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Textiles — Quantitative chemical analysis —

Part 1: General principles of testing

Textiles — Analyses chimiques quantitatives —

Partie 1: Principes généraux des essais

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Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1833-1 was prepared by Technical Committee ISO/TC 38, *Textiles*.

This first edition of ISO 1833-1 cancels and replaces ISO/TR 5090:1977 and partially revises ISO 1833:1977.

ISO 1833:1977 will be cancelled and replaced by ISO 1833-1, ISO 1833-3, ISO 1833-4, ISO 1833-5, ISO 1833-6, ISO 1833-7, ISO 1833-8, ISO 1833-9, ISO 1833-10, ISO 1833-11, ISO 1833-12, ISO 1833-13, ISO 1833-14, ISO 1833-15, ISO 1833-16, ISO 1833-17, ISO 1833-18 and ISO 1833-19.

ISO 1833 consists of the following parts, under the general title *Textiles — Quantitative chemical analysis*:

— *Part 1: General principles of testing*

— *Part 2: Ternary fibre mixtures*

— *Part 3: Mixtures of acetate and certain other fibres (method using acetone)*

— *Part 4: Mixtures of certain protein and certain other fibres (method using hypochlorite)*

— *Part 5: Mixtures of viscose, cupro or modal and cotton fibres (method using sodium zincate)*

— *Part 7: Mixtures of polyamide and certain other fibres (method using formic acid)*

— *Part 8: Mixtures of acetate and triacetate fibres (method using acetone)*

— *Part 9: Mixtures of acetate and triacetate fibres (method using benzyl alcohol)*

— *Part 10: Mixtures of triacetate or polylactide and certain other fibres (method using dichloromethane)*

— *Part 11: Mixtures of cellulose and polyester fibres (method using sulfuric acid)*

— *Part 12: Mixtures of acrylic, certain modacrylics, certain chlorofibres, certain elastanes and certain other fibres (method using dimethylformamide)*

— *Part 13: Mixtures of certain chlorofibres and certain other fibres (method using carbon disulfide/acetone)*

— *Part 14: Mixtures of acetate and certain chlorofibres (method using acetic acid)*

— *Part 15: Mixtures of jute and certain animal fibres (method by determining nitrogen content)*

— *Part 16: Mixtures of polypropylene fibres and certain other fibres (method using xylene)*

— *Part 17: Mixtures of chlorofibres (homopolymers of vinyl chloride) and certain other fibres (method using sulfuric acid)*

— *Part 18: Mixtures of silk and wool or hair (method using sulfuric acid)*

— *Part 19: Mixtures of cellulose fibres and asbestos (method by heating)*

— *Part 21: Mixtures of chlorofibres, certain modacrylics, certain elastanes, acetates, triacetates and certain other fibres (method using cyclohexanone)*

The following parts are under preparation:

— *Part 6: Mixtures of viscose or certain types of cupro or modal or lyocell and cotton fibres (method using formic acid and zinc chloride)*

— *Part 20: Mixtures of elastane and certain other fibres (method using dimethylacetamide)*

— *Part 22: Mixtures of viscose or certain types of cupro or modal or lyocell and flax fibres (method using formic acid and zinc chlorate)*

— *Part 23: Mixtures of polyethylene and polypropylene (method using cyclohexanone)*

— *Part 24: Mixtures of polyester and some other fibres (method using phenol and tetrachloroethane)*

Introduction

In general, the methods described in the different parts of ISO 1833 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. This part of ISO 1833 gives the information which is common to the analyses, by this method, of all fibre mixtures, whatever their composition. This information should be used in conjunction with the other parts of ISO 1833; these parts 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 part.

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 should be removed before analysis. A method of pre-treatment for removing oils, fats, waxes and water-soluble matter is given in Annex A of this part of ISO 1833.

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 this part of ISO 1833. The result is therefore obtained on the basis of clean, dry fibres.

Provision is made for recalculating the result on the basis of

a) agreed allowances for moisture content[1)],

b) agreed allowances for moisture and also for

   1) fibrous matter removed in the pre-treatment, and

   2) 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.

1) The values to use are the conventional conditioning rates for the respective fibres, when rates exist.

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 that use be made of 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, the method described in Annex B should be used in preference to the chemical methods of analysis given in the individual parts of ISO 1833.

Textiles — Quantitative chemical analysis —

Part 1: General principles of testing

1 Scope

This part of ISO 1833 specifies a common method for the quantitative chemical analysis of various binary mixtures of fibres. This method and the methods described in the other parts of ISO 1833 are applicable, in general, to fibres in any textile form. Where certain textile forms are excepted, these are listed in the scope of the appropriate part.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5089, *Textiles — Preparation of laboratory test samples and test specimens for chemical testing*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
non-fibrous matter
processing aids such as lubricants and sizes (but excluding jute-batching oils), and naturally occurring non-fibrous substances

4 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 proportions of soluble component are calculated from the loss in mass. Where relevant, the fibre in the larger proportion is removed first.

5 Reagents

Use only reagents of recognized analytical grade.

5.1 Light petroleum, re-distilled, distilling between 40 °C and 60 °C.

5.2 Distilled or deionized water.

6 Apparatus

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

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

NOTE In place of a glass filter crucible, any other apparatus giving identical results may be used.

6.2 Vacuum flask.

6.3 Desiccator containing self-indicating silica gel.

6.4 Ventilated oven for drying specimens at (105 ± 3) °C.

6.5 Analytical balance with a resolution of 0,000 2 g or better.

6.6 Soxhlet extraction apparatus, of sufficient size to give a volume, in millilitres, equal to 20 times the mass, in grams, of the specimen, or any other apparatus giving identical results.

7 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.

8 Sampling and pre-treatment of sample

8.1 Sampling

Take a laboratory test sample, as described in ISO 5089, 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 should be taken of this fact in the sampling of the fabric. Treat the sample as described in 8.2

8.2 Pre-treatment of laboratory test sample

Extract the air-dry sample in a Soxhlet apparatus with light petroleum for 1 h at a minimum rate of six cycles per hour.

Allow the light petroleum to evaporate from the sample. Soak the specimen in cold water for 1 h, and then in water at (65 ± 5) °C for a further 1 h. 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 shall 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 contains finishes which are insoluble in both light petroleum and water.

9 Procedure

9.1 General instructions

9.1.1 Drying

Conduct all drying operations for not less than 4 h and not more than 16 h at (105 ± 3) °C in a ventilated oven with the oven door closed throughout.

NOTE The specimen should be dried until constant mass is achieved.

9.1.2 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.

9.1.3 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.

9.1.4 Cooling

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

9.1.5 Weighing

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

Weigh to an accuracy of 0,000 2 g.

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

9.2 Testing execution

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 ISO 1833, reweigh the weighing bottle immediately, and obtain the dry mass of the specimen by the difference.

Complete the test procedure as specified in the appropriate part of ISO 1833, and examine the residue microscopically, or otherwise, as appropriate, to check that the treatment has in fact completely removed the soluble fibre.

10 Calculation and expression of results

10.1 General

Express the mass of the insoluble component as a percentage of the total mass of fibre in the mixture.

Calculate the result based on

a) clean dry mass (as in 10.2), or

b) clean dry mass with agreed percentage additions for moisture (as in 10.3), or

c) clean dry mass with agreed percentage additions for moisture and percentage losses of fibrous matter removed in the pre-treatment (as in 10.4), and

d) clean dry mass with agreed percentage additions for moisture and non-fibrous matter (as in 10.4).

Obtain the percentage of the soluble component by difference. State which of the calculation procedures has been used and, in cases b), c) and d), state the values of the percentage additions.

10.2 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 insoluble component in the reagent.

Suitable values of d are given in the different parts of ISO 1833.

10.3 Method based on clean dry mass with percentage additions for moisture

$$P_{\mathrm{M}} = \frac{100 P\left(1 + 0{,}01 a_2\right)}{P\left(1 + 0{,}01 a_2\right) + \left(100 - P\right)\left(1 + 0{,}01 a_1\right)}$$

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.

10.4 Method based on clean dry mass with percentage additions for moisture and non-fibrous matter, and/or percentage losses of fibrous matter by pre-treatment

$$P_A = \frac{100P\left[1+0{,}01\left(a_2+b_2\right)\right]}{P\left[1+0{,}01\left(a_2+b_2\right)\right]+\left(100-P\right)\left[1+0{,}01\left(a_1+b_1\right)\right]}$$

where

P_A is the percentage of clean insoluble component 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.

11 Precision of the methods

The precision indicated in individual parts of ISO 1833 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 %, i.e. the difference between two results in a series of analyses made in different laboratories would be exceeded only in five cases out of 100, when the standard method is applied to an identical consistent mixture.

12 Test report

The test report shall specify the following:

a) reference to this part of ISO 1833 (ISO 1833-1:2006);

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 percentage losses 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.

Annex A
(informative)

Methods for the removal of non-fibrous matter

A.1 General

The removal of certain types of non-fibrous matter, particularly when more than one substance is present, may demand the exercise of considerable chemical resource, and each material to be treated for removal of its non-fibrous matter should be regarded as an individual problem. The procedures suggested in this annex do not pretend to be complete, and it should not be assumed that those described in this annex will have no effect on the physical and chemical properties of the textile materials concerned. Furthermore, these procedures are only applicable where the non-fibrous substances are known or can be identified with certainty.

For the purposes of this annex, dyes are not considered as non-fibrous matter but as an integral part of the textile, and are not, therefore, mentioned. Some prints are made with resin bonded pigments which cannot be regarded as part of the fibre substance. They involve a greater addition of mass to the fibre than dyes and it would be desirable to remove them, but it is rarely if ever possible to do so. Similarly, certain finishes cannot be removed. In the present state of knowledge, quantitative analysis cannot, therefore, be carried out with the degree of accuracy provided for by the test methods described in the different parts of ISO 1833.

It may be assumed that Soxhlet extraction under the conditions described in the annex will ensure adequate removal of oils, fat and waxes. With other non-fibrous substances, it is necessary, wherever possible, to check that removal is complete.

If the extraction in light petroleum as described in A.5.1 is conducted, it is not necessary to repeat this procedure as required by 8.2.

CAUTION — Since certain hazards are associated with reagents and solvents employed in the methods given below, these methods should be used only by persons acquainted with the hazards and the precautions to be taken.

A.2 Scope of this annex

This annex describes procedures for the removal of certain commonly found types of non-fibrous substances from fibres. Fibres to which the procedures are applicable and those to which they are not applicable are listed in Table A.1, in relation to the non-fibrous substances to be removed. The names of these fibres are defined in ISO 2076 or ISO 6938. Identification of the non-fibrous matter and of the fibres present is not covered by this annex.

In certain cases, the elimination of all the added matter is impracticable. The quantity remaining should not effect the quantitative analysis; on the other hand, it is essential to minimize the chemical degradation of the fibres.

A.3 Principle

Where possible, remove non-fibrous matter by a suitable solvent.

NOTE In many cases, the removal of certain finishes involves their chemical modification. In addition, chemical degradation of the fibre substance cannot always be avoided.

A.4 Apparatus

The apparatus required is part of the normal equipment of a chemical laboratory.

Table A.1 — Use of procedures for removal of non-fibrous matter

Non-fibrous matter to be removed	Fibres in the presence of which the procedure is *applicable*	Method		Fibres in the presence of which the procedure is *not applicable*
		Subclause	Reagents	
Oils, fats and waxes	Most fibres	**A.5.1**	Light petroleum, Soxhlet	Elastane
Soaking oils	Nett silk	**A.5.2**	Toluene/methanol, Soxhlet	—
Starch	Cotton [a] , linen [b)], viscose, spun silk, jute [c)] and most other fibres	**A.5.3**	Amylase then boiling water	—
Locust-bean gum and starch	Cotton [a)], viscose, spun silk	**A.5.4**	Boiling water then A.5.3	—
Tamarind seed size	Cotton [a)], viscose	**A.5.5**	Boiling water twice	—
Acrylic (size or finish)	Most fibres [d)]	**A.5.6**	2 g/l soap, 2 g/l NaOH, 70 °C to 75 °C, rinse in water	Protein, deacetylated acetate, acetate, triacetate, acrylic, modacrylic
Gelatine and polyvinyl alcohol	Most fibres	**A.5.7**	1 g/l non-ionic surfactant, 1 g/l anionic surfactant, 1 g/l Na_2C0_3	Protein, deacetylated acetate, acetate, triacetate
Starch and polyvinyl alcohol	Cotton, polyester	**A.5.8**	A.5.3 followed by A.5.7	Protein, deacetylated acetate, acetate, triacetate
Polyvinyl acetate	Most fibres	**A.5.9**	Acetone, Soxhlet	Deacetylated acetate, acetate triacetate, chlorofibre
Linseed oil sizes	Viscose crêpe yarns	**A.5.10**	A.5.1 followed by A.5.7	Protein, deacetylated acetate, acetate, triacetate
Amino-formaldehyde resins	Cotton, cupro, viscose, modal, deacetylated acetate, acetate, triacetate, polyester, polyamide (nylon)	**A.5.11**	Orthophosphoric acid/urea, 80 °C, 10 min, rinse in water, then $NaHC0_3$	Asbestos
Bitumen, creosote and tar	Most fibres	**A.5.12**	Dichloromethane (methylene chloride), Soxhlet	Deacetylated acetate, acetate, triacetate, modacrylic, chlorofibre
Cellulose ethers	Most fibres	**A.5.13.1**	Soak in cold water	—
	Cotton	**A.5.13.2**	175 g/l NaOH solution at 10 °C, neutralized in 0,1 N acetic acid	Viscose, deacetylated acetate, triacetate, modacrylic, acrylic
Cellulose nitrate	Most fibres	**A.5.14**	Soaking in acetone, 1 h	Deacetylated acetate, acetate, triacetate
Polyvinyl chloride	Most fibres	**A.5.15**	Soaking in tetrahydrofuran (do not recover by distillation)	Deacetylated acetate, acetate, triacetate, chlorofibre

Table A.1 *(continued)*

Non-fibrous matter to be removed	Fibres in the presence of which the procedure is *applicable*	Method		Fibres in the presence of which the procedure is *not applicable*
		Subclause	Reagents	
Oleates	Most fibres	**A.5.16**	0,2 N HCl, extraction in dichloromethane, Soxhlet	Deacetylated acetate, acetate, triacetate, modacrylic, chlorofibre, polyamide (nylon), asbestos
Oxides of chromium, iron and copper	Cupro, viscose, modal, deacetylated acetate, acetate, triacetate	**A.5.17**	14 g/l hydrated oxalic acid at 80 °C, neutralize with NH_4OH	—
Pentachlorophenyl laurate	Most fibres	**A.5.18**	Toluene, Soxhlet	Polyethylene, polypropylene
Polyethylenes	Most fibres	**A.5.19**	Boiling toluene under reflux	Polypropylene
Polyurethanes	Polyamide (nylon), cupro, viscose, modal, deacetylated acetate, acetate, triacetate	**A.5.20**	Dimethyl sulfoxide or dichloromethane, if possible 50 g/l NaOH, ethanol at 50 °C	Deacetylated acetate, acetate, triacetate, polyester, acrylic, modacrylic
Natural rubber and styrene-butadiene, neoprene, nitrile	Cupro, viscose, modal, deacetylated acetate, acetate, triacetate, glass	**A.5.21**	Swell in benzene, scrape, heat in molten *p*-dichlorobenzene, *tert*-butyl hydroperoxide per 4 parts of *p*-dichlorobenzene, cool to 60 °C, add benzene	All synthetic fibres
Silicones	Most fibres	**A.5.22**	Hydrofluoric acid, 50 ml to 60 ml per litre, 65 °C	Polyamide (nylon), glass
Tin weighting	Silk	**A.5.23**	0,5 N hydrofluoric acid	—
Wax-based waterproof finishes	Cotton, protein, polyester, polyamide (nylon)	**A.5.24**	Dichloromethane, Soxhlet, if metallic complex: 10 g/l formic acid and 5 g/l acid stable surfactant	Deacetylated acetate, acetate, triacetate, modacrylic, chlorofibre

[a] Grey cotton loses mass when treated by this method. The loss amounts to approximately 3 % of the final oven-dry mass.

[b] Linen loses mass when treated by this method. The loss depends on the types of the yarn from which the fabric is produced. Losses are approximately as follows: bleached yarns 2 %, boiled yarns 3 % and grey yarns 4 %.

[c] Jute loses mass by approximately 0,5 % when treated by this method.

[d] Polyamide 6.6 (nylon 6.6) may undergo a loss in mass of fibre substance of up to 1 % when treated by this method. The loss in mass of polyamide 6 (nylon 6) may vary between 1 % and 3 %.

A.5 Procedures

A.5.1 Soaking oils using light petroleum

Extract the specimen is a Soxhlet apparatus or similar apparatus with light petroleum (distilling between 40 °C and 60 °C) for at least 1 h at a minimum rate of six cycles per hour. This is the same as the first part of the pre-treatment required by 8.2.

A.5.2 Soaking oils using a mixture of toluene and methanol

Extract the specimen in a Soxhlet apparatus or similar apparatus with a mixture of 1 volume of toluene with 3 volumes of methanol as the solvent, for at least 2 h at a minimum rate of six cycles per hour.

NOTE There is an accepted method for the removal of soaking oils from silk that includes benzene but because of the toxic properties of benzene the above method is suggested.

A.5.3 Starch

Immerse the specimen in a freshly prepared solution containing 0,1 % (mass fraction) of a non-ionic wetting agent together with an appropriate amylase preparation, using a liquor/specimen ratio of 100/1. The concentration of the amylase preparation and the pH, temperature and time of treatment should be those recommended by the manufacturer. Transfer the specimen to boiling water and boil it for 15 min. Test for complete removal of starch using a dilute aqueous solution of iodine in potassium iodide. When all the starch is removed, rinse the specimen thoroughly in water, squeeze or mangle it, and dry it.

A.5.4 Locust-bean gum and starch

Boil the specimen in water for 5 min, using a liquor/specimen ratio of 100/1. Repeat this procedure with a fresh portion of water. Follow this by the procedure described in A.5.3

A.5.5 Tamarind seed size

Boil the specimen in water for 5 min, using a liquor/specimen ratio of 100/1. Repeat this procedure with a fresh portion of water.

NOTE Size prepared from coarsely ground undecorticated tamarind seed powder may not be completely removed by this procedure.

A.5.6 Acrylic size

Immerse and agitate the specimen for 30 min in at least 100 times its own mass of a solution containing 2 g/l of soap or other suitable detergent and 2 g/l of sodium hydroxide at 70 °C to 75 °C. Give three 5 min rinses in distilled water at 85 °C, squeeze, mangle or centrifuge, and dry the specimen.

A.5.7 Gelatine and polyvinyl alcohol

Treat the specimen in a solution (using a minimum liquor/specimen ratio of 100/1) containing 1 g/l of non-ionic surfactant, 1 g/l of anionic surfactant, and 1 g/l of anhydrous sodium carbonate, for 90 min at 50 °C followed by 90 min in the same bath at 70 °C to 75 °C. Wash the specimen and dry it.

A.5.8 Starch and polyvinyl alcohol

Conduct the procedure described in A.5.3, followed by the procedure described in A.5.7, with intermediate drying.

A.5.9 Polyvinyl acetate

Extract the specimen in a Soxhlet apparatus with acetone for at least 3 h at a minimum rate of six cycles per hour.

A.5.10 Linseed oil sizes

Conduct the procedure described in A.5.1, followed by the procedure described in A.5.7.

A.5.11 Amino-formaldehyde resins

Extract the specimen with a solution of 25 g/l of 50 % orthophosphoric acid and 50 g/l of urea at 80 ° for 10 min, using a liquor/specimen ratio of 100/1. Wash the specimen in water, drain, wash it in a 0,1 % sodium bicarbonate solution, and finally wash it thoroughly in water.

NOTE This method causes some damage to cupro, viscose, modal, deacetylated acetate, acetate and triacetate.

A.5.12 Bitumen, creosote and tar

Extract the specimen with dichloromethane (methylene chloride) in a Soxhlet apparatus. The duration of treatment depends on the amount of non-fibrous matter present, and it may be necessary to renew the solvent.

NOTE Extraction of jute with dichloromethane will also remove the batching oil, which may be present to the extent of 5 % or more.

A.5.13 Cellulose ethers

A.5.13.1 Methyl cellulose soluble in cold water

Soak the specimen in cold water for 2 h. Rinse the specimen repeatedly in cold water, with vigorous squeezing.

A.5.13.2 Cellulose ethers insoluble in water but soluble in alkali

Immerse the specimen for 30 min in a solution containing approximately 175 g/l of sodium hydroxide cooled to a temperature of 5 °C to 10 °C. Then work the specimen thoroughly in a fresh portion of reagent, rinse it well in water, neutralize it with approximately 0,1 N acetic acid, rinse it again in water, and dry it.

A.5.14 Cellulose nitrate

Immerse the specimen in acetone at room temperature for 1 h, using a liquor/specimen ratio of 100/1. Drain, wash the specimen in three portions of fresh acetone, and allow the entrained solvent to evaporate.

A.5.15 Polyvinyl chloride

Immerse the specimen in tetrahydrofuran at room temperature for 1 h, using a liquor/specimen ratio of 100/1. If necessary, scrape off the softened polyvinyl chloride. Drain, wash the specimen in three portions of fresh tetrahydrofuran, drain and allow the entrained solvent to evaporate.

CAUTION — Because of the risk of explosion, tetrahydrofuran should not be recovered by distillation.

A.5.16 Oleates

Immerse the specimen in approximately 0,2 N hydrochloric acid at ambient temperature until it is thoroughly wetted. Wash the specimen well and dry it. Extract the specimen in a Soxhlet apparatus with dichloromethane (methylene chloride) for 1 h at a minimum rate of six cycles per hour.

A.5.17 Oxides of chromium, iron and copper

NOTE This method is not applicable if dyes containing chromium have been applied to the material under test.

Immerse the specimen in a solution containing 14 g/l of hydrated oxalic acid at 80 °C for 15 min, using a liquor/specimen ratio of 100/1. Wash it thoroughly (any copper present will remain as the colourless oxalate; remove this with 1 % acetic acid at 40 °C for 15 min and wash the specimen). Neutralize the specimen with ammonia and wash it thoroughly in water. Squeeze, mangle or centrifuge, and dry it.

A.5.18 Pentachlorophenyl laurate (PCPL)

Extract the specimen in a Soxhlet apparatus with toluene for 4 h at a minimum rate of six cycles per hour.

A.5.19 Polyethylenes

Extract the specimen in boiling toluene under reflux.

The material shall be completely immersed in the boiling solvent.

A.5.20 Polyurethanes

No completely satisfactory method is available but the following have been found suitable.

Some polyurethanes can be removed by dissolution in dimethyl sulfoxide or dichloromethane (methylene chloride), and subsequent repeated washing of the specimen with fresh quantities of solvent. When the fibre composition of the specimen permits, some polyurethanes can be removed by hydrolysis in a boiling aqueous solution containing 50 g/l of sodium hydroxide. Alternatively, an aqueous solution containing 50 g/l of sodium hydroxide and 100 g/l of ethanol may be used at a temperature above 50 °C.

CAUTION — Dimethyl sulfoxide has toxic properties.

A.5.21 Natural rubbers, and styrene-butadiene, neoprene, nitrile and most other synthetic rubbers

No completely satisfactory method is available but the following has been found useful.

Soak the specimen in a hot volatile solvent which swells it considerably (for example benzene), and when it is fully swollen remove as much of the rubber as possible by scraping. It may be possible in some cases, where the textile fibres are exposed, to wet only the textile face, and strip the rubber and textile layers apart almost at once. Continue by heating the residual specimen, with constant stirring, in 50 or more times its mass of molten *p*-dichlorobenzene; use a flat-bottomed flask with an attached wide-bore condenser (to allow adequate access of air), and preferably a magnetic stirrer and hot-plate.

After 45 min, add 1 part 70 % *tert*-butyl hydroperoxide per 4 parts *p*-dichlorobenzene present. Boil until decomposition of the rubber is complete (2 h is an average time). Cool the flask to about 60 °C and add an equal volume of benzene. Filter and wash the textile component repeatedly in warm benzene.

Nitrile rubber (for example, acrylonitrile-butadiene rubber) may require the addition of the same volume of nitrobenzene as of *tert*-butyl hydroperoxide to speed up the dissolution process.

NOTE 1 Natural rubber should dissolve after being boiled in *p*-dichlorobenzene alone for several hours in the presence of air. Dissolution may also be effected by heating in diphenyl ether at 150 °C to 160 °C for 2 h and then washing the specimen in benzene.

NOTE 2 The above treatments are strongly oxidative in character and the properties of the textile material may be affected appreciably.

A.5.22 Silicones

Scour the specimen in a solution containing 50 ml/l to 60 ml/l of 40 % hydrofluoric acid in a polyethylene vessel at 65 °C for 45 min. Thoroughly wash the specimen, neutralize it, and scour it in a solution containing 2 g/l of soap at 60 °C for 1 h.

WARNING — Hydrofluoric acid is a dangerous product.

A.5.23 Tin weighting

Immerse the specimen in 0,5 N hydrofluoric acid in a polyethylene vessel at 55 °C for 20 min, stirring occasionally. Rinse in warm water. Immerse the specimen in a 2 % solution of sodium carbonate at 55 °C for 20 min. Wash the specimen in warm water, squeeze, mangle or centrifuge, and dry it.

WARNING — Hydrofluoric acid is a dangerous product.

A.5.24 Wax-based waterproof finishes

Extract the specimen in a Soxhlet apparatus with dichloromethane (methylene chloride) for at least 3 h at a minimum of six cycles per hour. Then, to remove any metallic complexes, scour the specimen in a solution containing 10 g/l of formic acid and 5 g/l of acid stable surfactant at 80 °C for 15 min. Wash the specimen thoroughly in water until it is free from acid.

Annex B
(informative)

Method of quantitative analysis by manual separation

B.1 General

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

B.2 Principle

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

B.3 Apparatus

B.3.1 **Weighing bottle**, or any other apparatus giving identical results.

B.3.2 **Desiccator**, containing self-indicating silica gel.

B.3.3 **Ventilated oven** for drying specimens at (105 ± 3) °C.

B.3.4 **Analytical balance**, with a resolution of 0,000 2 g or better.

B.3.5 **Soxhlet extractor**, or other apparatus giving an identical result.

B.3.6 **Needle**.

B.3.7 **Twist tester**, or similar apparatus.

B.4 Reagents

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

B.4.2 **Distilled or deionized water**.

B.5 Conditioning and testing atmosphere

See Clause 7.

B.6 Laboratory test sample

See 8.1.

B.7 Pre-treatment of laboratory test sample

See 8.2.

B.8 Procedure

B.8.1 Analysis of yarn

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

Cut the yarn into pieces of a suitable length and separate the fibre types by means of a needle and, if necessary, a twist tester. The fibre types so obtained are placed in pre-weighed weighing bottles and dried at (105 ± 3) °C until a constant mass is obtained, as described in Clause 9.

B.8.2 Analysis of cloth

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

B.9 Calculation and expression of results

B.9.1 General

Express the mass of each fibre constituent as a percentage of the total mass of the fibres in the mixture. Calculate the results on the basis of clean, dry mass, adjusted by

a) the agreed allowances, and

b) the correction factors necessary to take account of loss of matter during pre-treatment.

B.9.2 Calculation of percentage masses of clean, dry fibre

The percentage masses of clean dry fibre, disregarding loss of fibre mass during pre-treatment is calculated as follows:

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

where

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

m_1 is the clean, dry mass of the first component;

m_2 is the clean, dry mass of the second component.

B.9.3 Calculation of the percentage of each component

For calculation of the percentage of each component with adjustment by agreed allowances and, where appropriate, by correction factors for loss of matter during pre-treatment, see 10.4.

B.10 Precision of the methods

The precision indicated in individual methods relates to the reproducibility.

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

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

By this is meant that the difference between two results in a series of analyses made in different laboratories would, given a normal and correct application of the method to an identical and consistent mixture, be exceeded only in five cases out of a 100.

B.11 Test report

a) State that the analysis was conducted in accordance with this method.

b) Give details of any special pre-treatment (see 8.2).

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

Bibliography

[1] ISO 2076, *Textiles — Man-made fibres — Generic names*

[2] ISO 6938, *Textiles — Natural fibres — Generic names and definitions*

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