controlling temperature* at 20 ± 2 °C.

18.3.2 ***Reagents***

18.3.2.1 *Hypochlorite reagent* consisting of either

a) *lithium hypochlorite solution*. Dissolve either 100 g of lithium hypochlorite containing 35 % active chlorine or 115 g containing 30 % active chlorine in approximately 700 mL of water. Add 5 g of sodium hydroxide (NaOH) dissolved in approximately 200 mL of water and dilute to 1 L with water. Prepare this solution freshly each day; or

b) *sodium hypochlorite solution*, containing 35 ± 2 g/L of active chlorine and 5 ± 0.5 g of sodium hydroxide, prepared as follows.

Standardize iodometrically a supply of sodium hypochlorite solution available commercially to establish its active chlorine content. Calculate the dilution necessary to convert it to an active chlorine content of 3.5 %, and therefore the volume of concentrated hypochlorite solution needed to produce 1 L of the required reagent. To this amount of the concentrated solution, add the whole of a solution containing 5 ± 0.5 g of sodium hydroxide in 200 mL of water and dilute to 1 L with water. Mix well, and prepare freshly each day

NOTE Lithium hypochlorite is recommended in cases involving a small number of analyses or for analyses conducted at fairly lengthy intervals, because the percentage of active chlorine in solid lithium hypochlorite, unlike that in sodium hypochlorite, is virtually constant.

18.3.2.2 *Acetic acid solution*. Dilute 5 mL of glacial acetic acid (approximately 99.7 % (*m*/*m*), ρ approximately 1.06 g/m mL) to 1 L with water.

18.4 Procedure

Follow the procedure described in **8.2** and proceed as follows.

Mix approximately 1 g of the specimen with approximately 100 mL of the hypochlorite solution (**18.3.2.1**) in the flask (**18.3.1.1**) and agitate thoroughly to wet out the specimen.

Maintain the flask for 40 min at 20 ± 2 °C and agitate continuously.

NOTE 1 To ensure fibres completely dissolve in 40 min, a wetting agent may be required.

NOTE 2 Since the dissolution of the wool proceeds exothermically, the heat of reaction of this method has to be distributed and removed. Otherwise, considerable errors may be caused by the incipient dissolution of the non-soluble fibres.

After 40 min, filter the flask contents through a weighed glass-filter crucible (**3.1**) 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 (**18.3.2.2**) 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 and residue, and cool and weigh them (see **8.1.3**, **8.1.4** and **8.1.5**).

18.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of d is 1.00, except for cotton, viscose and modal, for which d = 1.01, and unbleached cotton, for which d = 1.03.

18.6 Precision

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

19 Method 3. Binary mixtures of viscose, cupro or certain types of modal and cotton (method using formic acid and zinc chloride)

19.1 General

This method is applicable, after removal of non-fibrous matter, to binary mixtures of viscose, cupro or certain types of modal with cotton. If modal is found to be present, a preliminary test should be carried out to see whether it is soluble in the reagent, under the conditions of test. For certain types of modal fibre which are difficult to dissolve, the procedure, modified as described in **19.4**, may be used.

This method is not applicable to mixtures in which the cotton has suffered extensive chemical degradation, nor when the viscose, cupro or modal is rendered incompletely soluble by the presence of certain dyes or finishes that cannot be removed completely by pretreatment.

19.2 Principle

The viscose, cupro or modal is dissolved from a known dry mass of the mixture, using a reagent containing 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 is found by difference.

19.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

19.3.1 ***Apparatus***

19.3.1.1 *Conical flask*, capacity 250 mL, with ground glass stopper.

19.3.1.2 *Means of controlling the temperature of the flask and contents* at 40 ± 2 °C (or 70 ± 2 °C if the high temperature method is used).

19.3.2 ***Reagents***

19.3.2.1 *Formic acid/zinc chloride solution*

Either prepare a solution containing 20 g of anhydrous zinc chloride and 68 g (56 mL) of formic acid (96 % to 100 % *m*/*m*) made up to 100 g with water or alternatively prepare a solution of 20 parts by mass of zinc chloride to 76 parts by mass of 90 % *m*/*m* formic acid (63 mL) plus 4 parts of water.

19.3.2.2 *Dilute ammonia solution*. Dilute 20 mL of concentrated ammonia solution (approximately 35 % (*m*/*m*), ρ approximately 0.88 g/mL) to 1 L with water.

19.4 Test procedure

Follow the procedure described in **8.2** and proceed as follows.

Place 100 mL of the formic acid and zinc chloride solution per gram of specimen in the conical flask, heat it to a temperature of 40 °C and add the specimen. Insert the stopper and shake the flask vigorously. Keep the flask and its contents at a constant temperature of 40 ± 2 °C for 2.5 h, shaking the flask at hourly intervals. Filter the contents of the flask through the weighed filter crucible and, using more reagent, transfer to the crucible any fibres remaining in the flask. Wash the crucible and residue with a further 20 mL of the reagent at 40 °C and then wash the crucible and residue thoroughly with water at a temperature of 40 °C. For certain modal fibres for which the method is not suitable, use the same method but maintain the temperature of the reagent at 70 ± 2 °C for a period of 20 ± 1 min. Rinse with a further 10 mL of reagent preheated to 70 °C and then wash the crucible and residue thoroughly with water also at 70 °C. Rinse the fibrous residue with approximately 100 mL of cold ammonia solution ensuring that the residue remains wholly immersed in solution for 10 min.

NOTE To ensure that the fibrous residue is immersed for 10 min in the ammonia solution, it is possible, for example, to add to the filtering apparatus an adaptor fitted with a tap by which the flow of ammonia solution can be regulated.

Drain the crucible with suction and then rinse with cold water. 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 (see **8.1.3**, **8.1.4** and **8.1.5**).

19.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of *d* is 1.02.

If the high temperature procedure is used, the value of *d* is 1.03.

19.6 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 %.

20 Method 4. Binary mixtures of polyamide or nylon and certain other fibres (method using formic acid 80 % *m*/*m*)

20.1 General

This method is applicable, after removal of non-fibrous matter, to binary mixtures of polyamide or nylon with cotton, animal hair, cupro, modal, viscose, chlorofibre, polypropylene, acrylic, polyester or glass fibre. It is applicable also to mixtures with wool or animal hair, but when the animal fibre content exceeds 25 %, the method described in clause **18** shall be used.

NOTE This method applies to polyamide 6 or polyamide 6.6 and not to other polyamides.

20.2 Principle

The polyamide is dissolved from a known dry mass of the mixture using aqueous formic acid. 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 polyamide or nylon is found by difference.

20.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

20.3.1 ***Apparatus***

20.3.1.1 *Conical flask*, capacity minimum 200 mL, with ground glass stopper.

20.3.2 ***Reagents***

20.3.2.1 *Formic acid solution* (approximately 80 % (*m*/*m*), ρ approximately 1.19 g/mL). Dilute 880 mL of formic acid (approximately 90 % (*m*/*m*), ρ approximately 1.20 g/mL) to 1 L with water.

The concentration is not critical within the range 77 % to 83 % *m*/*m* formic acid.

Alternatively, dilute 780 mL of 98 % to 100 % *m*/*m* formic acid relative density at (20 °C : 1.220) to 1 L with water.

20.3.2.2 *Dilute ammonia solution*. Dilute 80 mL of concentrated ammonia solution (approximately 35 % (*m*/*m*), ρ approximately 0.88 g/mL) to 1 L with water.

20.4 Test procedure

Follow the procedure described in **8.2** and proceed as follows.

To the specimen contained in the glass-stoppered conical flask (**20.3.1.1**), add 100 mL of the formic acid per gram of specimen, insert the stopper, shake the flask to wet out the specimen and stand it for 15 min, shaking it at intervals. Filter the contents of the flask through a weighed filter crucible and transfer any residual fibres to the crucible by washing out the flask with more 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 (see **8.1.3**, **8.1.4** and **8.1.5**).

20.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of d is 1.00.

20.6 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 %.

21 Method 5. Binary mixtures of acetate and triacetate (method using benzyl alcohol)

21.1 General

This method is applicable after the removal of non-fibrous matter, to binary mixtures of acetate with triacetate.

21.2 Principle

The acetate is dissolved from a known dry mass of the mixture using benzyl alcohol. 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 acetate is found by difference.

21.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

21.3.1 ***Apparatus***

21.3.1.1 *Conical flask*, capacity 250 mL, with ground glass stopper.

21.3.1.2 *Mechanical shaker*.

21.3.1.3 *Means of controlling the temperature of the flask and contents* at 52 ± 2 °C.

21.3.2 ***Reagents***

21.3.2.1 *Benzyl alcohol.*

21.3.2.2 *Ethanol* complying with BS 507.

21.4 Test procedure

Follow the procedure described in **8.2** and proceed as follows.

To the specimen contained in the conical flask (**21.3.1.1**), 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 a temperature of 52 ± 2 °C, and shake for 20 min at this temperature.

Decant the liquid through the weighed filter crucible retaining the fibres in the flask. Add a further quantity of benzyl alcohol to the flask and shake as before for 20 min. Decant the liquid through the crucible retaining the fibres in the flask. Repeat the cycle of operations a third time. Finally, pour the liquid and the residue into the crucible; wash any fibres remaining in the flask into the crucible with more benzyl alcohol at a temperature of 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 retaining the fibres in the flask. Repeat this rinsing operation two or three times. Transfer the residue to the crucible and drain thoroughly. Dry the crucible and residue and cool and weigh them (see **8.1.3**, **8.1.4** and **8.1.5**).

21.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of d is 1.00.

21.6 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 %.

22 Method 6. Binary mixtures of triacetate and certain other fibres (method using dichloromethane)

22.1 General

This method is applicable, after removal of non-fibrous matter, to binary mixtures of triacetate with wool, animal hair, silk, cotton, cupro, modal, viscose, polyamide, or nylon, polyester, protein, acrylic or glass fibre. It is not applicable to triacetate which has received a finish leading to partial hydrolysis.

22.2 Principle

The triacetate is dissolved 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 triacetate is found by difference.

22.3 Apparatus and reagents

The following apparatus and reagent, together with those specified in clauses **3** and **4**, are required.

22.3.1 ***Apparatus***

22.3.1.1 *Conical flask*, capacity 250 mL, with ground glass stopper.

22.3.2 ***Reagent***

22.3.2.1 *Dichloromethane (methylene chloride)*, complying with BS 1994.

22.4 Test procedure

Follow the procedure described in **8.2** and proceed as follows.

To the specimen contained in the glass-stoppered conical flask, add 100 mL of dichloromethane per gram of specimen, insert the stopper, shake the flask to wet out the specimen, and allow the flask to stand for 30 min, shaking it occasionally. Decant the liquid through the weighed filter crucible retaining the fibres in the flask. Add 60 mL of dichloromethane to the residue in the flask, replace the stopper, shake it by hand, and filter the contents of the flask through the filter crucible. Transfer any residual fibres to the crucible by washing out the flask with more dichloromethane. Drain the crucible with suction, refill the crucible with dichloromethane, and allow it to drain under gravity. Finally, drain the crucible with suction.

If the residue is glass fibre, polyamide, polyester, protein, or acrylic, dry the crucible and residue and cool and weigh them (see **8.1.3**, **8.1.4** and **8.1.5**).

If the residue is other fibres, treat it in the following way to remove solvent that cannot be removed by oven-drying.

Transfer the residue by means of forceps to a beaker containing about 100 mL of boiling water. Continue boiling for 5 min, and then decant through the same filter crucible retaining the fibres in the beaker. Add about 100 mL of boiling water to the residue in the beaker, boil it for 5 min, and again decant retaining the fibres in the beaker. Give a third boiling treatment and transfer the residue to the filter crucible. Allow the water to drain under gravity and then drain with suction. Wash any residual fibres from the beaker into the crucible with water. Finally, drain the crucible with suction, dry the crucible and residue and cool and weigh them (see **8.1.3**, **8.1.4** and **8.1.5**).

22.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of *d* is 1.00 except for polyester fibre, for which *d* is 1.01.

22.6 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 %.

23 Method 7. Binary mixtures of certain cellulose fibres and polyester (method using sulphuric acid 75 % *m*/*m*)

23.1 General

This method is applicable, after removal of non-fibrous matter, to binary mixtures of polyester and flax, hemp, ramie, cupro, modal, viscose or cotton.

23.2 Principle

The cellulose fibre is dissolved from a known dry mass of the mixture using sulphuric acid, approximately 75 % (*m*/*m*).

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 cellulose fibre is found by difference.

23.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

23.3.1 ***Apparatus***

23.3.1.1 *Conical flask*, capacity 500 mL, with ground glass stopper.

23.3.1.1 *Means of controlling the temperature of the flask and contents* at 50 ± 5 °C.

23.3.2 ***Reagents***

23.3.2.1 *Sulphuric acid solution* (approximately 75 % *m*/*m*, ρ approximately 1.67 g/mL). Prepare by adding, carefully and slowly, while cooling, 700 mL of sulphuric acid (approximately 98 % *m*/*m*), ρ approximately 1.84 g/mL) to 350 mL of water. After the solution has cooled to 20 °C, dilute it to 1 L with water.

23.3.2.2 *Dilute ammonia solution*. Dilute 80 mL concentrated ammonia solution (approximately 35 % (*m*/*m*), ρ approximately 0.88 g/mL) to 1 L with water.

23.4 Test procedure

Follow the procedure described in **8.2** and proceed as follows.

To the specimen contained in the glass-stoppered conical flask, add 200 mL of sulphuric acid (**23.3.2.1**) per gram of specimen, insert the stopper, and shake the flask carefully to wet out the specimen. Maintain the flask at a temperature of 50 ± 5 °C for 1 h, shaking the flask and contents gently at intervals of about 10 min. Filter the contents of the flask through a weighed filter crucible by means of suction. Transfer any residual fibres to the crucible by washing out the flask with more sulphuric acid reagent. Drain the crucible with suction, and wash the residue on the filter once by filling the crucible with a fresh portion of sulphuric acid reagent. Do not apply suction until this has drained under gravity or stood for 1 min. 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 crucible with suction, dry the crucible and residue, and cool and weigh them (see **8.1.3**, **8.1.4** and **8.1.5**).

23.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of *d* is 1.00.

23.6 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 %.

24 Method 8. Binary mixtures of acrylics, certain modacrylics or certain chlorofibres and certain other fibres (method using dimethylformamide)

24.1 General

This method is applicable, after removal of non-fibrous matter, to binary mixtures of acrylics, certain modacrylics or certain chlorofibres with wool, animal hair, silk, cotton, cupro, modal, viscose, polyamide or nylon and polyester. It is equally applicable to acrylics and certain modacrylics, dyed with pre-metallized dyes, but not to those dyed with afterchrome dyes.

24.2 Principle

The acrylic, modacrylic or chlorofibre is dissolved from a known dry mass of the mixture using dimethylformamide. 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 acrylic, modacrylic or chlorofibre is found by difference.

24.3 Apparatus and reagent

The following apparatus and reagent, together with those specified in clauses **3** and **4**, are required.

24.3.1 ***Apparatus***

24.3.1.1 *Conical flask*, capacity 200 mL (minimum), with ground glass stopper.

24.3.1.2 *Water bath*, at boiling point.

24.3.2 ***Reagent***

24.3.2.1 *Dimethylformamide* (boiling point 152 °C to 154 °C) containing not more than 0,1 % water.

NOTE This reagent is toxic. The use of a fume hood is essential and protective gloves should be worn.

24.4 Test procedure

Follow the procedure described in **8.2** and proceed as follows.

To the specimen contained in the glass-stoppered conical flask, add 80 mL of dimethylformamide, preheated to 90 °C to 95 °C, per gram of specimen, insert the stopper, shake the flask to wet out the specimen and maintain the flask and contents for 1 h at 90 °C to 95 °C. Shake the flask and contents gently by hand five times during this period. Decant the liquid through a weighed filter crucible, retaining the fibres in the flask. Add a further 60 mL of dimethylformamide to the flask and heat for 30 min at 90 °C to 95 °C, 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 flask with dimethylformamide (**24.3.2.1**). Drain the crucible with suction. Wash the residue with 1 L of hot water at 70 °C to 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 apply slight suction.

Finally, dry the crucible and residue, cool and weigh them (see **8.1.3**, **8.1.4** and **8.1.5**).

24.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of d is 1.00, except in the following cases:

wool	$d = 1.01$
cotton	$d = 1.01$
cupro	$d = 1.01$
modal	$d = 1.01$
polyester	$d = 1.01$

24.6 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 %.

25 Method 9. Binary mixtures of certain chlorofibres and certain other fibres (method using carbon disulphide/acetone 55.5/44.5)

25.1 General

This method is applicable, after removal of non-fibrous matter, to binary mixtures of chlorofibres, namely certain polyvinyl chloride fibres, whether after-chlorinated or not, with wool, animal hair, silk, cotton, polyamide or nylon, polyester, acrylic, glass fibre, cupro, modal and viscose. When the wool, animal hair or silk content of a mixture exceeds 25 % the method described in clause **18** shall be used. When the polyamide or nylon content of a mixture exceeds 25 %, the method described in clause **20** shall be used.

25.2 Principle

The chlorofibre is dissolved from a known dry mass of the mixture using an azeotropic mixture of carbon disulphide and acetone. 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 chlorofibre is found by difference.

25.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

25.3.1 ***Apparatus***

25.3.1.1 *Conical flask*, capacity 250 mL, with ground glass stopper.

25.3.1.2 *Mechanical shaker*.

25.3.1.3 *Small watch glass*.

25.3.2 ***Reagents***

25.3.2.1 *Azeotropic mixture of carbon disulphide* complying with BS 662-1 and acetone complying with BS 509-1. Mix 555 mL of carbon disulphide with 445 mL acetone.

NOTE The toxic effects and flammability of carbon disulphide should be borne in mind and full precautions should be taken when it is used.

25.3.2.2 *Ethanol* complying with BS 507.

25.4 Test procedure

Follow the procedure described in **8.2** and proceed as follows.

To the specimen contained in the glass-stoppered conical flask, add 100 mL of carbon disulphide/acetone per gram of specimen. Insert the stopper and shake the flask on a mechanical shaker for 20 min, loosening the stopper once or twice at the beginning of the process to release any excess pressure. Decant the supernatant liquid through the weighed filter crucible, retaining the fibres in the flask. Repeat the treatment with a further 100 mL of fresh reagent. Continue with this cycle of operations until no deposit is left on a watch glass when a drop of the extraction liquor is evaporated.

Transfer the residue from the flask to the filter crucible using more reagent, drain with suction, and wash the crucible and residue three times with 20 mL of ethanol and then three times with water. 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 (see **8.1.3**, **8.1.4** and **8.1.5**).

NOTE With certain mixtures having a high polyvinyl chloride content there may have been substantial shrinkage of the specimen during the initial drying procedure, as a result of which the dissolution of polyvinyl chloride by the solvent may be retarded. This does not, however, affect the ultimate dissolution of the polyvinyl chloride by the solvent.

25.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of *d* is 1.00.

25.6 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 %.

26 Method 10. Binary mixtures of acetate and certain chlorofibres (method using glacial acetic acid)

26.1 General

This method is applicable, after removal of non-fibrous matter, to binary mixtures of acetate with certain chlorofibres namely polyvinylchloride whether after-chlorinated or not.

26.2 Principle

The acetate is dissolved from a known dry mass of the mixture using glacial acetic acid. 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 acetate is found by difference.

26.3 Apparatus and reagent

The following apparatus and reagent, together with those specified in clauses **3** and **4**, are required.

26.3.1 ***Apparatus***

26.3.1.1 *Conical flask*, capacity 250 mL, with ground glass stopper.

26.3.1.2 *Mechanical shaker*.

26.3.2 ***Reagent***

26.3.2.1 *Glacial acetic acid* distilling at 117 °C to 119 °C.

26.4 Test procedure

Follow the procedure described in **8.2** and proceed as follows.

To the specimen contained in the glass-stoppered conical flask add 100 mL of glacial acetic acid per gram of specimen. Insert the stopper and shake for 20 min on a mechanical shaker. Decant the supernatant liquid through the weighed filter crucible retaining the fibres in the flask. Repeat the treatment twice more using 100 mL of fresh reagent each time, making three extractions in all. Transfer the residue to the filter crucible, drain with suction, and wash the crucible and residue with 50 mL of acetic acid, and then three times with water. 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 (see **8.1.3**, **8.1.4** and **8.1.5**).

26.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of *d* is 1.00.

26.6 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 %.

27 Method 11. Binary mixtures of silk and wool or hair (method using sulphuric acid 75 % *m*/*m*)

27.1 General

This method is applicable, after removal of non-fibrous matter, to binary mixtures of silk with wool or animal hair. It is not applicable to mixtures containing wild silk, e.g. tussah.

27.2 Principle

The silk fibre is dissolved from a known dry mass of the mixture, using sulphuric acid 75 % (*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 silk is found by difference.

27.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

27.3.1 ***Apparatus***

27.3.1.1 *Conical flask*, capacity 250 mL, with ground glass stopper.

27.3.2 ***Reagents***

27.3.2.1 *Sulphuric acid solution* (approximately 75 % *m*/*m*, ρ approximately 1.67). Prepare by adding, carefully and slowly, while cooling, 700 mL of sulphuric acid (approximately 98 % *m*/*m*, ρ approximately 1.84 g/mL) to 350 mL of water. After the solution has cooled to 20 °C, dilute to 1 L with water.

27.3.2.2 *Sulphuric acid, dilute solution.* Add 100 mL sulphuric acid (approximately 98 % *m*/*m*, ρ approximately 1.84 g/mL) slowly to 1 900 mL of water.

27.3.2.3 *Dilute ammonia solution.* Dilute 200 mL concentrated ammonia solution (approximately 35 % *m*/*m*, ρ approximately 0.88 g/mL) to 1 L with water.

27.4 Test procedure

Follow the procedure described in **8.2** and proceed as follows.

To the specimen contained in the glass-stopper conical flask, add 100 mL of sulphuric acid 75 % *m*/*m* (**27.3.2.1**) per gram of specimen and insert the stopper. Shake the flask vigorously and stand it for 30 min at room temperature. Shake the flask again and stand it for 30 min. Shake it a last time and filter the contents of the flask through the weighed filter crucible. Wash any remaining fibres from the flask with more 75 % sulphuric acid reagent. Wash the residue on the crucible successively with 50 mL of the dilute sulphuric acid reagent (**27.3.2.2**) 50 mL water and 50 mL of the dilute ammonia solution (**27.3.2.3**). Each time ensure the fibres remain in contact with the liquid for at least 10 min before applying suction.

Finally rinse with water, leaving the fibres in contact with the water for about 30 min. Drain the crucible with suction, dry the crucible and residue, and cool and weigh them (see **8.1.3**, **8.1.4** and **8.1.5**).

27.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of d is 0.985 for wool.

27.6 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 %.

28 Method 12. Deleted

This method has been deleted because it has proved to be insufficiently accurate.

29 Method 13. Binary mixtures of jute and certain animal fibres (method of determining the nitrogen content)

29.1 General

Because this method differs in principle from the general method based on selective solubility set out in section 2, it is given in a form that is more detailed than other methods.

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

29.2 Principle

The nitrogen content of the mixture is determined, and from this and the known or assumed nitrogen contents of the two components, the proportion of each component is calculated.

29.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

29.3.1 ***Apparatus***

29.3.1.1 *Kjeldahl digestion flask*, capacity 200 mL to 300 mL.

29.3.1.2 *Kjeldahl distillation apparatus with steam injection.*

29.3.1.3 *Titration apparatus* allowing precision of 0.05 mL.

29.3.1.4 *One-mark volumetric flask*, capacity 200 mL, complying with BS 1792.

29.3.1.5 *Conical flask*, capacity 100 mL.

29.3.2 ***Reagents***

NOTE All reagents should be of a recognized analytical reagent quality.

29.3.2.1 *Toluene* complying with BS 135, BS 458, BS 805.

29.3.2.2 *Methanol* complying with BS 506-1.

29.3.2.3 *Sulphuric acid*, approximately 98 % *m*/*m*, ρ approximately 1.84 g/mL.

29.3.2.4 *Potassium sulphate*.

NOTE This reagent should be nitrogen free.

29.3.2.5 *Selenium dioxide*.

NOTE This reagent should be nitrogen free.

29.3.2.6 *Sodium hydroxide solution* 400 g/L. Dissolve 400 g of sodium hydroxide in 400 mL to 500 mL of water and dilute to 1 L with water.

29.3.2.7 *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.

29.3.2.8 *Boric acid solution*. Dissolve 20 g boric acid in 1 L of water.

29.3.2.9 *Sulphuric acid*, $c(H_2SO_4) = 0.01$ mol/L.

29.4 Test sample

Take a sample that is representative of the bulk and sufficient to provide all the specimens, each of about 1 g, that are required. Treat the sample as described in **29.5**.

29.5 Pretreatment of sample

Extract the air-dry sample in a Soxhlet apparatus with a mixture of 1 volume of toluene and 3 volumes of methanol for 4 h at a minimum rate of 5 cycles/h. Allow the solvent to evaporate from the sample in air, and remove the last traces in an oven at a temperature of 105 ± 3 °C. Then extract the sample in water (50 mL/g of sample) by boiling under reflux for 30 min. Filter, return the sample to the flask, and repeat the extraction with a similar 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 should be borne in mind, and full precautions should be taken when they are used.

29.6 Test procedure

29.6.1 *General instructions*. Observe the general instructions given in **8.1**.

29.6.2 *Detailed procedure*. Take from the pretreated sample a test specimen of mass about 1 g. Dry the specimen in a weighing bottle, cool it in a desiccator, and weigh it. Transfer the specimen to a dry Kjeldahl digestion flask, reweigh the weighing bottle immediately, and obtain the dry mass of the specimen by difference.

To the specimen in the digestion flask add, in the following order, 2.5 g potassium sulphate (**29.3.2.4**), 0.1 g to 0.2 g selenium dioxide (**29.3.2.5**) and 10 mL sulphuric acid (**29.3.2.3**). 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 min. Allow the flask to cool, dilute the contents carefully with 10 mL to 20 mL water, cool, transfer the contents quantitatively to a 200 mL flask (**29.3.1.4**) and make up to volume with water to form the digest solution.

Place about 20 mL of boric acid solution in a conical flask (**29.3.1.5**) 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 (**29.3.2.6**) to the funnel, lift the stopper slightly, and allow the sodium hydroxide solution to run slowly into the flask. In the digest solution and sodium hydroxide solution remain as two separate layers, mix them by gentle agitation.

Heat the distillation flask gently and pass into it steam from the generator. Collect about 20 mL of distillate, lower the receiver so that the tip of the delivery tube is about 20 mm above the surface of the liquid, and distill for 1 min more. Rinse the tip of the delivery tube with water, catching the washings in the receiver. Remove the receiver and replace it with a second receiver containing about 10 mL of boric acid solution, and collect about 10 mL of distillate. Titrate the two distillates separately with sulphuric acid (**29.3.2.9**) using 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.

29.7 Calculation and expression of results

29.7.1 Calculate the percentage nitrogen in the dry specimen as follows:

$$A = \frac{56(V - b)M}{W} \quad (\%)$$

where

A is the percentage nitrogen in the clean dry specimen;

V is the total titre of standard sulphuric acid for the determination (mL);

b is the total titre of standard sulphuric acid for the blank determination (mL);

M is the molarity of standard sulphuric acid (mol/L);

W is the dry mass of specimen (g).

29.7.2 Using the values of 0.22 % for the nitrogen content of jute and 16.2 % for the nitrogen content of animal fibre, both values being expressed on the dry mass of the fibre, calculate the composition of the mixture as follows:

$$P_A = \frac{A - 0.22}{16.2 - 0.22} \times 100 \qquad (\%)$$

where

P_A is the percentage animal fibre in the clean dry specimen.

30 Method 14. Binary mixtures of flax and viscose (method using sulphuric acid)

30.1 General

This method is applicable, after removal of non-fibrous matter, to binary mixtures of grey or bleached flax with viscose (including many modal fibres). If modal is found to be present, a preliminary test should be made to see whether it is soluble in the reagent under the conditions of test. The method is not applicable to mixtures in which the flax has suffered extensive chemical degradation, nor when the viscose or modal has been rendered incompletely soluble by the presence of certain finishes, or dyes which cannot be removed completely by the pretreatment.

30.2 Principle

The viscose or modal is dissolved from a known dry mass of the mixture using sulphuric acid, after preliminary treatment with dilute hydrochloric acid. The residue is collected, washed, dried and weighed. Its mass, corrected for loss, both in the preliminary treatment and in the separation treatment, is expressed as a percentage of the dry mass of the mixture. The percentage of viscose or modal is found by the difference.

30.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

30.3.1 ***Apparatus***

30.3.1.1 *Flask*, capacity 500 mL.

30.3.1.2 *Glass-stoppered flask*, capacity 250 mL.

30.3.1.3 *Mechanical shaker*.

30.3.2 ***Reagents***

30.3.2.1 *Dilute hydrochloric acid solution*. Dilute 20 mL concentrated hydrochloric acid (approximately 37 % (m/m), ρ approximately 1.18 g/mL) with 500 mL of water.

30.3.2.2 *Dilute ammonia solution*. Dilute 80 mL of concentrated ammonia solution (approximately 35 % (m/m), ρ approximately 0.88 g/mL) to 1 L with water.

30.3.2.3 *Sulphuric acid*, 60 % m/m. Prepare by adding carefully, while cooling, 903 mL sulphuric acid (ρ approximately 1.84 at 20 °C) to 1 L of water.

After cooling to 20 °C, the mixture should have a relative density of between 1.495 and 1.500. If necessary, adjust it to that value by adding acid or water as appropriate.

30.4 Additional pretreatment of sample

Follow the procedure described in clause **7** and proceed as follows.

Take from the pretreated sample a test sample of mass approximately 2 g. Dry the test sample in a weighing bottle, cool it in a desiccator and weigh it. Transfer the test sample to a 500 mL flask (**30.3.1.1**), reweigh the weighing bottle immediately, and obtain the dry mass of the test sample by difference. Add 200 mL of the dilute hydrochloric acid solution to the test sample in the flask and reflux for 10 min. Filter the contents of the flask through a weighed filter crucible and transfer any residual fibres to the crucible by washing out the flask with hot water. Wash the residue successively with hot water, dilute ammonia solution 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 and residue, and cool and weigh them (see **8.1.3**, **8.1.4** and **8.1.5**).

30.5 Test procedure

Follow the procedure described in **8.2** but cut the specimen into lengths of about 5 mm. Proceed as follows.

To the specimen contained in the glass-stoppered conical flask, add 100 mL of the sulphuric acid reagent per gram of specimen. Insert the stopper and shake the flask vigorously on a mechanical shaker for 30 ± 1 min. Filter the contents of the flask through a weighed filter crucible and transfer any residual fibres to the crucible by washing out the flask with a small amount of the sulphuric acid reagent. Drain the crucible with suction and wash the residue on the filter successively three times with 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 (see **8.1.3**, **8.1.4** and **8.1.5**).

If the results of duplicate tests differ by more than 2 % make two repeat determinations.

30.6 Calculation and expression of results

Calculate the percentage of clean dry flax as follows:

$$p = \frac{100dbM_2}{aM_1}$$

where

P is the percentage of clean dry flax;

a is the mass of the oven-dry sample before pretreatment with hydrochloric acid (g);

b is the mass of the oven-dry sample after pretreatment with hydrochloric acid (g);

M_1 is the oven-dry mass of the test specimen after pretreatment with hydrochloric acid (g);

M_2 is the oven-dry mass of the flax residue (g);

d is the correction factor for loss in mass of the flax component in the reagent.

The value of d for bleached flax is 1.16 and for grey flax is 1.22.

31 Method 15. Binary mixtures of polypropylene and certain other fibres (method using xylene)

31.1 General

This method is applicable, after removal of non-fibrous matter, to binary mixtures of polypropylene fibres with wool, silk, cotton, viscose, cupro, modal, acetate, triacetate, polyamide, polyester, acrylic, and glass fibres.

31.2 Principle

The polypropylene fibre is dissolved 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.

31.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

31.3.1 ***Apparatus***

31.3.1.1 *Conical flask*, capacity 200 mL, with ground glass stopper.

31.3.1.2 *Reflux condenser* (suitable for liquids of high boiling point) fitting the conical flask.

31.3.2 ***Reagent***

31.3.2.1 *Xylene*, distilling between 137 °C and 142 °C.

NOTE The harmful effects of this reagent should be borne in mind, and full precautions should be taken when it is used.

31.4 Test procedure

Follow the procedure described in **8.2** and proceed as follows.

To the specimen contained in the conical flask add 100 mL of the xylene per gram of specimen. Attach the condenser and boil the contents for 3 min. Decant the hot liquid through the weighed filter crucible. Repeat this treatment twice more, each time using a fresh 50 mL of solvent.

Wash the residue remaining in the flask successively with 30 mL of boiling xylene (twice) then with 75 mL of the light petroleum (twice). After the second wash with light petroleum, filter the residue through the filter crucible and allow it to drain. Dry the crucible and residue, cool and weigh them (see **8.1.3**, **8.1.4** and **8.1.5**).

Hot extraction apparatus using the appropriate procedure, giving identical results, may be used[3].

NOTE 1 Preheat the filter crucible through which the xylene is to be filtered.

NOTE 2 After the treatment with boiling xylene, ensure that the flask containing the residue is cooled sufficiently before the light petroleum is introduced.

31.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of d is 1.00.

31.6 Precision

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

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[3] See, for example, the apparatus described in *Melliand Textilberichte*, **56** (1975) 643–645.

32 Method 16. Binary mixtures of chlorofibres (homopolymers of vinyl chloride) and certain other fibres (method using concentrated sulphuric acid)

32.1 General

This method is applicable, after removal of non-fibrous material, to binary mixtures of chlorofibres based on homopolymers of vinyl chloride (after-chlorinated or not) with cotton, viscose, cupro, modal, acetate, triacetate, polyamide, polyester and certain acrylic and certain modacrylic fibres. (The modacrylics concerned are those which give a limpid solution when immersed in concentrated sulphuric acid ρ approximately 1.84.)

It can be used particularly in place of methods 8 and 9 in all cases where a preliminary test shows that the chlorofibres do not dissolve completely either in dimethylformamide or in the azeotropic mixture of carbon disulphide and acetone.

32.2 Principle

The constituent other than the chlorofibre is dissolved from a known dry mass of the mixture with concentrated sulphuric acid. The residue, consisting of the chlorofibre, is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of the second constituent is obtained by difference.

32.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

32.3.1 ***Apparatus***

32.3.1.1 *Conical flask*, capacity 200 mL, with ground glass stopper.

32.3.1.2 *Glass rod* with flattened end.

32.3.2 ***Reagents***

32.3.2.1 *Sulphuric acid*, concentrated, approximately 98 % m/m, ρ approximately 1.84 g/mL.

NOTE Concentrated sulphuric acid is highly corrosive and appropriate safety precautions should be taken when it is used.

32.3.2.2 *Sulphuric acid*, 50 % (m/m) aqueous solution.

32.3.2.3 *Ammonia*, dilute solution.

Dilute 60 mL of concentrated ammonia solution (approximately 35 % m/m, ρ approximately 0.88 g/mL) to 1 L with water.

32.4 Test procedure

Follow the procedure described in **8.2** and proceed as follows.

To the specimen contained in the conical flask, add 100 mL of the concentrated sulphuric acid (**32.3.2.1**) per gram of specimen.

Allow the contents of the flask to remain at room temperature for 10 min, 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 the concentrated sulphuric acid 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 to the flask in order to remove any fibres adhering to the wall. Drain the filter crucible with suction. After emptying or changing the filter-flask, wash the residue in the crucible successively with the 50 % sulphuric acid solution (**32.3.2.2**), water, the ammonia solution and finally with water, draining the crucible with suction after each addition until the water drained from the crucible is neutral to litmus. (Do not apply suction during the washing operation, but only after the liquid has drained through the crucible.)

Dry the crucible and residue, cool and weigh them (see **8.1.3**, **8.1.4** and **8.1.5**).

32.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of d is 1.00.

32.6 Precision

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

33 Method 17. Binary mixtures of asbestos and certain fibres (method based on heating to 450 °C)

NOTE This method differs in principle from the general method based on selective solubility set out in clause **1**.

33.1 General

This method is applicable to binary mixtures of cotton, viscose, acetate, triacetate, modal, cupro, polyester, or polyamide (excluding aramid) and chrysotile or crocidolite asbestos.

"WARNING Asbestos health hazard

NOTE Full precautions should be taken to avoid inhalation of asbestos at all stages of this method. For further details see the foreword.

33.2 Principle

The constituent other than the asbestos is removed from a known dry mass of the mixture by heating at 450 ± 10 °C for 1 h. The residue consisting of asbestos is weighed; its corrected mass is expressed as a percentage of the dry mass of the mixture. The percentage of the second constituent is calculated by difference.

33.3 Apparatus

The following apparatus, together with that specified in clause **3**, is required.

33.3.1 *Electric furnace*, with automatic temperature control at 450 ± 10 °C.

33.3.2 *Silica or metal crucible.*

33.4 Sampling

Take a laboratory test sample that is representative of the laboratory bulk sample and sufficient to provide all the specimens, each about 5 g, that are required.

NOTE Pretreatment of the laboratory test sample described in clause **7** is not applicable to the analysis of this mixture.

33.5 Test procedure

33.5.1 *General instructions.* Observe the general instructions given in **8.1**.

33.5.2 *Detailed procedure.* Take from the laboratory test sample a test specimen weighing about 5 g. Determine the dry mass of the test specimen accurately in a weighing bottle, transfer it to a crucible of known mass and heat it in the electric furnace with automatic temperature control at 450 ± 10 °C for 1 h.

Cool the crucible and its contents to room temperature in a desiccator. Determine the mass of the crucible and residue within 2 min of its removal from the desiccator.

33.6 Calculation and expression of results

Calculate the results as described in **9.1**. The value of d is 1.02.

34 Method 18. Binary mixtures of chlorofibres, certain modacrylics, certain elastanes, acetates, triacetates and certain other fibres (method using cyclohexanone)

34.1 General

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

a) acetate, triacetate, chlorofibre, certain modacrylics and certain elastanes with

b) wool, animal hair, silk, cotton, cupro, modal, viscose, polyamide, acrylic and glass fibre.

Where modacrylics or elastanes are present a preliminary test should 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 9 or method 16.

34.2 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.

34.3 Apparatus and reagents

The following apparatus and reagents, together with those specified in clauses **3** and **4**, are required.

34.3.1 ***Apparatus***

34.3.1.1 *Hot extraction apparatus* (see Figure 1).

NOTE This is a variant of the apparatus used in *Melliand Textilberichte*, **56** (1975) 643–645.

34.3.1.2 *Filter crucible.*

34.3.1.3 *Porous baffle.*

34.3.1.4 *Reflux condenser.*

34.3.1.5 *Source of heat.*

34.3.2 *Reagents*

34.3.2.1 *Cyclohexanone*, boiling point 156 °C.

NOTE Cyclohexanone is flammable and toxic. Suitable precautions should be taken when it is used.

34.3.2.2 *Ethanol*, 50 % *V*/*V*.

34.4 Procedure

Follow the procedure described in **8.2** and proceed as follows.

Pour 100 mL of cyclohexanone (**34.3.2.1**) per gram of specimen into the distillation flask (**34.3.1.1**). Insert the extraction container in which the filter crucible, containing the specimen and the porous baffle (**34.3.1.3**), slightly inclined, have previously been placed. Insert the reflux condenser (**34.3.1.4**). Bring to the boil and continue extraction for 1 h at a minimum rate of 12 cycles/h. 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 ethanol (**34.3.2.2**) heated to approximately 60 °C and subsequently with 1 L of water at 60 °C.

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

Finally dry the crucible and residue, cool and weigh them (see **8.1.3**, **8.1.4** and **8.1.5**).

34.5 Calculation and expression of results

Calculate the results as described in **9.1**. The value of *d* is 1.00 with the following exceptions:

silk	$d = 1.01$
acrylic	$d = 0.98$.

34.6 Precision

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

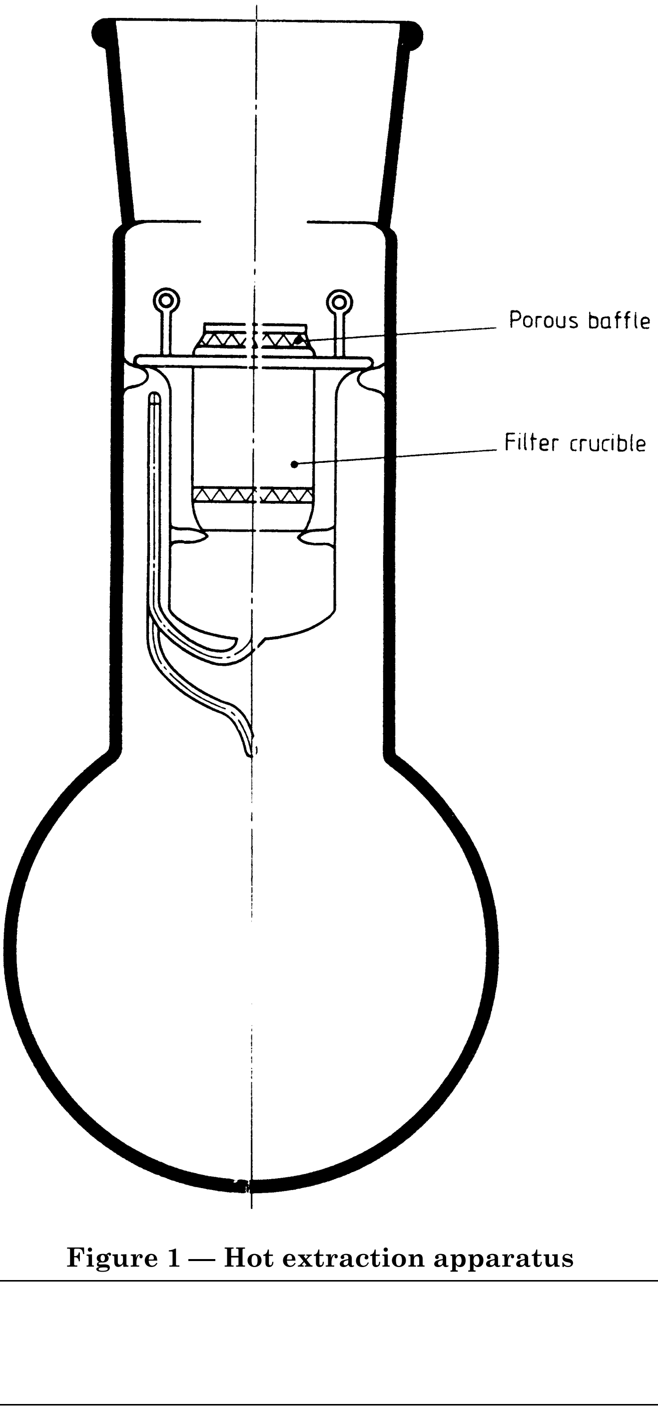

Figure 1 — Hot extraction apparatus

Appendix A Procedures to be adopted for ternary fibre mixtures

NOTE The Directive 73/44/EEC relating to the quantitative analysis of ternary fibre mixtures allows the use of a choice of methods based on the procedures for binary fibre mixtures.

The Directive 73/44/EEC allows the analyst to choose the variants used for analysis of any ternary fibre mixture, but trials have shown that this is not entirely satisfactory. Although there is insufficient information on the validity of the individual variants for specific ternary fibre mixtures it is preferable to limit the choice of variants to those listed in annex **3** of 73/44/EEC and given in Table 2 of this standard.

Four variants may be adopted for analysis of ternary fibre mixtures.

Variant 1. Component (*a*) is removed from the first test specimen and component (*b*) is removed from the second test specimen, component (*c*) is found by difference.

Variant 2. Component (*a*) is removed from the first test specimen, leaving as a 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*).

Variant 3. Components (*a* + *b*) are removed from the first test specimen, leaving as a residue the third component (*c*), then two components (*b* + *c*) are removed from the second test specimen, leaving as a residue the first component (*a*).

Variant 4. Two components are successively removed from a single test specimen.

Table 2 — Procedures to be used for quantitative chemical analysis of ternary fibre mixtures

Mixture No.	Component fibres			Variant[a]	Number of method for binary mixtures and reagent
	1st component (*a*)	2nd component (*b*)	3rd component (*c*)		
1	wool or hair	viscose, cupro or certain types of modal	cotton	1 and/or 4	2 (alkaline hypochlorite) and 3 (zinc chloride/formic acid)
2	wool or hair	polyamide 6 or 6.6	cotton, viscose, cupro or modal	1 and/or 4	2 (alkaline hypochlorite) and 4 (formic acid)
3	wool, hair or silk	certain chlorofibres	viscose, cupro, modal or cotton	1 and/or 4	2 (alkaline hypochlorite) and 9 (carbon disulphide/acetone)
4	wool or hair	polyamide 6 or 6.6	polyester, polypropylene, acrylic or glass fibre	1 and/or 4	2 (alkaline hypochlorite) and 4 (formic acid)
5	wool, hair or silk	certain chlorofibres	polyester, acrylic, polyamide or glass fibre	1 and/or 4	2 (alkaline hypochlorite) and 4 (carbon disulphide/acetone)
6	silk	wool or hair	polyester	2	11 (sulphuric acid) and 2 (alkaline hypochlorite)
7	polyamide 6 or 6.6	acrylic	cotton, viscose, cupro or modal	1 and/or 4	4 (formic acid) and 8 (dimethylformamide)
8	certain chlorofibres	polyamide 6 or 6.6	cotton, viscose, cupro or modal	1 and/or 4	8 (dimethylformamide) and 4 (formic acid) 9 (carbon disulphide/acetone) and 4 (formic acid)
9	acrylic	polyamide 6 or 6.6	polyester	1 and/or 4	8 (dimethylformamide) and 4 (formic acid)
10	acetate	polyamide 6 or 6.6	viscose, cotton, cupro or modal	4	1 (acetone) and 4 (formic acid)
11	certain chlorofibres	acrylic	polyamide	2 and/or 4	9 (carbon disulphide/acetone) and 8 (dimethylformamide)
12	certain chlorofibres	polyamide 6 or 6.6	acrylic	1 and/or 4	9 (carbon disulphide/acetone) and 4 (formic acid)

[a] When variant 4 is used, first remove the first component using the first reagent.

Table 2 — Procedures to be used for quantitative chemical analysis of ternary fibre mixtures

Mixture No.	Component fibres			Variant[a]	Number of method for binary mixtures and reagent
	1st component (*a*)	2nd component (*b*)	3rd component (*c*)		
13	polyamide 6 or 6.6	viscose, cupro, modal or cotton	polyester	4	4 (formic acid) and 7 (sulphuric acid)
14	acetate	viscose, cupro, modal or cotton	polyester	4	1 (acetone) and 7 (sulphuric acid)
15	acrylic	viscose, cupro, modal or cotton	polyester	4	8 (dimethylformamide) and 7 (sulphuric acid)
16	acetate	wool, hair or silk	cotton, viscose, cupro, modal, polyamide, polyester or acrylic	4	1 (acetone) and 2 (alkaline hypochlorite)
17	triacetate	wool, hair or silk	cotton, viscose, cupro, modal, polyamide, polyester or acrylic	4	6 (dichloromethane) and 2 (alkaline hypochlorite)
18	acrylic	wool, hair or silk	polyester	1 and/or 4	8 (dimethylformamide) and 2 (alkaline hypochlorite)
19	acrylic	silk	wool or hair	4	8 (dimethylformamide) and 11 (sulphuric acid)
20	acrylic	wool, hair or silk	cotton, viscose, cupro or modal	1 and/or 4	8 (dimethylformamide) and 2 (alkaline hypochlorite)
21	wool, hair or silk	cotton, viscose, modal or cupro	polyester	4	2 (alkaline hypochlorite) and 7 (sulphuric acid)
22	viscose, cupro or certain types of modal	cotton	polyester	2 and/or 4	3 (zinc chloride/formic acid) and 7 (sulphuric acid)
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) 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)
28	triacetate	silk	wool or hair	4	6 (dichloromethane) and 11 (sulphuric acid)

[a] When variant 4 is used, first remove the first component using the first reagent.

Table 2 — Procedures to be used for quantitative chemical analysis of ternary fibre mixtures

Mixture No.	Component fibres			Variant[a]	Number of method for binary mixtures and reagent
	1st component (*a*)	2nd component (*b*)	3rd component (*c*)		
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 6 or 6.6	cotton, viscose, cupro or modal	4	6 (dichloromethane) and 4 (formic acid)
32	triacetate	cotton, viscose, cupro or modal	polyester	4	6 (dichloromethane) and 7 (sulphuric acid)
33	acetate	polyamide 6 or 6.6	polyester or acrylic	4	1 (acetone) and 4 (formic acid)
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) or 9 (carbon disulphide/acetone) and 7 (sulphuric acid)

[a] When variant 4 is used, first remove the first component using the first reagent.

Appendix B Agreed allowances for moisture and other matter to be used in connection with the analysis of fibre mixtures as given in Annex II of the EEC Directive on textile descriptions, 71/307/EEC, as amended by Directive 83/623/EEC

Fibres	Percentages
Wool and animal hair:	
combed fibres	18.25
carded fibres	17.00
Animal hair:	
combed fibres	18.25
carded fibres	17.00
Horsehair:	
combed fibres	16.00
carded fibres	15.00
Silk	11.00
Cotton:	
normal fibres	8.50
mercerized fibres	10.50
Kapok	10.90
Flax	12.00
Hemp	12.00
Jute	17.00
Abaca	14.00
Alfa	14.00
Coir	13.00
Broom	14.00
Ramie (bleached fibre)	8.50
Sisal	14.00
Sunn	12.00
Henequen	14.00
Maguey	14.00
Acetate	9.00
Alginate	20.00
Cupro	13.00
Modal	13.00
Protein	17.00
Triacetate	7.00
Viscose	13.00
Acrylic	2.00
Chlorofibre	2.00
Fluorofibre	0.00
Modacrylic	2.00
Polyamide or nylon:	
discontinuous fibre	6.25
filament	5.75
Polyester:	
discontinuous fibre	1.50
filament	1.50
Polyethylene	1.50
Polypropylene	2.00
Polycarbamide	2.00
Polyurethane:	
discontinuous fibre	3.50
filament	3.00
Vinylal	5.00
Trivinyl	3.00
Elastodiene	1.00
Elastane	1.50
Glass fibre:	
filament with a diameter of over 5 µm	2.00
filament with a diameter of 5 µm or less	3.00
Metal fibre	2.00
Metallized fibre	2.00
Asbestos	2.00
Paper yarn	13.75

Publications referred to

BS 135, *Specifications for benzene, xylenes and toluenes.*

BS 458, *Specifications for benzene, xylenes and toluenes.*

BS 805, *Specifications for benzene, xylenes and toluenes.*

BS 506, *Methanol for industrial use.*

BS 506-1, *Specification for methanol.*

BS 507, *Specification for ethanol for industrial use.*

BS 509, *Acetone for industrial use.*

BS 509-1, *Specification for acetone.*

BS 662, *Carbon disulphide for industrial use.*

BS 662-1, *Specification.*

BS 1752, *Specification for laboratory sintered or fritted filters including porosity grading.*

BS 1792, *Specification for one-mark volumetric flasks.*

BS 1994, *Specification for dichloromethane (methylene chloride).*

BS 2071, *Specification for Soxhlet extractors.*

BS 3978, *Specification for water for laboratory use.*

BS 4658, *Methods of test for textiles — preparation of laboratory test samples and test specimens for chemical testing.*

BS 4815, *Glossary of generic names for man-made fibres.*

ISO/TR 5090, *Textiles — Methods for the removal of non-fibrous matter prior to quantitative analysis of fibres mixtures*4).

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4) Mentioned in the foreword only.

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