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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1833

TEXTILES

BINARY FIBRE MIXTURES

QUANTITATIVE CHEMICAL ANALYSIS

1st EDITION

October 1971

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BRIEF HISTORY

The ISO Recommendation R 1833, *Textiles – Binary fibre mixtures – Quantitative chemical analysis,* was drawn up by Technical Committee ISO/TC 38, *Textiles,* the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1833, which was circulated to all the ISO Member Bodies for enquiry in January 1970.

The Draft has been approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	India	South Africa, Rep. of
Belgium	Israel	Sweden
Canada	Japan	Switzerland
Czechoslovakia	Netherlands	Turkey
Denmark	New Zealand	U.A.R.
France	Norway	United Kingdom
Germany	Poland	U.S.A.
Greece	Portugal	U.S.S.R.
Hungary	Romania	

The following Member Body opposed the approval of the Draft :

Italy

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

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TEXTILES

BINARY FIBRE MIXTURES

QUANTITATIVE CHEMICAL ANALYSIS

INTRODUCTION

In general, the methods described in this ISO Recommendation are based on the selective solution of an individual component. After the removal of a component, the insoluble residue is weighed, and the proportion of soluble component is calculated from the loss in mass. The information common to the analyses by this method of all fibre mixtures, whatever their composition, is given in section 1 of this ISO Recommendation. This should be used in conjunction with the succeeding individual sections of the document which contain the detailed procedures applicable to particular fibre mixtures. Where, occasionally, an analysis is based on a principle other than selective solution, full details are given in the appropriate section.

Mixtures of fibres during processing and, to a lesser extent, finished textiles may contain fats, waxes or dressings, either occurring naturally or added to facilitate processing. Salts and other water-soluble matter may also be present. Some or all of these substances would be removed during analysis, and calculated as the soluble-fibre component. To avoid this error, non-fibrous matter must be removed before analysis and a method of pre-treatment for removing oils, fats, waxes and water-soluble matter is given in section 1.

In addition, textiles may contain resins or other matter added to bond the fibres together or to confer special properties, such as water-repellence or crease-resistance. Such matter, including dyestuffs in exceptional cases, may interfere with the action of the reagent on the soluble component and/or it may be partially or completely removed by the reagent. This type of added matter may also cause errors and should be removed before the sample is analysed. If it is impossible to remove such added matter, the methods of analysis are no longer applicable. Dye in dyed fibres is considered to be an integral part of the fibre and is not removed.

Most textile fibres contain water, the amount depending on the type of fibre and on the relative humidity of the surrounding air. Analyses are conducted on the basis of dry mass, and a procedure for determining the dry mass of test specimens and residues is given in section 1. The result is therefore obtained on the basis of clean, dry fibres.

Provision is made for recalculating the result on the basis of

(1) agreed allowances for moisture content*, and

(2) agreed allowances for moisture and also for

  (*a*) fibrous matter removed in the pre-treatment, and

  (*b*) non-fibrous matter (for example, fibre dressing, processing oil, or size) that can be properly regarded as part of the fibre as an article of commerce.

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

The procedures given apply to single determinations; at least two determinations on separate test specimens should be made, but more may be carried out if desired. Before proceeding with any analysis, all the fibres present in the mixture should have been identified. For confirmation, unless it is technically impossible, it is recommended to use alternative procedures whereby the constituent that would be the residue in the standard method is dissolved out first.

If it is practicable to separate the components of a mixture manually, that method should be used in preference to the chemical methods of analysis given in this ISO Recommendation.

* Suitable values are the official regains, where available, of the respective fibres.

1. INFORMATION COMMON TO THE METHODS GIVEN FOR QUANTITATIVE CHEMICAL ANALYSIS OF BINARY FIBRE MIXTURES

1.1 Scope and field of application

This ISO Recommendation contains methods for the quantitative chemical analysis of various binary mixtures of fibres. The methods given are applicable in general to fibres in any textile form. Where certain textile forms are excepted, these are listed in the "field of application" clause of the individual method.

1.2 Principle

After the identification of the components of a mixture, one component is removed, usually by selective solution, the insoluble residue is weighed, and the proportion of soluble component calculated from the loss in mass. The fibre in the larger proportion is removed first.

1.3 Reagents

All reagents used should be chemically pure.

1.3.1 *Light petroleum,* redistilled, distilling between 40 and 60 °C.

1.3.2 *Distilled or deionized water.*

1.3.3 *Additional reagents* as specified in the appropriate sections of this ISO Recommendation.

1.4 Apparatus

1.4.1 *Glass filter crucible,* capacity 30 to 40 ml, with sealed-in sintered disk filter with pore size of 90 to 150 μm.

The crucible should be provided with either a ground glass stopper or a watch-glass cover.

1.4.2 *Vacuum flask.*

1.4.3 *Desiccator* containing self-indicating silica gel.

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

1.4.5 *Analytical balance,* accurate to 0.0002 g.

1.4.6 *Soxhlet extraction apparatus,* of sufficient size to give a volume, in millilitres, equal to 20 times the mass, in grammes, of the specimen.

1.4.7 *Additional apparatus* as specified in the appropriate sections of this ISO Recommendation.

1.5 Conditioning and testing atmosphere

Because dry masses are determined, it is unnecessary to condition the specimen. The analysis is carried out under ordinary room conditions.

1.6 Sampling and pre-treatment of sample

1.6.1 *Sampling.* 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. Fabrics may contain yarns of different composition and account must be taken of this fact in the sampling of the fabric. Treat the sample as described in clause 1.6.2.

* See ISO Recommendation R . . ., *Methods of sampling for chemical testing* (at present, document ISO/TC 38 N 378).

1.6.2 *Pre-treatment of laboratory test sample.* Extract the air-dry sample in a Soxhlet apparatus with light petroleum for 1 hour at a minimum rate of 6 cycles per hour. Allow the light petroleum to evaporate from the sample and then soak the specimen in cold water for 1 hour and then in water at 65 ± 5 °C for a further hour. In both cases use a liquor : specimen ratio of 100 : 1 and agitate the liquor from time to time. Remove the excess water from the sample by squeezing, suction, or centrifuging and then allow the sample to become air-dry.

Where non-fibrous matter cannot be extracted with light petroleum and water, it should be removed by a suitable method that does not substantially alter any of the fibre constituents. However, for some unbleached, natural vegetable fibres (for example 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 does contain finishes insoluble in both light petroleum and water.

1.7 Test procedure

1.7.1 *General instructions*

1.7.1.1 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.

1.7.1.2 DRYING OF SPECIMEN. Dry the specimen in a weighing bottle with its stopper beside it. After drying, stopper the weighing bottle before removing it from the oven, and transfer it quickly to a desiccator.

1.7.1.3 DRYING OF CRUCIBLE AND RESIDUE. Dry the filter crucible with its stopper or cover beside it in the oven. After drying, close the crucible and transfer it quickly to a desiccator.

1.7.1.4 COOLING. Conduct all cooling operations until complete cooling is attained, and in any case for not less than 2 hours with the desiccator beside the balance.

1.7.1.5 WEIGHING. After cooling, complete the weighing of the weighing bottle or crucible within 2 minutes of its removal from the desiccator. Weigh to an accuracy of 0.0002 g.

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

1.7.2 *Procedure.* Take from the pre-treated laboratory test sample a test specimen weighing about 1 g. Cut yarn or dissected cloth into lengths of about 10 mm. Dry the specimen in a weighing bottle, cool it in a desiccator and weigh it. Transfer the specimen to the glass vessel specified in the appropriate part of this ISO Recommendation, reweigh the weighing bottle immediately, and obtain the dry mass of the specimen by difference.

Complete the test procedure as specified in the appropriate section of this ISO Recommendation, and examine the residue microscopically, or otherwise, as appropriate, to check that the treatment has in fact completely removed the soluble fibre.

1.8 Calculation and expression of results

Express the mass of the insoluble component as a percentage of the total mass of fibre in the mixture. Calculate the result on a clean dry mass basis as in clause 1.8.1, or on clean dry mass with agreed percentage additions for moisture as in clause 1.8.2, or on clean dry mass with agreed percentage additions for moisture and

(*a*) fibrous matter removed in the pre-treatment, and

(*b*) non-fibrous matter as in clause 1.8.3.

Obtain the percentage of the soluble component by difference. State which of the calculation procedures has been used and, if it is the second or third, state the values of the percentage additions.

1.8.1 *Method based on clean dry mass*

$$P = \frac{100\, m_1\, d}{m_0}$$

where

P is the percentage of clean dry insoluble component;

m_0 is the dry mass of the specimen;

m_1 is the dry mass of the residue;

d is the correction factor of variation in mass of the insoluble component in the reagent. Suitable values of d are given in the appropriate sections of this ISO Recommendation.

1.8.2 *Method based on clean dry mass with percentage additions for moisture*

$$P_M = \frac{100\, P\,(1 + 0.01\, a_2)}{P\,(1 + 0.01\, a_2) + (100 - P)\,(1 + 0.01\, a_1)}$$

where

P_M is the percentage of clean insoluble component with percentage additions for moisture;

P is the percentage of clean dry insoluble component;

a_1 is the percentage addition to the soluble component for moisture;

a_2 is the percentage addition to the insoluble component for moisture.

1.8.3 *Method based on clean dry mass with percentage additions for moisture and*

(*a*) *fibrous matter removed in the pre-treatment, and*

(*b*) *non-fibrous matter*

$$P_A = \frac{100\, P\,[1 + 0.01\,(a_2 + b_2)]}{P\,[1 + 0.01\,(a_2 + b_2)] + (100 - P)\,[1 + 0.01\,(a_1 + b_1)]}$$

where

P_A is the percentage of clean insoluble component in the mixture with percentage additions for moisture and non-fibrous matter;

P is the percentage of clean dry insoluble component;

a_1 is the percentage addition to the soluble component for moisture;

a_2 is the percentage addition to the insoluble component for moisture;

b_1 is the percentage loss of soluble fibrous matter caused by the pre-treatment, and/or the percentage addition to the soluble component for non-fibrous matter;

b_2 is the percentage loss of insoluble fibrous matter caused by the pre-treatment, and/or the percentage addition to the insoluble component for non-fibrous matter.

The percentage of the second component (P_{2A} %) is equal to $100 - P_A$ %.

Where a special pre-treatment has been used, the values of b_1 and b_2 should be determined, if possible, by submitting each of the pure fibre constituents to 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.

1.9 Precision of the methods

The precision indicated in individual methods relates to the reproducibility. This 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 on specimens of an identical, consistent mixture.

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

By this is meant that the difference between two results in a series of analyses made in different laboratories would be exceeded only in 5 cases out of 100, when the standard method is applied to an identical, consistent mixture.

1.10 Test report

The test report should include the following particulars :

(*a*) reference to this ISO Recommendation;

(*b*) whether the result relates to the overall composition of the material or to an individual component of the assembly;

(*c*) details of any special treatment for the removal of size or finish given in addition to the specified pre-treatment;

(*d*) the individual results and the arithmetic mean, each to an accuracy of 0.1;

(*e*) whether the result is based on

(1) clean dry mass;

(2) clean dry mass with percentage additions for moisture, giving the values of the percentage additions;

(3) clean dry mass with percentage additions for moisture and loss of fibrous matter caused by the pre-treatment, giving the values of the percentage additions;

(4) clean dry mass with percentage additions for moisture and non-fibrous matter, giving the values of the percentage additions.

2. MIXTURES OF ACETATE AND CERTAIN OTHER FIBRES

2.1 Field of application

This method is applicable, after removal of non-fibrous matter, to binary mixtures of acetate with wool, silk, regenerated protein, cotton (scoured, kiered, or bleached), cupro, viscose, modal, polyamide, polyester, acrylic and glass fibres. It is not applicable to mixtures containing modacrylic fibres, nor to mixtures containing viscose fibres that have been deactylated on the surface.

2.2 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 acetate is found by difference.

2.3 Additional reagent

Acetone, distilling between 55 and 57 °C.

2.4 Additional apparatus

Conical flask, minimum capacity 200 ml, glass-stoppered.

2.5 Test procedure

Follow the procedure described in clause 1.7.2 and then proceed as follows :

To the specimen contained in the conical flask (2.4) add 100 ml of acetone (2.3) per gramme of specimen, shake the flask, allow it to stand for 30 minutes at room temperature 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. Wash the residue into the filter crucible with acetone, and drain with suction. Refill the crucible with acetone and allow it to drain under gravity. Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

2.6 Calculation and expression of results

Calculate the results as described in clause 1.8. The value of d is 1.00.

2.7 Precision

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

3. MIXTURES OF CERTAIN PROTEIN AND CERTAIN OTHER FIBRES

3.1 Field of application

This method is applicable, after removal of non-fibrous matter, to binary mixtures of certain non-protein fibres and one protein fibre, as follows :

– wool, chemically treated wool, raw and degummed silk, raw and bleached tussah silk, mohair, cashmere, regenerated protein fibres based on casein,

mixed with :

– cotton, cupro, viscose, modal, acrylic, chlorofibres, polyamide, polyester, polypropylene and glass.

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

3.2 Principle

The protein fibre is dissolved out from a known dry mass of the mixture with alkaline sodium hypochlorite. 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 protein fibre is found by difference.

3.3 Additional reagents

3.3.1 *Hypochlorite reagent.* 1 M sodium hypochlorite solution to which has been added a sufficient quantity of sodium hydroxide to bring the concentration of sodium hydroxide to 5 g/l. The solution may be standardized iodometrically but its concentration is not critical within the range 0.9 M to 1.1 M.

3.3.2 *Acetic acid,* dilute solution. Dilute 5 ml of glacial acetic acid to 1 litre with water.

3.4 Additional apparatus

Glass beaker, minimum capacity 250 ml.

3.5 Test procedure

Follow the procedure described in clause 1.7.2 and then proceed as follows :

To the specimen contained in the glass beaker (3.4) add 100 ml of hypochlorite reagent (3.3.1) per gramme of specimen, stir vigorously to wet out the specimen and leave for 30 minutes, stirring vigorously at intervals. Filter the contents of the beaker through the weighed filter crucible and transfer any residual fibres to the crucible by washing out the beaker with a little hypochlorite reagent. Drain the crucible with suction and wash the residue successively with water, dilute acetic acid (3.3.2), and finally water, draining the crucible with suction after each addition. (Do not apply suction until each washing liquor has drained under gravity.) Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

3.6 Calculation and expression of results

Calculate the results as described in clause 1.8. The value of d is 1.00 except for raw cotton, for which $d = 1.03$.

3.7 Precision

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

4. MIXTURES OF VISCOSE OR CUPRO AND COTTON FIBRES USING SODIUM ZINCATE

4.1 Field of application

This method is applicable, after removal of non-fibrous matter, to binary mixtures of viscose or most of the current cupro and modal fibres with raw, scoured, kiered, or bleached cotton. If a cupro or modal fibre is found to be present, a preliminary test should be carried out to see whether it is soluble in the reagent. The method is not applicable to mixtures in which the cotton has suffered extensive chemical degradation, nor when the viscose, cupro or modal fibre is rendered incompletely soluble by the presence of certain permanent finishes or reactive dyes that cannot be removed completely.

4.2 Principle

The viscose, cupro or modal fibre is dissolved from a known dry mass of the mixture, with sodium zincate solution. 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 viscose, cupro or modal fibre is found by difference.

4.3 Additional reagents

4.3.1 *Sodium zincate (stock solution).* Determine the NaOH content of sodium hydroxide pellets and dissolve the equivalent of 180 g of NaOH in 180 to 200 ml of water. Stir the solution continuously with a mechanical stirrer (4.4.2), and add gradually 80 g of zinc oxide of analytical reagent quality, at the same time gradually heating the solution. When all the zinc oxide has been added, heat the solution until it boils gently; continue boiling the solution until it becomes clear or only slightly turbid, then cool it, add 20 ml of water, stir thoroughly, cool to room temperature, and make up to 500 ml with water in a graduated flask. Filter the solution through a sintered glass filter, with pore size 40 to 90 μm, before use.

4.3.2 *Sodium zincate, dilute solution (working solution).* To 1 volume (accurately measured) of stock sodium zincate solution (4.3.1) add, while stirring, 2 volumes of water. Mix thoroughly, and use within 24 hours of preparation.

4.3.3 *Ammonia,* dilute solution. Dilute 200 ml of concentrated ammonia solution (relative density 0.880) to 1 litre with water.

4.3.4 *Acetic acid,* dilute solution. Dilute 50 ml of glacial acetic acid to 1 litre with water.

4.4 Additional apparatus

4.4.1 *Mechanical shaker.*

4.4.2 *Mechanical stirrer.*

4.4.3 *Conical flask,* minimum capacity 500 ml, glass-stoppered.

4.5 Test procedure

Follow the procedure described in clause 1.7.2 and then proceed as follows :

To the specimen contained in the conical flask (4.4.3) add 150 ml of freshly prepared dilute sodium zincate solution (4.3.2) per gramme of specimen.

Insert the stopper and shake the flask vigorously on the mechanical shaker for 20 ± 1 minutes. Filter the contents of the flask through the weighed filter crucible. Apply suction to the crucible to remove excess liquor, again replace the residue in the flask by means of forceps, add 100 ml of ammonia solution (4.3.3), and shake the flask for 5 minutes on a mechanical shaker (4.4.1). Filter the contents of the flask through the same weighed filter crucible and wash any fibres from the flask into the crucible with water. Wash the crucible and residue with 100 ml of acetic acid solution (4.3.4) and then thoroughly with water. (Do not apply suction until each washing liquor has drained under gravity.) Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

4.6 Calculation and expression of results

Calculate the results as described in clause 1.8. The value of d for raw, scoured, kiered, or bleached cotton is 1.02.

5. MIXTURES OF VISCOSE OR CERTAIN TYPES OF CUPRO OR MODAL AND COTTON FIBRES USING FORMIC ACID AND ZINC CHLORIDE

5.1 Field of application

This method is applicable, after removal of non-fibrous matter, to binary mixtures of viscose or most of the current cupro and modal fibres with raw, scoured, kiered, or bleached cotton. If a cupro or modal fibre is found to be present, a preliminary test should be carried out to see whether it is soluble in the reagent. The method is not applicable to mixtures in which the cotton has suffered extensive chemical degradation, nor when the viscose, cupro or modal fibre is rendered incompletely soluble by the presence of certain permanent finishes or reactive dyes that cannot be removed completely.

5.2 Principle

The viscose, cupro or modal fibre is dissolved out from a known dry mass of the mixture, with a reagent composed 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 viscose, cupro or modal fibre is found by difference.

5.3 Additional reagents

5.3.1 *Formic acid/zinc chloride reagent.* Prepare a solution containing 20 g of anhydrous zinc chloride and 68 g of anhydrous formic acid, made up to 100 g with water.

NOTE. – The harmful effects of this reagent should be borne in mind, and full precautions should be taken in its use.

5.3.2 *Ammonia,* dilute solution. Dilute 200 ml of concentrated ammonia solution (relative density 0.880) to 1 litre with water.

5.4 Additional apparatus

5.4.1 *Conical flask,* minimum capacity 200 ml, glass-stoppered.

5.4.2 *Heating apparatus* suitable for maintaining the temperature of the flask at 40 ± 2 °C.

5.5 Test procedure

Follow the procedure described in clause 1.7.2 and then proceed as follows :

Place the specimen without delay in the conical flask (5.4.1) pre-heated to 40 °C. Add 100 ml of formic acid/zinc chloride reagent (5.3.1) per gramme of specimen, pre-heated to 40 °C. Stopper the flask and shake it. Allow the flask and contents to remain at 40 °C for 2 hours, shaking it twice during this time at intervals of 45 minutes.

Filter the contents of the flask through the weighed filter crucible and wash any fibres from the flask into the crucible with the reagent. Rinse with a further 20 ml of reagent. Wash the crucible and residue thoroughly with water at 40 °C. Rinse with 100 ml of cold ammonia solution (5.3.2) and then thoroughly with cold water. (Do not apply suction until each washing liquor has drained under gravity.) Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

5.6 Calculation and expression of results

Calculate the results as described in clause 1.7.1. The value of d for raw cotton is 0.98 and for scoured, kiered or bleached cotton is 0.96.

5.7 Precision

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

6. MIXTURES OF NYLON 6 OR NYLON 6.6 AND CERTAIN OTHER FIBRES

6.1 Field of application

This method is applicable after removal of non-fibrous matter, to binary mixtures of nylon 6 or nylon 6.6 with cotton, viscose, cupro, modal, polyester, polypropylene, chlorofibre, acrylic or glass fibre. It is applicable also to mixtures with wool, but when the wool content exceeds 25 %, the method described in section 3 should be used.

6.2 Principle

The polyamide is dissolved out from a known dry mass of the mixture, with aqueous 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 nylon 6 or nylon 6.6 fibre is found by difference.

6.3 Additional reagents

6.3.1 *Formic acid,* 80 % (m/m) (relative density 1.186). Dilute 880 ml of 90 % (m/m) formic acid (relative density 1.204) to 1 litre with water. Alternatively, dilute 780 ml of 98 to 100 % (m/m) formic acid (relative density 1.220) to 1 litre with water. The concentration is not critical within the range 77 to 83 % (m/m) formic acid.

6.3.2 *Ammonia,* dilute solution. Dilute 80 ml of concentrated ammonia solution (relative density 0.880) to 1 litre with water.

6.4 Additional apparatus

Conical flask, minimum capacity 200 ml, glass-stoppered.

6.5 Test procedure

Follow the procedure described in clause 1.7.2 and then proceed as follows :

To the specimen contained in the conical flask (6.4) add 100 ml of formic acid (6.3.1) per gramme of specimen, insert the stopper, shake the flask to wet out the specimen and allow the flask to stand for 15 minutes, shaking it at intervals. Filter the contents of the flask through a weighed filter crucible and transfer any residual fibres to the crucible by washing out the flask with a little more formic acid (6.3.1). Drain the crucible with suction and wash the residue on the filter successively with formic acid (6.3.1), hot water, dilute ammonia solution (6.3.2), 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.

6.6 Calculation and expression of results

Calculate the results as described in clause 1.8. The value of d is 1.00.

6.7 Precision

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

7. MIXTURES OF ACETATE AND TRIACETATE FIBRES USING ACETONE

7.1 Field of application

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

7.2 Principle

The acetate fibre is dissolved out from a known dry mass of the mixture, with aqueous acetone. 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 acetate is found by difference.

7.3 Additional reagent

Acetone, aqueous solution, 70 % (V/V). Dilute 700 ml of acetone to 1 litre with water.

7.4 Additional apparatus

7.4.1 *Conical flask*, minimum capacity 200 ml, glass-stoppered.

7.4.2 *Mechanical shaker.*

7.5 Test procedure

Follow the procedure described in clause 1.7.2 and then proceed as follows :

To the specimen contained in the conical flask (7.4.1) add 80 ml of aqueous acetone (7.3) per gramme of specimen, shake the flask for 1 hour on a mechanical shaker, and then decant the liquid through the weighed filter crucible. Add 60 ml of aqueous acetone to the residue in the flask, shake by hand and decant the liquid through the filter crucible. Repeat this treatment twice more, on the last occasion transferring the fibres to the crucible. Wash any residual fibres into the crucible with aqueous acetone and drain with suction. Refill the crucible with aqueous acetone and allow it to drain under gravity. Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

7.6 Calculation and expression of results

Calculate the results as described in clause 1.8. The value of d is 1.01.

8. MIXTURES OF ACETATE AND TRIACETATE FIBRES USING BENZYL ALCOHOL

8.1 Field of application

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

8.2 Principle

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

8.3 Additional reagents

8.3.1 *Benzyl alcohol.*

8.3.2 *Ethyl ether.*

8.4 Additional apparatus

8.4.1 *Conical flask,* minimum capacity 200 ml, glass-stoppered.

8.4.2 *Mechanical shaker.*

8.4.3 *Heating apparatus* suitable for maintaining the temperature of the flask at 52 ± 2 °C (for example, water bath with thermostat).

8.5 Test procedure

Follow the procedure described in clause 1.7.2 and then proceed as follows :

To the specimen contained in the conical flask (8.4.1) add 100 ml of benzyl alcohol per gramme of specimen. Insert the stopper, and shake the flask vigorously on the mechanical shaker so that it plunges into the water bath, kept at a temperature of 52 ± 2 °C. Shake the flask 20 ± 1 minutes at this temperature. Filter the contents of the flask through the weighed filter crucible. Replace the residue in the flask by means of forceps, add to the flask a fresh portion of benzyl alcohol and shake as before at a temperature of 52 ± 2 °C for 20 ± 1 minutes. Filter the contents of the flask through the same weighed filter crucible and repeat the cycle a third time with a third 100 ml portion of benzyl alcohol. Pour the liquid and the residue into the same weighed filter crucible; wash any fibres from the flask into the crucible with an extra quantity of benzyl alcohol at a temperature of 52 ± 2 °C. Drain the crucible with suction.

Transfer the fibres into a flask, rinse with ethyl ether, and after manual shaking, decant through the same filter crucible. Repeat this rinsing operation three times. Transfer the residue into the same filter crucible. Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

8.6 Calculation and expression of results

Calculate the results as described in clause 1.8. The value of d is 1.00.

8.7 Precision

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

9. MIXTURES OF TRIACETATE AND CERTAIN OTHER FIBRES

9.1 Field of application

This method is applicable, after removal of non-fibrous matter, to binary mixtures of triacetate with wool, regenerated protein, cotton (scoured, kiered, or bleached), viscose, cupro, modal, polyamide, polyester, acrylic and glass fibres. The method is only applicable to triacetate fibres partially hydrolysed by a special finish when a suitable additional factor is used.

9.2 Principle

The triacetate fibre is 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 triacetate is found by difference.

9.3 Additional reagent

Dichloromethane (methylene chloride).

NOTE. – The toxic effects of this reagent should be borne in mind, and full precautions should be taken in its use.

9.4 Additional apparatus

Conical flask, minimum capacity 200 ml, glass-stoppered.

9.5 Test procedure

Follow the procedure described in clause 1.7.2 and then proceed as follows :

To the specimen contained in the conical flask (9.4), add 100 ml of dichloromethane (9.3) per gramme of specimen, insert the stopper, shake the flask to wet out the specimen, and allow the flask to stand for 30 minutes, shaking it at intervals of about 10 minutes. Decant the liquid through the weighed filter crucible. Add 60 ml of dichloromethane to the residue in the flask, shake it by hand, and filter the contents of the flask through the filter crucible. Transfer any residual fibres to the crucible by washing out the flask with a little more dichloromethane. Drain the crucible with suction, refill the crucible with dichloromethane, and allow it to drain under gravity. Finally, drain the crucible with suction.

Dry the crucible and residue, and cool and weigh them.

9.6 Calculation and expression of results

Calculate the results as described in clause 1.8. The value of d is 1.00 except for polyester fibre, for which $d = 1.01$.

NOTE. – In the case of triacetate that is not completely soluble in the reagent, the percentage triacetate after calculation in the normal manner should be multiplied by 1.02. The percentage triacetate thus calculated should be deducted from 100 to give the percentage of the other fibre.

9.7 Precision

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

10. MIXTURES OF CELLULOSE AND POLYESTER FIBRES

10.1 Field of application

This method is applicable, after removal of non-fibrous matter, to mixtures of natural and regenerated cellulose fibres and polyester fibre.

10.2 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 cellulose fibre is found by difference.

10.3 Additional reagents

10.3.1 *Sulphuric acid,* 75 % (m/m). A suitable reagent may be prepared by adding carefully, while cooling, 700 ml of concentrated sulphuric acid (relative density 1.84) to 350 ml of distilled water. After the solution has cooled to room temperature, dilute it to 1 litre with water. The concentration is not critical within the range 73 to 77 % (m/m) sulphuric acid.

10.3.2 *Ammonia,* dilute solution. Dilute 80 ml of concentrated ammonia solution (relative density 0.880) to 1 litre with water.

10.4 Additional apparatus

10.4.1 *Conical flask,* minimum capacity 500 ml, glass-stoppered.

10.4.2 *Heating apparatus* suitable for maintaining the temperature of the flask at 50 ± 5 °C.

10.5 Test procedure

Follow the procedure described in clause 1.7.2 and then proceed as follows :

To the specimen contained in the conical flask (10.4.1) add 200 ml of sulphuric acid (10.3.1) per gramme of specimen, insert the stopper, and shake the flask carefully to wet out the specimen. Maintain the flask at 50 ± 5 °C for 1 hour, shaking the flask and contents gently at intervals of about 10 minutes. Filter the contents of the flask through the weighed filter crucible by means of suction. Transfer any residual fibres to the crucible by washing out the flask with a little more sulphuric acid (10.3.1). Drain the crucible with suction, and wash the residue on the filter once by filling the crucible with a fresh portion of sulphuric acid (10.3.1). Do not apply suction until the crucible has drained under gravity or stood for 1 minute. Wash the residue successively several times with cold water, twice with dilute ammonia solution (10.3.2), and then thoroughly with cold water, draining the crucible with suction after each addition. (Do not apply suction until each washing liquor has drained under gravity.) Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

10.6 Calculation and expression of results

Calculate the results as described in clause 1.8. The value of d is 1.00.

10.7 Precision

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