

Regulation (EU) No 1007/2011 of the European Parliament and of the Council of 27 September 2011 on textile fibre names and related labelling and marking of the fibre composition of textile products and repealing Council Directive 73/44/EEC and Directives 96/73/EC and 2008/121/EC of the European Parliament and of the Council (Text with EEA relevance)

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ANNEX VIII

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

CHAPTER 2

METHODS FOR QUANTITATIVE ANALYSIS OF CERTAIN BINARY TEXTILE FIBRE MIXTURES

I. General information common to the methods given for the quantitative chemical analysis of textile fibre mixtures

I.1. FIELD OF APPLICATION

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

I.2. PRINCIPLE

After the identification of the components of a mixture, the non-fibrous material is removed by suitable pre-treatment and then one of the components, usually by selective solution⁽¹⁾. The insoluble residue is weighed and the proportion of soluble component calculated from the loss in mass. Except where this presents technical difficulties, it is preferable to dissolve the fibre present in the greater proportion, thus obtaining the fibre present in the smaller proportion as residue.

I.3. MATERIALS AND EQUIPMENT

I.3.1. Apparatus

I.3.1.1. Filter crucibles and weighing bottles large enough to contain such crucibles, or any other apparatus giving identical results.

I.3.1.2. Vacuum flask.

I.3.1.3. Desiccator containing self-indicating silica gel.

I.3.1.4. Ventilated oven for drying specimens at 105 ± 3 °C.

I.3.1.5. Analytical balance, accurate to 0,0002 g.

I.3.1.6. Soxhlet extractor or other apparatus giving identical results.

I.3.2. Reagents.

I.3.2.1. Light petroleum, redistilled, boiling range 40 to 60 °C.

I.3.2.2. Other reagents are specified in the appropriate section of each method.

I.3.2.3. Distilled or deionised water.

I.3.2.4. Acetone.

I.3.2.5. Orthophosphoric acid.

I.3.2.6. Urea.

I.3.2.7. Sodium bicarbonate.

All reagents used shall be chemically pure.

I.4. CONDITIONING AND TESTING ATMOSPHERE

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

I.5. LABORATORY TEST SAMPLE

Take a laboratory test sample that is representative of the laboratory bulk sample and sufficient to provide all the specimens, each of at least 1 g, that are required.

I.6. PRE-TREATMENT OF LABORATORY TEST SAMPLE⁽²⁾

Where a substance not to be taken into account in the percentage calculations (see Article 19) is present, it shall first be removed by a suitable method that does not affect any of the fibre constituents.

For this purpose, non-fibrous matter which can be extracted with light petroleum and water is removed by treating the laboratory test sample in a Soxhlet extractor with light petroleum for 1 hour at a minimum rate of six cycles per hour. Allow the light petroleum to evaporate from the sample, which is then extracted by direct treatment consisting in soaking the laboratory test sample in water at room temperature for 1 hour and then soaking it in water at 65 ± 5 °C for a further hour, agitating the liquor from time to time. Use a liquor-laboratory test sample ratio of 100:1. Remove the excess water from the sample by squeezing, suction or centrifuging and then allow the sample to become air-dry.

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

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

Where non-fibrous matter cannot be extracted with light petroleum and water, it shall be removed by substituting for the water method described above a suitable method that does not substantially alter any of the fibre constituents. However, for some unbleached, natural vegetable fibres (e.g. jute, coir) it is to be noted that normal pre-treatment with light petroleum and water does not remove all the natural non-fibrous substances; nevertheless additional pre-treatment is not applied unless the sample contains finishes insoluble in both light petroleum and water.

Analysis reports shall include full details of the methods of pre-treatment used.

I.7. TEST PROCEDURE

I.7.1. General instructions

I.7.1.1. Drying

Conduct all drying operations for not less than 4 hours and not more than 16 hours at 105 ± 3 °C in a ventilated oven with the oven door closed throughout. If the drying period is less than 14 hours, the specimen must be weighed to check that its mass has become constant. The mass may be considered to have become constant if, after a further drying period of 60 minutes, its variation is less than 0,05 %.

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

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

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

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

I.7.1.2. Cooling

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

I.7.1.3. Weighing

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

I.7.2. Procedure

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

I.8. CALCULATION AND EXPRESSION OF RESULTS

Express the mass of the insoluble component as a percentage of the total mass of fibre in the mixture. The percentage of soluble component is obtained by difference. Calculate the results on the basis of clean, dry mass, adjusted by (a) the agreed allowances and (b) the correction factors necessary to take account of loss of matter during pre-treatment and analysis. Calculations shall be made by applying the formula given in I.8.2.

I.8.1. Calculation of percentage of insoluble component on clean, dry mass basis, disregarding loss of fibre mass during pre-treatment:

$$P_1 \% = \frac{100 \cdot r_d}{m}$$

where

$P_1 \%$	is the percentage of clean, dry insoluble component,
m	is the dry mass of the test specimen after pre-treatment,
r	is the dry mass of the residue,
d	is the correction factor for loss in mass of the insoluble component in the reagent during the analysis. Suitable values for 'd' are given in the relevant section of each method.

Of course, these values for 'd' are the normal values applicable to chemically undegraded fibres.

I.8.2. Calculation of percentage of insoluble component on clean, dry mass basis, with adjustment by conventional factors and, where appropriate, correction factors for loss of mass during pre-treatment:

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

where

$P_{1A} \%$	is the percentage of insoluble component adjusted by agreed allowances and for loss in mass during pre-treatment,
P_1	is the percentage of clean dry insoluble component as calculated from the formula shown in I.8.1,
a_1	is the agreed allowance for the insoluble component (see Annex IX),
a_2	is the agreed allowance for the soluble component (see Annex IX),
b_1	is the percentage loss of insoluble component caused by pre-treatment,
b_2	is the percentage loss of soluble component caused by pre-treatment.

The percentage of the second component is $P_{2A} \% = 100 - P_{1A} \%$.

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

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

If normal pre-treatment by extraction with light petroleum and water is applied, correction factors b_1 and b_2 may generally be ignored, except in the case of unbleached cotton, unbleached flax (or linen) and unbleached hemp, where the loss due to the pre-treatment is conventionally taken as 4 %, and in the case of polypropylene, where it is taken as 1 %.

In the case of other fibres, losses due to the pre-treatment are conventionally disregarded in calculations.

II. Method of quantitative analysis by manual separation

II.1. FIELD OF APPLICATION

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

II.2. PRINCIPLE

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

II.3. APPARATUS

- II.3.1. Weighing bottle or any other apparatus giving identical results.
- II.3.2. Desiccator containing self-indicating silica gel.
- II.3.3. Ventilated oven for drying specimens at 105 ± 3 °C.
- II.3.4. Analytical balance, accurate to 0,0002 g.
- II.3.5. Soxhlet extractor, or other apparatus giving an identical result.

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II.3.6. Needle.

II.3.7. Twist tester or similar apparatus.

II.4. REAGENTS

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

II.4.2. Distilled or deionised water.

II.4.3. Acetone.

II.4.4. Orthophosphoric acid.

II.4.5. Urea.

II.4.6. Sodium bicarbonate.

All reagents used shall be chemically pure.

II.5. CONDITIONING AND TESTING ATMOSPHERE

See I.4.

II.6. LABORATORY TEST SAMPLE

See I.5.

II.7. PRE-TREATMENT OF LABORATORY TEST SAMPLE

See I.6.

II.8. PROCEDURE

II.8.1. Analysis of yarn

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

Cut the yarn into pieces of a suitable length and separate the fibre types by means of a needle and, if necessary, a twist tester. The fibre types so obtained are placed in pre-weighed weighing bottles and dried at 105 ± 3 °C until a constant mass is obtained, as described in I.7.1 and I.7.2.

II.8.2. Analysis of cloth

Select from the pre-treated laboratory test sample, well away from all selvages, a specimen of mass not less than 1 g, with edges carefully trimmed to avoid fraying and running parallel with weft or warp yarns, or in the case of knitted fabrics in the line of wales and courses. Separate the different fibre types, collect them in pre-weighed weighing bottles and proceed as described in II.8.1.

II.9. CALCULATION AND EXPRESSION OF RESULTS

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

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

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

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

II.9.2. For calculation of the percentage of each component with adjustment by agreed allowances and, where appropriate, by correction factors for loss of matter during pre-treatment, see I.8.2.

III.1. PRECISION OF THE METHODS

The precision indicated in individual methods relates to the reproducibility.

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

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

Therefore, the difference between two results in a series of analyses made in different laboratories would, given a normal and correct application of the method to an identical and consistent mixture, exceed the confidence limit only in five cases out of 100.

III.2. TEST REPORT

III.2.1. State that the analysis was conducted in accordance with this method.

III.2.2. Give details of any special pre-treatment (see I.6).

III.2.3. Give the individual results and the arithmetic mean, each to an accuracy of 0,1.

IV. Special methods

[^FI]SUMMARY TABLE

Method	Field of application ^a		Reagent
	Soluble component	Insoluble component	
1.	Acetate	Certain other fibres	Acetone
2.	Certain protein fibres	Certain other fibres	Hypochlorite
3.	Viscose, cupro or certain types of modal	Certain other fibres	Formic acid and zinc chloride
4.	Polyamide or nylon	Certain other fibres	Formic acid, 80 % m/m
5.	Acetate	Certain other fibres	Benzyl alcohol
6.	Triacetate or polylactide	Certain other fibres	Dichloromethane

^a Detailed list of fibres under each method.]

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7.	Certain cellulose fibres	Certain other fibres	Sulphuric acid, 75 % m/m
8.	Acrylics, certain modacrylics or certain chlorofibres	Certain other fibres	Dimethylformamide
9.	Certain chlorofibres	Certain other fibres	Carbon disulphide/acetone, 55,5/44,5 v/v
10.	Acetate	Certain other fibres	Glacial acetic acid
11.	Silk, polyamide or nylon	Certain other fibres	Sulphuric acid, 75 % m/m
12.	Jute	Certain animal fibres	Nitrogen content method
13.	Polypropylene	Certain other fibres	Xylene
14.	Certain fibres	Certain other fibres	Concentrated sulphuric acid method
15.	Chlorofibres, certain modacrylics, certain elastanes, acetates, triacetates	Certain other fibres	Cyclohexanone
16.	Melamine	Certain other fibres	Hot formic acid 90 % m/m
[^{F2} 17.	Polyester	Certain other fibres	Trichloroacetic acid and chloroform]

a Detailed list of fibres under each method.]

Textual Amendments

- F1** Substituted by [Commission Delegated Regulation \(EU\) No 286/2012 of 27 January 2012 amending, in order to include a new textile fibre name, Annex I, and, for the purposes of their adaptation to technical progress, Annexes VIII and IX to Regulation \(EU\) No 1007/2011 of the European Parliament and of the Council on textile fibre names and related labelling and marking of the fibre composition of textile products \(Text with EEA relevance\).](#)
- F2** Inserted by [Commission Delegated Regulation \(EU\) 2018/122 of 20 October 2017 amending Annexes I, II, VI, VIII and IX to Regulation \(EU\) No 1007/2011 of the European Parliament and of the Council on textile fibre names and related labelling and marking of the fibre composition of textile products \(Text with EEA relevance\).](#)

METHOD No 1 ACETATE AND CERTAIN OTHER FIBRES (Acetone method)

1. FIELD OF APPLICATION

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

- acetate (19)
with

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2. [^{F3}wool (1), animal hair (2 and 3), silk (4), cotton (5), flax (7) true hemp (8), jute (9), abaca (10), alfa (11), coir (12), broom (13), ramie (14), sisal (15), cupro (21), modal (22), protein (23), viscose (25), acrylic (26), polyamide or nylon (30), polyester (35), polypropylene (37), elastomultiester (45), elastolefin (46), melamine (47), polypropylene/polyamide bicomponent (49) and polyacrylate (50).

In no circumstances is the method applicable to acetate fibres which have been deacetylated on the surface.]

Textual Amendments

- F3** Substituted by [Commission Delegated Regulation \(EU\) 2018/122 of 20 October 2017 amending Annexes I, II, VI, VIII and IX to Regulation \(EU\) No 1007/2011 of the European Parliament and of the Council on textile fibre names and related labelling and marking of the fibre composition of textile products \(Text with EEA relevance\).](#)

2. PRINCIPLE

The acetate is dissolved out from a known dry mass of the mixture, with acetone. The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of dry acetate is found by difference.

3. APPARATUS AND REAGENTS (additional to those specified in the general instructions)

3.1. Apparatus

Glass-stoppered conical flasks of at least 200 ml capacity.

3.2. Reagent

Acetone.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the test specimen contained in a glass-stoppered conical flask of at least 200 ml capacity, add 100 ml of acetone per gram of test specimen, shake the flask, stand it for 30 minutes at room temperature, stirring from time to time, and then decant the liquid through the weighed filter crucible.

Repeat the treatment twice more (making three extractions in all), but for periods of 15 minutes only, so that the total time of treatment in acetone is 1 hour. Transfer the residue to the filter crucible. Wash the residue in the filter crucible with acetone and drain with suction. Refill the crucible with acetone and allow to drain under gravity.

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

[^{F3}5. CALCULATION AND EXPRESSION OF RESULTS

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

6. PRECISION

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

METHOD No 2 CERTAIN PROTEIN FIBRES AND CERTAIN OTHER FIBRES(Method using hypochlorite)

1. FIELD OF APPLICATION

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

1. certain protein fibres, namely: wool (1), animal hair (2 and 3), silk (4), protein (23) with
2. [^F cotton (5), cupro (21), viscose (25), acrylic (26), chlorofibres (27), polyamide or nylon (30), polyester (35), polypropylene (37), elastane (43), glass fibre (44) elastomultiester (45), elastolefin (46), melamine (47) and polypropylene/polyamide bicomponent (49).

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

2. PRINCIPLE

The protein fibre is dissolved out from a known dry mass of the mixture, with a hypochlorite solution. The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of dry protein fibre is found by difference.

Either lithium hypochlorite or sodium hypochlorite can be used for the preparation of the hypochlorite solution.

Lithium hypochlorite is recommended in cases involving a small number of analyses or for analyses conducted at fairly lengthy intervals. This is because the percentage of hypochlorite in solid lithium hypochlorite — unlike that in sodium hypochlorite — is virtually constant. If the percentage of hypochlorite is known, hypochlorite content need not be checked iodometrically for each analysis, since a constant weighed portion of lithium hypochlorite can be employed.

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

3.1. Apparatus

- (a) Erlenmeyer flask with ground-glass stopper, 250 ml.
- (b) Thermostat, adjustable to 20 ± 2 °C.

3.2. Reagents

- (a) Hypochlorite reagent
 - (i) Lithium hypochlorite solution

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

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(ii) Sodium hypochlorite solution

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

Check the active chlorine content of the solution iodometrically before each analysis.

(b) Acetic acid, dilute solution

Dilute 5 ml of glacial acetic acid to 1 litre with water.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows: mix approximately 1 gram of the test specimen with approximately 100 ml of the hypochlorite solution (lithium or sodium hypochlorite) in the 250 ml flask and agitate thoroughly in order to wet out the test specimen.

Then heat the flask for 40 minutes in a thermostat at 20 °C and agitate continuously, or at least at regular intervals. Since the dissolution of the wool proceeds exothermically, the reaction heat of this method must be distributed and removed. Otherwise, considerable errors may be caused by the incipient dissolution of the non-soluble fibres.

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

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

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of 'd' is 1,00, except for cotton, viscose, modal and melamine for which 'd' = 1,01, and unbleached cotton, for which 'd' = 1,03.

6. PRECISION

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

METHOD No 3^[F1]VISCOSE, CUPRO OR CERTAIN TYPES OF MODAL AND CERTAIN OTHER FIBRES(Method using formic acid and zinc chloride)]

1. FIELD OF APPLICATION

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

1. viscose (25) or cupro (21), including certain types of modal fibre (22)
with
2. [^{F1}cotton (5), polypropylene (37), elastolefin (46) and melamine (47).

If a modal fibre is found to be present, a preliminary test shall be carried out to see whether it is soluble in the reagent.

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

2. PRINCIPLE

The viscose, cupro or modal fibre is dissolved from a known dry mass of the mixture, with a reagent consisting of formic acid and zinc chloride. The residue is collected, washed, dried and weighed; its corrected mass is expressed as a percentage of the dry mass of the mixture. The percentage of dry viscose, cupro or modal fibre is found by difference.

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

3.1. Apparatus

- (a) Glass-stoppered conical flasks of at least 200 ml capacity.
- (b) Apparatus for maintaining flasks at 40 ± 2 °C.

3.2. Reagents

- (a) Solution containing 20 g of fused anhydrous zinc chloride and 68 g of anhydrous formic acid made up to 100 g with water (namely 20 parts by mass of fused anhydrous zinc chloride to 80 parts by mass of 85 % m/m formic acid).

Note:

Attention is drawn, in this respect, to point I.3.2.2, which lays down that all reagents used shall be chemically pure; in addition, it is essential to use only fused anhydrous zinc chloride.

- (b) Ammonium hydroxide solution: dilute 20 ml of a concentrated ammonia solution (relative density at 20 °C: 0,880) to 1 litre with water.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows: place the specimen immediately in the flask, pre-heated to 40 °C. Add 100 ml of the solution of formic acid and zinc chloride, pre-heated to 40 °C per gram of specimen. Insert the stopper and shake the flask vigorously. Keep the flask and its contents at a constant temperature of 40 °C for 2,5 hours, shaking the flask at hourly intervals.

Filter the contents of the flask through the weighed filter crucible and with the help of the reagent transfer to the crucible any fibres remaining in the flask. Rinse with 20 ml of reagent pre-heated to 40 °C.

Wash crucible and residue thoroughly with water at 40 °C. Rinse the fibrous residue in approximately 100 ml of cold ammonia solution (3.2(b)) ensuring that this residue remains wholly immersed in the solution for 10 minutes⁽³⁾; then rinse thoroughly 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.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of 'd' is 1,00, except for cotton, for which 'd' = 1,02 and for melamine, for which 'd' = 1,01.]

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6. PRECISION

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

METHOD No 4POLYAMIDE OR NYLON, AND CERTAIN OTHER FIBRES(Method using 80 % m/m formic acid)

1. FIELD OF APPLICATION

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

1. polyamide or nylon (30)
with
2. wool (1), animal hair (2 and 3), cotton (5), cupro (21), modal (22), viscose (25), acrylic (26), chlorofibre (27), polyester (35), polypropylene (37), glass fibre (44), elastomultiester (45), elastolefin (46) and melamine (47).

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

2. PRINCIPLE

The polyamide or nylon fibre is dissolved out from a known dry mass of the mixture, with formic acid. The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of dry polyamide or nylon is found by difference.

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

3.1. Apparatus

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

3.2. Reagents

- (a) Formic acid (80 % m/m, relative density at 20 °C: 1,186). Dilute 880 ml of 90 % m/m formic acid (relative density at 20 °C: 1,204) to 1 litre with water. Alternatively, dilute 780 ml of 98 to 100 % m/m formic acid (relative density at 20 °C: 1,220) to 1 litre with water.

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

- (b) Ammonia, dilute solution: dilute 80 ml of concentrated ammonia solution (relative density at 20 °C: 0,880) to 1 litre with water.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows: to the specimen contained in the conical flask of at least 200 ml capacity, add 100 ml of formic acid per gram of specimen. Insert the stopper, shake the flask to wet out the specimen. Stand the flask for 15 minutes at room temperature, shaking it at intervals. Filter the contents of the flask through the weighed filter crucible and transfer any residual fibres to the crucible by washing out the flask with a little formic acid reagent.

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Drain the crucible with suction and wash the residue on the filter successively with formic acid reagent, hot water, dilute ammonia solution, and finally cold water, draining the crucible with suction after each addition. Do not apply suction until each washing liquor has drained under gravity.

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

5. CALCULATION AND EXPRESSION OF RESULTS

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

6. PRECISION

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

METHOD No 5^[F1] ACETATE AND CERTAIN OTHER FIBRES(Method using benzyl alcohol)]

1. FIELD OF APPLICATION

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

1. acetate (19)
with
2. [^{F3}triacetate (24), polypropylene (37), elastolefin (46), melamine (47), polypropylene/polyamide bicomponent (49) and polyacrylate (50).]

2. PRINCIPLE

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

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

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

3.1. Apparatus

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

3.2. Reagents

- (a) Benzyl alcohol.
- (b) Ethanol.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

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To the specimen contained in the conical flask, add 100 ml of benzyl alcohol per gram of specimen. Insert the stopper, secure the flask to the shaker so that it is immersed in the water-bath, kept at 52 ± 2 °C, and shake for 20 minutes at this temperature.

(Instead of using a mechanical shaker, the flask may be shaken vigorously by hand).

Decant the liquid through the weighed filter crucible. Add a further dose of benzyl alcohol in the flask and shake as before at 52 ± 2 °C for 20 minutes.

Decant the liquid through the crucible. Repeat the cycle of operations a third time.

Finally pour the liquid and the residue into the crucible; wash any remaining fibres from the flask into the crucible with an extra quantity of benzyl alcohol at 52 ± 2 °C. Drain the crucible thoroughly.

Transfer the fibres into a flask, rinse with ethanol and after shaking manually decant through the filter crucible.

Repeat this rinsing operation two or three times. Transfer the residue into the crucible and drain thoroughly. Dry the crucible and the residue and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

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

6. PRECISION

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

METHOD No 6 [TRIACETATES OR POLYLACTIDE AND CERTAIN OTHER FIBRES (Method using dichloromethane)]

1. FIELD OF APPLICATION

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

1. triacetate (24) or polylactide (34)
with
2. [^{F3}wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (21), modal (22), viscose (25), acrylic (26), polyamide or nylon (30), polyester (35), polypropylene (37), glass fibre (44), elastomultiester (45), elastolefin (46), melamine (47), polypropylene/polyamide bicomponent (49) and polyacrylate (50).
Note:

Triacetate fibres which have received a finish leading to partial hydrolysis cease to be completely soluble in the reagent. In such cases, the method is not applicable.]

2. PRINCIPLE

The triacetate or polylactide fibres are dissolved out from a known dry mass of the mixture, with dichloromethane. The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of dry triacetate or polylactide is found by difference.

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

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3.1. Apparatus

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

3.2. Reagent

Dichloromethane.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the test specimen contained in the 200 ml glass-stoppered conical flask, add 100 ml of dichloromethane per gram of the test specimen, insert the stopper, shake the flask to wet out the test specimen and stand for 30 minutes at room temperature, shaking the flask every 10 minutes. Decant the liquid through the weighed filter crucible. Add 60 ml of dichloromethane to the flask containing the residue, shake manually and filter the contents of the flask through the filter crucible. Transfer the residual fibres to the crucible by washing out the flask with a little more dichloromethane. Drain the crucible with suction to remove excess liquid, refill the crucible with dichloromethane and allow it to drain under gravity.

Finally, apply suction to eliminate excess liquid, then treat the residue with boiling water to eliminate all the solvent, apply suction, dry the crucible and residue, cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of 'd' is 1,00, except in the case of polyester, elastomultiester, elastolefin and melamine for which the value of 'd' is 1,01.

6. PRECISION

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

METHOD No 7^[F1]CERTAIN CELLULOSE FIBRES AND CERTAIN OTHER FIBRES(Method using 75 % m/m sulphuric acid)]

1. FIELD OF APPLICATION

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

1. cotton (5), flax (or linen) (7), true hemp (8), ramie (14), cupro (21), modal (22), viscose (25)
with
2. [^{F1}polyester (35), polypropylene (37), elastomultiester (45), elastolefin (46) and polypropylene/polyamide bicomponent (49).]

2. PRINCIPLE

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

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

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3.1. Apparatus

- (a) Glass-stoppered conical flask of at least 500 ml capacity.
- (b) Thermostat or other apparatus for maintaining the flask at 50 ± 5 °C.

3.2. Reagents

- (a) Sulphuric acid, 75 ± 2 % m/m

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

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

- (b) Ammonia, dilute solution

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

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the specimen contained in the glass-stoppered conical flask of at least 500 ml capacity, add 200 ml of 75 % sulphuric acid per gram of specimen, insert the stopper and carefully shake the flask to wet out the specimen.

Maintain the flask at 50 ± 5 °C for 1 hour, shaking it at regular intervals of approximately 10 minutes. Filter the contents of the flask through the weighed filter crucible by means of suction. Transfer any residual fibres by washing out the flask with a little 75 % sulphuric acid. Drain the crucible with suction and wash the residue on the filter once by filling the crucible with a fresh portion of sulphuric acid. Do not apply suction until the acid has drained under gravity.

Wash the residue successively several times with cold water, twice with dilute ammonia solution, and then thoroughly with cold water, draining the crucible with suction after each addition. Do not apply suction until each washing liquor has drained under gravity. Finally, drain the remaining liquid from the crucible with suction, dry the crucible and residue, and cool and weigh them.

[F15. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of 'd' is 1,00, except for polypropylene/polyamide bicomponent, for which the value of 'd' is 1,01.]

6. PRECISION

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

METHOD No 8 ACRYLICS, CERTAIN MODACRYLICS OR CERTAIN CHLOROFIBRES AND CERTAIN OTHER FIBRES(Method using dimethylformamide)

1. FIELD OF APPLICATION

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

- 1. acrylics (26), certain modacrylics (29), or certain chlorofibres (27)⁽⁴⁾
with

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2. [F3 wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (21), modal (22), viscose (25), polyamide or nylon (30), polyester (35), polypropylene (37), elastomultiester (45), elastolefin (46), melamine (47), polypropylene/polyamide bicomponent (49) and polyacrylate (50).

It is equally applicable to acrylics, and certain modacrylics, treated with pre-metallised dyes, but not to those dyed with afterchrome dyes.]

2. PRINCIPLE

The acrylic, modacrylic or chlorofibre is dissolved out from a known dry mass of the mixture, with dimethylformamide heated in a water-bath at boiling point. The residue is collected, washed, dried and weighed. Its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture and the percentage of dry acrylic, modacrylic or chlorofibre is found by difference.

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

3.1. Apparatus

- (a) Glass-stoppered conical flask of at least 200 ml capacity.
- (b) Water bath at boiling point.

3.2. Reagent

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

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

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the specimen contained in the glass-stoppered conical flask of at least 200 ml capacity, add per gram of specimen 80 ml of dimethylformamide, pre-heated in the water-bath at boiling point, insert the stopper, shake the flask to wet out the specimen and heat in the water-bath at boiling point for 1 hour. Shake the flask and its contents gently by hand five times during this period.

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

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

Transfer any residual fibre to the crucible by washing out the beaker with dimethylformamide. Drain the crucible with suction. Wash the residue with about 1 litre of hot water at 70-80 °C, filling the crucible each time.

After each addition of water, apply suction briefly but not until the water has drained under gravity. If the washing liquor drains through the crucible too slowly slight suction may be applied.

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

[F35. CALCULATION AND EXPRESSION OF RESULTS

Changes to legislation: There are outstanding changes not yet made to Regulation (EU) No 1007/2011 of the European Parliament and of the Council. Any changes that have already been made to the legislation appear in the content and are referenced with annotations. (See end of Document for details) View outstanding changes

Calculate the results as described in the general instructions. The value of 'd' is 1,00, except in the case of wool, cotton, cupro, modal, polyester, elastomultiester, melamine and polyacrylate, for which 'd' is 1,01.]

6. PRECISION

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

METHOD No 9 CERTAIN CHLOROFIBRES AND CERTAIN OTHER FIBRES (Method using 55,5/44,5 % v/v mixture of carbon disulphide and acetone)

1. FIELD OF APPLICATION

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

1. certain chlorofibres (27), namely certain polyvinyl chloride fibres, whether after-chlorinated or not⁽⁶⁾
with
2. [^{F3}wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (21), modal (22), viscose (25), acrylic (26), polyamide or nylon (30), polyester (35), polypropylene (37), glass fibre (44), elastomultiester (45), melamine (47), polypropylene/polyamide bicomponent (49) and polyacrylate (50).

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

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

2. PRINCIPLE

The chlorofibre is dissolved out from a known dry mass of the mixture, with an azeotropic mixture of carbon disulphide and acetone. The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of dry polyvinyl chloride fibre is found by difference.

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

3.1. Apparatus

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

3.2. Reagents

- (a) Azeotropic mixture of carbon disulphide and acetone (55,5 % by volume carbon disulphide to 44,5 % acetone). As this reagent is toxic, the use of a hood is recommended.
- (b) Ethanol (92 % by volume) or methanol.

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

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To the specimen contained in the glass-stoppered conical flask of at least 200 ml capacity, add 100 ml of the azeotropic mixture per gram of specimen. Seal the flask securely, and shake the flask on a mechanical shaker, or vigorously by hand, for 20 minutes at room temperature.

Decant the supernatant liquid through the weighed filter crucible.

Repeat the treatment with 100 ml of fresh reagent. Continue this cycle of operations until no polymer deposit is left on a watch glass when a drop of the extraction liquid is evaporated. Transfer the residue to the filter crucible using more reagent, apply suction to remove the liquid, and rinse the crucible and residue with 20 ml of alcohol and then three times with water. Allow the washing liquor to drain under gravity before draining with suction. Dry the crucible and residue and cool and weigh them.

Note:

With certain mixtures having a high chlorofibre content there may be substantial shrinkage of the specimen during the drying procedure, as a result of which the dissolution of chlorofibre by the solvent is retarded.

This does not, however, affect the ultimate dissolution of the chlorofibre in the solvent.

[^{F3}5. CALCULATION AND EXPRESSION OF RESULTS

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

6. PRECISION

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

METHOD No 10[^{F1}ACETATE AND CERTAIN OTHER FIBRES(Method using glacial acetic acid)]

1. FIELD OF APPLICATION

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

1. acetate (19)
with
2. [^{F1}certain chlorofibres (27) namely polyvinyl chloride fibres, whether after-chlorinated or not, polypropylene (37), elastolefin (46), melamine (47) and polypropylene/polyamide bicomponent (49).]

2. PRINCIPLE

The acetate fibre is dissolved out from a known dry mass of the mixture, with glacial acetic acid. The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of dry acetate is found by difference.

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

3.1. Apparatus

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

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3.2. Reagent

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

4. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the specimen contained in the glass-stoppered conical flask of at least 200 ml capacity, add 100 ml glacial acetic acid per gram of specimen. Seal the flask securely and shake on the mechanical shaker, or vigorously by hand, for 20 minutes at room temperature. Decant the supernatant liquid through the weighed filter crucible. Repeat this treatment twice, using 100 ml of fresh reagent each time, making three extractions in all.

Transfer the residue to the filter crucible, drain with suction to remove the liquid and rinse the crucible and the residue with 50 ml of glacial acetic acid, and then three times with water. After each rinse, allow the liquid to drain under gravity before applying suction. Dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of 'd' is 1,00.

6. PRECISION

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

METHOD No 11^[F1] SILK OR POLYAMIDE AND CERTAIN OTHER FIBRES(Method using 75 % m/m sulphuric acid)]

[F1]1. FIELD OF APPLICATION

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

1. silk (4) or polyamide or nylon (30)
with
2. wool (1), animal hair (2 and 3), polypropylene (37), elastolefin (46), melamine (47) and polypropylene/polyamide bicomponent (49).]

[F1]2. PRINCIPLE

The silk or polyamide or nylon fibre is dissolved out from a known dry mass of the mixture, with 75 % m/m sulphuric acid⁽⁶⁾.

The residue is collected, washed, dried and weighed. Its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of the dry silk or polyamide or nylon is found by difference.]

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

3.1. Apparatus

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

3.2. Reagents

Changes to legislation: There are outstanding changes not yet made to Regulation (EU) No 1007/2011 of the European Parliament and of the Council. Any changes that have already been made to the legislation appear in the content and are referenced with annotations. (See end of Document for details) View outstanding changes

(a) Sulphuric acid (75 ± 2 % m/m)

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

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

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

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

[F14. TEST PROCEDURE

Follow the procedure described in the general instructions and proceed as follows:

To the specimen contained in a glass-stoppered conical flask of at least 200 ml capacity, add 100 ml of 75 % m/m sulphuric acid per gram of specimen and insert the stopper. Shake vigorously and stand for 30 minutes at room temperature. Shake again and stand for 30 minutes. Shake a last time and filter the contents of the flask through the weighed filter crucible. Wash any remaining fibres from the flask with the 75 % sulphuric acid reagent. Wash the residue on the crucible successively with 50 ml of the dilute sulphuric acid reagent, 50 ml water and 50 ml of the dilute ammonia solution. Each time allow the fibres to remain in contact with the liquid for about 10 minutes before applying suction. Finally rinse with water, leaving the fibres in contact with the water for about 30 minutes. Drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

In the case of binary mixtures of polyamide with polypropylene/polyamide bicomponent, after filtering fibres through the weighed filter crucible and before applying the described washing procedure, wash twice the residue on the filter crucible with 50 ml of 75 % sulphuric acid reagent each time.]

[F15. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of 'd' is 1,00, except for wool, for which 'd' = 0,985, for polypropylene/polyamide bicomponent, for which 'd' = 1,005 and for melamine, for which 'd' = 1,01.

6. PRECISION

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

METHOD No 12. JUTE AND CERTAIN ANIMAL FIBRES (Method by determining nitrogen content)

1. FIELD OF APPLICATION

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

1. jute (9)

with

2. certain animal fibres.

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

2. PRINCIPLE

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

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

3.1. Apparatus

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

3.2. Reagents

- (a) Toluene.
- (b) Methanol.
- (c) Sulphuric acid, relative density at 20 °C: 1,84⁽⁷⁾.
- (d) Potassium sulphate⁽⁷⁾.
- (e) Selenium dioxide⁽⁷⁾.
- (f) Sodium hydroxide solution (400 g/litre). Dissolve 400 g of sodium hydroxide in 400-500 ml of water and dilute to 1 litre with water.
- (g) Mixed indicator. Dissolve 0,1 g of methyl red in 95 ml of ethanol and 5 ml of water, and mix with 0,5 g of bromocresol green dissolved in 475 ml of ethanol and 25 ml of water.
- (h) Boric acid solution. Dissolve 20 g of boric acid in 1 litre of water.
- (i) Sulphuric acid, 0,02N (standard volumetric solution).

4. PRE-TREATMENT OF TEST SAMPLE

The following pre-treatment is substituted for the pre-treatment described in the general instructions:

Extract the air-dry laboratory test sample in a Soxhlet apparatus with a mixture of 1 volume of toluene and 3 volumes of methanol for 4 hours at a minimum rate of 5 cycles per hour. Allow the solvent to evaporate from the sample in air, and remove the last traces in an oven at 105 ± 3 °C. Then extract the sample in water (50 ml per g of sample) by boiling under reflux for 30 minutes. Filter, return the sample to the flask, and repeat the extraction with an identical volume of water. Filter, remove excess water from the sample by squeezing, suction, or centrifuging and then allow the sample to become air-dry.

Note:

The toxic effects of toluene and methanol shall be borne in mind and full precautions shall be taken in their use.

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5. TEST PROCEDURE

5.1. General instructions

Follow the procedure described in the general instructions as regards the selection, drying and weighing of the specimen.

5.2. Detailed procedure

Transfer the specimen to a Kjeldahl digestion flask. To the specimen weighing at least 1 g contained in the digestion flask, add, in the following order, 2,5 g potassium sulphate, 0,1-0,2 g selenium dioxide and 10 ml sulphuric acid (relative density at 20 °C: 1,84). Heat the flask, gently at first, until the whole of the fibre is destroyed, and then heat it more vigorously until the solution becomes clear and almost colourless. Heat it for a further 15 minutes. Allow the flask to cool, dilute the contents carefully with 10-20 ml water, cool, transfer the contents quantitatively to a 200 ml graduated flask and make up to volume with water to form the digest solution. Place about 20 ml of boric acid solution in a 100 ml conical flask and place the flask under the condenser of the Kjeldahl distillation apparatus so that the delivery tube dips just below the surface of the boric acid solution. Transfer exactly 10 ml of digest solution to the distillation flask, add not less than 5 ml of sodium hydroxide solution to the funnel, lift the stopper slightly and allow the sodium hydroxide solution to run slowly into the flask. If the digest solution and sodium hydroxide solution remain as two separate layers, mix them by gentle agitation. Heat the distillation flask gently and pass it into steam from the generator. Collect about 20 ml of distillate, lower the conical flask so that the tip of the delivery tube of the condenser is about 20 mm above the surface of the liquid and distil for 1 minute more. Rinse the tip of the delivery tube with water, catching the washings in the conical flask. Remove the conical flask and replace it with another conical flask containing roughly 10 ml of boric acid solution and collect about 10 ml distillate.

Titrate the two distillates separately with 0,02 N sulphuric acid, use the mixed indicator. Record the total titre for the two distillates. If the titre for the second distillate is more than 0,2 ml, repeat the test and start the distillation again using a fresh aliquot of digest solution.

Carry out a blank determination, i.e. digestion and distillation using the reagents only.

6. CALCULATION AND EXPRESSION OF RESULTS

6.1. Calculate the percentage nitrogen content in the dry specimen as follows:

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

where

A	= percentage nitrogen in the clean dry specimen,
V	= total volume in ml of standard sulphuric acid used in the determination,
b	= total volume in ml of standard sulphuric acid used in the blank determination,
N	= normality of standard sulphuric acid,
W	= dry mass (g) of specimen.

6.2. Using the values of 0,22 % for the nitrogen content of jute and 16,2 % for the nitrogen content of animal fibre, both percentages being expressed on the dry mass of the fibre, calculate the composition of the mixture as follows:

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

where

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PA% = percentage of animal fibre in the clean dry specimen.

7. PRECISION

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

METHOD No 13 **POLYPROPYLENE FIBRES AND CERTAIN OTHER FIBRES** (Xylene method)

1. FIELD OF APPLICATION

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

1. polypropylene fibres (37)
with
2. [^{F3}wool (1), animal hair (2 and 3), silk (4), cotton (5), acetate (19), cupro (21), modal (22), triacetate (24), viscose (25), acrylic (26), polyamide or nylon (30), polyester (35), glass fibre (44), elastomultiester (45), melamine (47) and polyacrylate (50).]

2. PRINCIPLE

The polypropylene fibre is dissolved out from a known dry mass of the mixture with boiling xylene. The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of polypropylene is found by difference.

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

3.1. Apparatus

- (a) Glass-stoppered conical flask of at least 200 ml capacity.
- (b) Reflux condenser (suitable for liquids of high boiling point), fitting the conical flask (a).
- (c) Heating mantle at boiling point of xylene.

3.2. Reagent

Xylene distilling between 137 and 142 °C.

Note:

Xylene is highly flammable and has a toxic vapour. Suitable precautions must be taken in its use.

4. TEST PROCEDURE

Follow the procedure described in the general instructions then proceed as follows:

To the specimen contained in the conical flask (3.1(a)), add 100 ml of xylene (3.2) per gram of specimen. Attach the condenser (3.1(b)), bring the contents to the boil and maintain at boiling point for 3 minutes.

Immediately decant the hot liquid through the weighed filter crucible (see Note 1). Repeat this treatment twice more, each time using a fresh 50 ml portion of solvent.

Wash the residue remaining in the flask successively with 30 ml of boiling xylene (twice), then with 75 ml of light petroleum (I.3.2.1 of general instructions) (twice). After the second wash

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with light petroleum, filter the contents of the flask through the crucible, transfer any residual fibres to the crucible with the aid of a small quantity of light petroleum and allow the solvent to evaporate. Dry the crucible and residue, cool and weigh them.

Notes:

1. The filter crucible through which the xylene is to be decanted must be pre-heated.
2. After the treatment with boiling xylene, ensure that the flask containing the residue is cooled sufficiently before the light petroleum is introduced.
3. In order to reduce the fire and toxicity hazards to the operator, a hot extraction apparatus using the appropriate procedures, giving identical results, may be used⁽⁸⁾.

[^{F3}5. CALCULATION AND EXPRESSION OF RESULTS

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

6. PRECISION

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

METHOD No 14[^{F1}**CERTAIN FIBRES AND CERTAIN OTHER FIBRES**(Method using concentrated sulphuric acid)]

1. FIELD OF APPLICATION

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

1. cotton (5), acetate (19), cupro (21), modal (22), triacetate (24), viscose (25), certain acrylics (26), certain modacrylics (29), polyamide or nylon (30), polyester (35) and elastomultiester (45)
with
2. [^{F1}chlorofibres (27) based on homopolymers of vinyl chloride, whether after-chlorinated or not, polypropylene (37), elastolefin (46), melamine (47) and polypropylene/polyamide bicomponent (49).

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

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

[^{F1}2. PRINCIPLE

The constituent other than the chlorofibre, polypropylene, elastolefin, melamine or polypropylene/polyamide bicomponent (i.e. the fibres mentioned in paragraph 1.1) is dissolved out from a known dry mass of the mixture with concentrated sulphuric acid (relative density 1,84 at 20 °C). The residue, consisting of the chlorofibre, polypropylene, elastolefin, melamine or polypropylene/polyamide bicomponent is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of the second constituents is obtained by difference.]

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

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

(b) Glass rod with flattened end.

3.2. Reagents

(a) Sulphuric acid, concentrated (relative density at 20 °C: 1,84).

(b) Sulphuric acid, approximately 50 % (m/m) aqueous solution.

Prepare by adding carefully, while cooling, 400 ml of sulphuric acid (relative density at 20 °C: 1,84) to 500 ml of distilled or deionised water. After cooling to room temperature, dilute the solution to one litre with water.

(c) Ammonia, dilute solution.

Dilute 60 ml of concentrated ammonia solution (relative density at 20 °C: 0,880) to one litre with distilled water.

4. TEST PROCEDURE

Follow the procedure described in the general instructions, then proceed as follows:

To the test specimen contained in the flask (3.1(a)) add 100 ml of sulphuric acid (3.2(a)) per gram of specimen.

Allow the contents of the flask to remain at room temperature for 10 minutes and during that time stir the test specimen occasionally by means of the glass rod. If a woven or knitted fabric is being treated, wedge it between the wall of the flask and the glass rod and exert a light pressure in order to separate the material dissolved by the sulphuric acid.

Decant the liquid through the weighed filter crucible. Add to the flask a fresh portion of 100 ml of sulphuric acid (3.2(a)) and repeat the same operation. Transfer the contents of the flask to the filter crucible and transfer the fibrous residue there with the aid of the glass rod. If necessary, add a little concentrated sulphuric acid (3.2(a)) to the flask in order to remove any fibres adhering to the wall. Drain the filter crucible with suction; remove the filtrate by emptying or changing the filter-flask, wash the residue in the crucible successively with 50 % sulphuric acid solution (3.2(b)), distilled or deionised water (I.3.2.3 of the general instructions), ammonia solution (3.2(c)) and finally wash thoroughly with distilled or deionised water, draining the crucible with suction after each addition. (Do not apply suction during the washing operation, but only after the liquid has drained off by gravity.) Dry the crucible and residue, cool and weigh them.

[F15. CALCULATION AND EXPRESSION OF RESULTS

Calculate the results as described in the general instructions. The value of 'd' is 1,00, except for melamine and polypropylene/polyamide bicomponent, for which the value of 'd' is 1,01.]

6. PRECISION

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

METHOD No 15 CHLOROFIBRES, CERTAIN MODACRYLICS, CERTAIN ELASTANES, ACETATES, TRIACETATES AND CERTAIN OTHER FIBRES (Method using cyclohexanone)

1. FIELD OF APPLICATION

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

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

with

2. [^{F3}wool (1), animal hair (2 and 3), silk (4), cotton (5), cupro (21), modal (22), viscose (25), acrylic (26), polyamide or nylon (30), glass fibre (44), melamine (47) and polyacrylate (50).

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

Mixtures containing chlorofibres may also be analysed by using method No 9 or 14.]

2. PRINCIPLE

The acetate and triacetate fibres, chlorofibres, certain modacrylics, and certain elastanes are dissolved out from a known dry mass with cyclohexanone at a temperature close to boiling point. The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of chlorofibre, modacrylic, elastane, acetate and triacetate is found by difference.

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

3.1. Apparatus

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

3.2. Reagents

- (a) Cyclohexanone, boiling point 156 °C.
- (b) Ethyl alcohol, 50 % by volume.

Note:

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

4. TEST PROCEDURE

Follow the procedure described in the general instructions and then proceed as follows:

Pour into the distillation flask 100 ml of cyclohexanone per gram of material, insert the extraction container in which the filter crucible, containing the specimen and the porous baffle, slightly inclined, have previously been placed. Insert the reflux condenser. Bring to the boil and continue extraction for 60 minutes at a minimum rate of 12 cycles per hour.

After extraction and cooling remove the extraction container, take out the filter crucible and remove the porous baffle. Wash the contents of the filter crucible three or four times with 50 % ethyl alcohol heated to about 60 °C and subsequently with 1 litre of water at 60 °C.

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

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

^{F3}5. CALCULATION AND EXPRESSION OF RESULTS

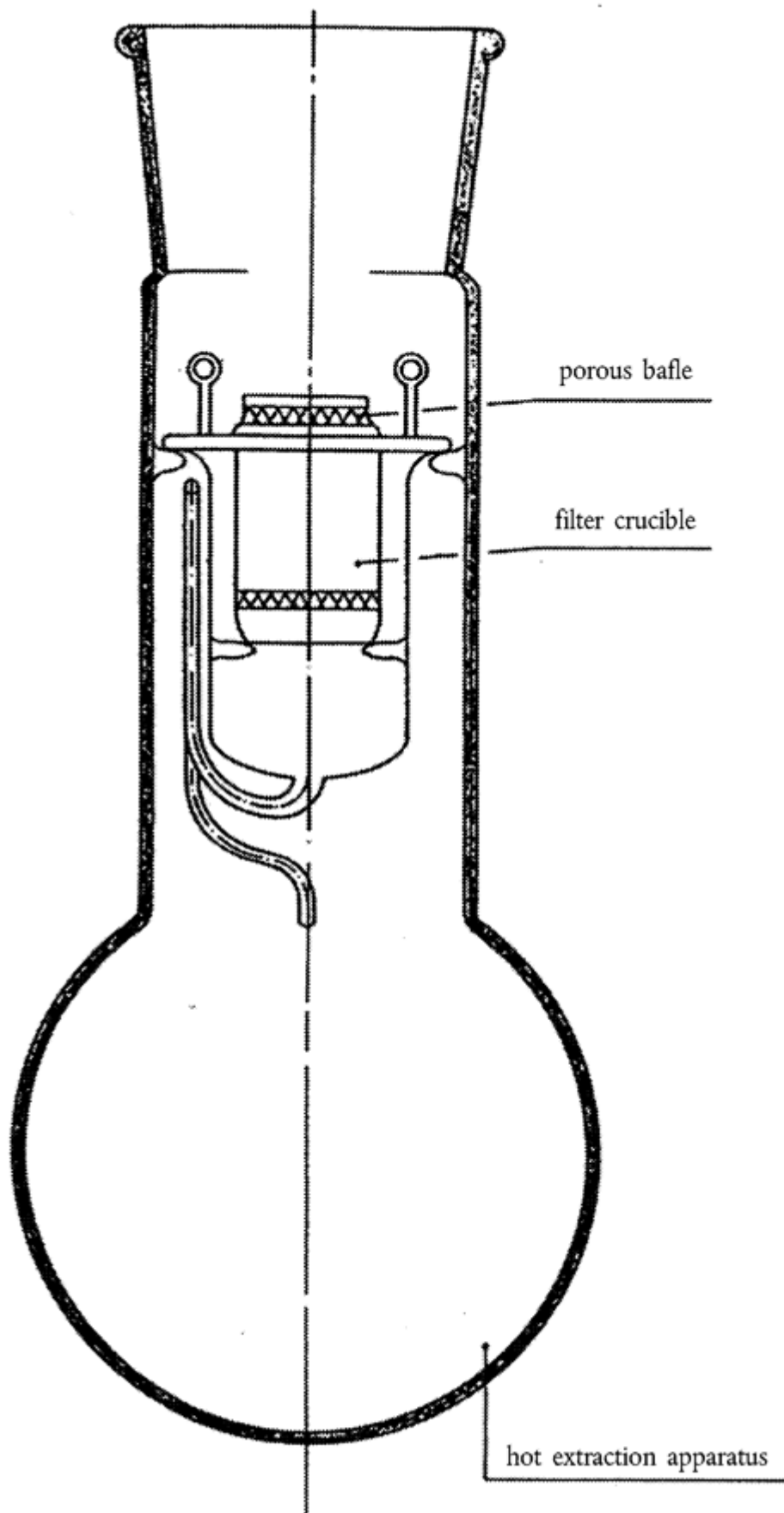
Calculate the results as described in the general instructions. The value of 'd' is 1,00, except in the case of polyacrylate, for which 'd' is 1,02, silk and melamine, for which 'd' is 1,01, and acrylic, for which 'd' is 0,98.]

6. PRECISION

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

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



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METHOD No 16^[1] MELAMINE AND CERTAIN OTHER FIBRES (Method using hot formic acid)

1. FIELD OF APPLICATION

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

1. melamine (47)
with
2. ^[1]cotton (5), aramid (31) and polypropylene (37).]

2. PRINCIPLE

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

The residue is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of the second constituents is obtained by difference.

Note:

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

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

3.1. Apparatus

- (a) Glass-stoppered conical flask of at least 200 ml capacity.
- (b) Shaking water bath or other apparatus to shake and maintain the flask at 90 ± 2 °C.

3.2. Reagents

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

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

- (b) Ammonia, dilute solution: dilute 80 ml of concentrated ammonia solution (relative density at 20 °C: 0,880) to 1 litre with water.

4. TEST PROCEDURE

Follow the procedure described in the general instructions, then proceed as follows:

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

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each washing liquor has drained under gravity. Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

5. CALCULATION AND EXPRESSION OF RESULTS

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

6. PRECISION

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

[^{F2}METHOD No 17 **POLYESTER AND CERTAIN OTHER FIBRES**(Method using trichloroacetic acid and chloroform)]

1. FIELD OF APPLICATION

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

1. polyester (35)
with
2. polyacrylate (50)

2. GENERAL INFORMATION

The principle, apparatus and reagent, test procedure, calculation and expression of results that apply to binary fibre mixtures of polyester with polyacrylate are those described in standard EN ISO 1833-25:2013. The 'd' value is 1,01.]

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- (1) Method 12 is an exception. It is based on a determination of the content of a constituent substance of one of the two components.
- (2) See Chapter 1.1.
- (3) To ensure that the fibrous residue is immersed in the ammonia solution for 10 minutes, one may, for example, use a filter crucible adaptor fitted with a tap by which the flow of the ammonia solution can be regulated.
- (4) The solubility of such modacrylics or chlorofibres in the reagent shall be checked before carrying out the analysis.
- (5) Before carrying out the analysis, the solubility of the polyvinyl chloride fibres in the reagent shall be checked.
- (6) [^{F1}Wild silks, such as tussah silk, are not completely soluble in 75 % m/m sulphuric acid.]
- (7) These reagents should be nitrogen-free.
- (8) See for example the apparatus described in Melliand Textilberichte 56 (1975), pp. 643-645.

Textual Amendments

- F1** Substituted by [Commission Delegated Regulation \(EU\) No 286/2012 of 27 January 2012 amending, in order to include a new textile fibre name, Annex I, and, for the purposes of their adaptation to technical progress, Annexes VIII and IX to Regulation \(EU\) No 1007/2011 of the European Parliament and of the Council on textile fibre names and related labelling and marking of the fibre composition of textile products \(Text with EEA relevance\).](#)

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Changes and effects yet to be applied to the whole legislation item and associated provisions

- Annex 8 Ch. 2 para. 1.7.2 word omitted by [S.I. 2018/1398 reg. 3\(16\)\(a\)](#)
- Annex 8 Ch. 3 para. 1.7.2 word omitted by [S.I. 2018/1398 reg. 3\(16\)\(b\)](#)
- Annex 8 Ch. 3 s. 5 heading word omitted by [S.I. 2018/1398 reg. 3\(16\)\(c\)](#)
- Art. 16(3)(4) substituted for Art. 16(3) by [S.I. 2018/1398 reg. 3\(8\)](#)
- Art. 16(4) substituted by S.I. 2018/1398, reg. 3(8) (as amended) by [S.I. 2020/1347 reg. 7\(2\)\(d\)](#)