
Status: Point in time view as at 31/01/2020.

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)

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)

*Status: Point in time view as at 31/01/2020.**Changes to legislation: There are outstanding changes not yet made to Regulation (EU) No 1007/2011 of the European Parliament and of the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)*

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 MIXTURESIV. **Special methods**[^{F1}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
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	Certain other fibres	Cyclohexanone

^a Detailed list of fibres under each method.]

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	elastanes, acetates, triacetates		
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:

1. acetate (19)
with
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.

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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

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. [^{F1}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,

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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.

- (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.

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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.

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

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- (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⁽¹⁾; 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.

[^F15. 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.]

6. PRECISION

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

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

1. FIELD OF APPLICATION

This method is 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).

Status: Point in time view as at 31/01/2020.

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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.

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

Status: Point in time view as at 31/01/2020.

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)

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:

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

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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^[F1] **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)

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.

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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)

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:

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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 8ACRYLICS, 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)⁽²⁾
with
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.

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(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

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⁽³⁾
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

Status: Point in time view as at 31/01/2020.

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)

(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:

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.

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

Status: Point in time view as at 31/01/2020.

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)

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.

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.

Status: Point in time view as at 31/01/2020.

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)

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⁽⁴⁾.

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

- (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.

^[F1]4. 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

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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.

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.

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

4. PRE-TREATMENT OF TEST SAMPLE

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

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

Note:

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

5. TEST PROCEDURE

5.1. General instructions

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

5.2. Detailed procedure

Transfer the specimen to a Kjeldahl digestion flask. To the specimen weighing at least 1 g contained in the digestion flask, add, in the following order, 2,5 g potassium sulphate, 0,1-0,2 g selenium dioxide and 10 ml sulphuric acid (relative density at 20 °C: 1,84). Heat the flask, gently at first, until the whole of the fibre is destroyed, and then heat it more vigorously until the solution becomes clear and almost colourless. Heat it for a further 15 minutes. Allow the flask to cool, dilute the contents carefully with 10-20 ml water, cool, transfer the contents quantitatively to a 200 ml graduated flask and make up to volume with water to form the digest solution. Place about 20 ml of boric acid solution in a 100 ml conical flask and place the flask under the condenser of the Kjeldahl distillation apparatus so that the delivery tube dips just below the surface of the boric acid solution. Transfer exactly 10 ml of digest solution to the distillation flask, add not less than 5 ml of sodium hydroxide solution to the funnel, lift the stopper slightly and allow the sodium hydroxide solution to run slowly into the flask. If the digest solution and

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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

- 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:

- polypropylene fibres (37)
with
- [^{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

Status: Point in time view as at 31/01/2020.

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)

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 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⁽⁶⁾.

[F35. 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 %.

Status: Point in time view as at 31/01/2020.

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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

- (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:

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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:

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.

Status: Point in time view as at 31/01/2020.

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)

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.

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.

[F35. 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.]

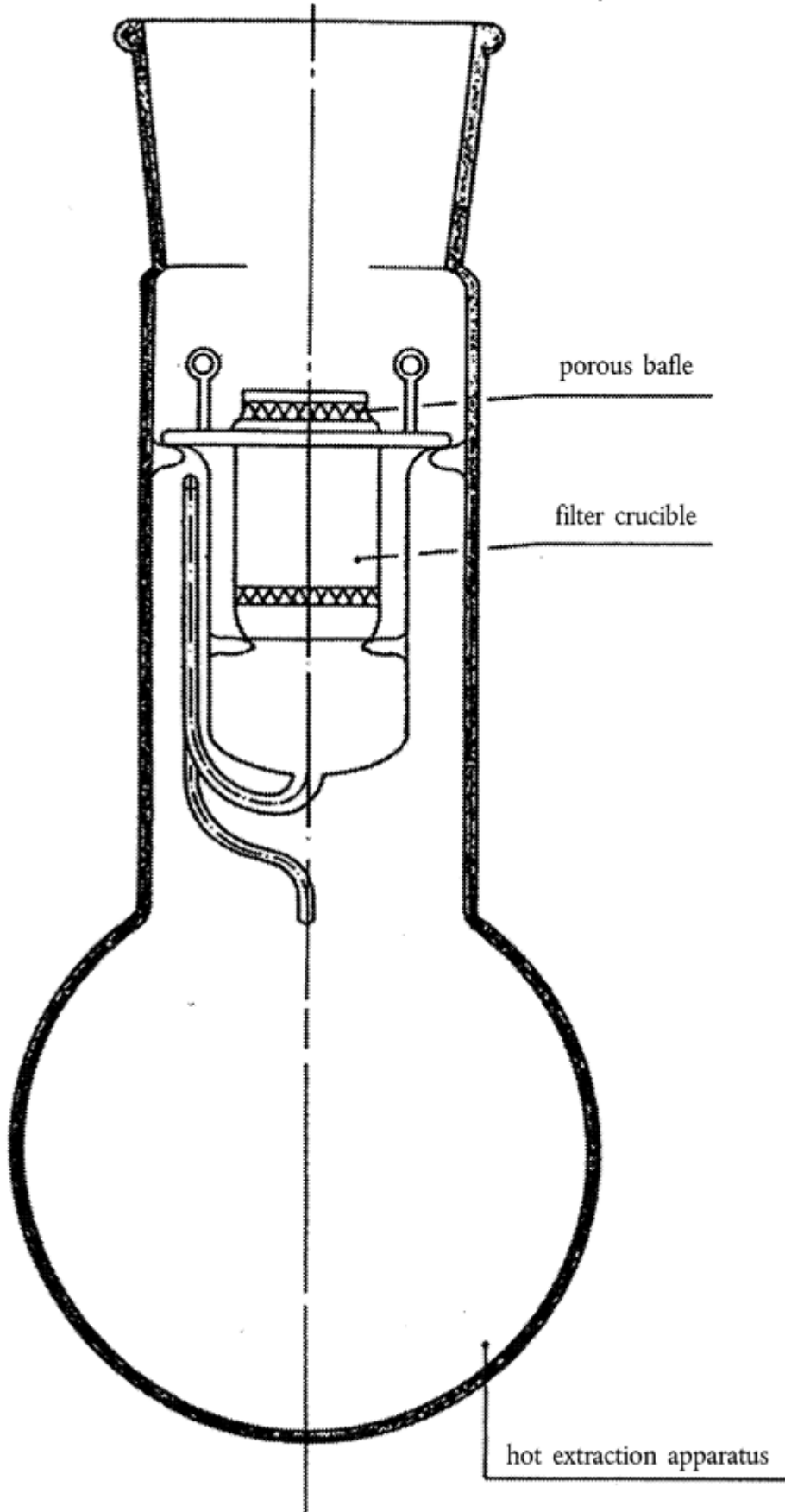
6. PRECISION

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 %.

Status: Point in time view as at 31/01/2020.

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)

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



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METHOD No 16^[F1] 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. [^{F1}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) 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.
- (2) The solubility of such modacrylics or chlorofibres in the reagent shall be checked before carrying out the analysis.
- (3) Before carrying out the analysis, the solubility of the polyvinyl chloride fibres in the reagent shall be checked.
- (4) [^{F1}Wild silks, such as tussah silk, are not completely soluble in 75 % m/m sulphuric acid.]
- (5) These reagents should be nitrogen-free.
- (6) 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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