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Second edition
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# Textiles — Quantitative chemical analysis —

## Part 1:
## General principles of testing

*Textiles — Analyse chimique quantitative —*

*Partie 1: Principes généraux des essais*

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# Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 38, *Textiles*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 248, *Textile and textile products*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 1833-1:2006), which has been technically revised. It also incorporates the Technical Corrigendum ISO 1833-1:2006/Cor 1:2009.

The main changes compared to the previous edition are as follows:

- Introduction, A.2 and bibliography: a reference to ISO/TR 11827 regarding the fibre identification has been added;
- Clause 2: normative references have been added;
- Clause 4: references to ISO 2076 and ISO 6938 have been added for the use of the generic names in the ISO 1833 series;
- Clause 5: warning sentences and reference to grade 3 water have been introduced;
- Clause 6: stoppered weighing bottles have been added;
- 8.2: all pretreatments have been described in Annex A (including pretreatment with light petroleum) a requirement for reporting if any pretreatment is carried out [see Clause 12, e)] has been introduced;
- 9.1.1: instructions for handling have been added;
- 9.1.2: instructions for short drying period have been added;
- 9.1.6 (former 9.1.5): a note has been removed;
- 9.2: a requirement regarding the number of test specimens has been added;

- Clause 10: deletion of former 10.3 (consideration of only percentage additions for moisture, case included in the new 10.3 covering percentage additions for moisture and non-fibrous matter, and/or percentage losses of fibrous matter by pre-treatment);
- 10.3: additional instructions in case of calculated percentage greater than 100 % have been given;
- Annexes A and B have been changed to normative;
- Table A.1 and concerned subclauses: additional cases regarding elastane (see A.5.1.2), elastolefin (see A.5.1.3), bast fibres (see A.5.25) have been introduced;
- B.8.1 and B.8.2: a requirement regarding the number of test specimens has been added.

A list of all parts in the ISO 1833 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

# Introduction

Methods for the quantitative analysis of fibre mixtures are based on two main processes, the manual separation and the chemical separation of fibres.

It is preferable to use the method of manual separation, which is given in Annex B, whenever possible since it generally gives more accurate results than the chemical method. It can be used for all textiles whose component fibres do not form an intimate mixture, for example in the case of yarns composed of several elements each of which is made up of only one type of fibre, or woven fabrics in which the fibre of the warp is of a different kind to that of the weft, or knitted fabrics capable of being unravelled made up of yarns of different types.

In general, the methods described in the different parts of ISO 1833 are based on the selective dissolution 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 document gives the information which is common to the analyses, by this method, of all fibre mixtures, whatever their composition. This information is intended to 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 dissolution, full details are given in the appropriate part.

Mixtures of fibres obtained during processing and, to a lesser extent, in finished textiles can contain non-fibrous matter, such as fats, waxes or dressings, or water-soluble matter, either occurring naturally or added to facilitate processing. Non-fibrous matters are removed before analysis.

A method of pre-treatment for removing oils, fats, waxes and water-soluble matter is given in Annex A.

Dye in dyed fibres is considered to be an integral part of the fibre and is not removed.

In addition, textiles can 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, can interfere with the action of the reagent on the soluble component and/or it can be partially or completely removed by the reagent. This type of added matter can also cause errors and are removed before the sample is analysed. If it is impossible to remove such added matter, the methods of analysis are no longer applicable.

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 document.

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 moisture regain

NOTE The agreed moisture regain of each fibre is specified in some regional legislation or after agreement between interested parties.

b) agreed moisture regain 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 can be partially dissolved in the reagent used to dissolve the soluble component. Where possible, the reagents that have been chosen are those that have little or no effect on the insoluble fibres. If loss in mass is known to occur during the analysis, the result is 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 might 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 are made, both in the case of manual separation and in the case of chemical separation, but more may be carried out if desired.

Before proceeding with any quantitative analysis, it is assumed that all the fibres present in the mixture have been identified. For this purpose, ISO/TR 11827 may be used.

For confirmation, unless it is technically impossible, it is recommended that use be made of alternative procedures whereby the component that would be the residue in the standard method is dissolved out first.

# Textiles — Quantitative chemical analysis —

## Part 1:
## General principles of testing

## 1 Scope

This document specifies a common method for the quantitative chemical analysis of various 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 105-C10, *Textiles — Tests for colour fastness — Part C10: Colour fastness to washing with soap or soap and soda*

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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

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

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 5089 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

**3.1**
**non-fibrous matter**
processing aids such as lubricants and sizes or naturally occurring non-fibrous substances

## 4 Principle

After the identification of the components of a mixture, the non-fibrous matter is removed by suitable pre-treatment and then one of the components is determined usually by selective dissolution or manual separation (see Annex B). The insoluble residue is dried and weighed, and the proportion of soluble component calculated from the loss in mass. It is usually preferable to dissolve the fibre present in the greater proportion, thus obtaining the fibre present in the smaller proportion as residue.

The fibre names in the ISO 1833 series are in accordance with the generic names listed in ISO 6938 for the natural fibres and ISO 2076 for the man-made fibres.

## 5 Reagents

Use only reagents of analytical grade. Reagents are listed in Table A.1.

**WARNING — The ISO 1833 series may call for the use of substances/procedures that may be injurious to the health/environment if appropriate conditions are not observed. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety/environment at any stage.**

**5.1 Water**, grade 3 (according to ISO 3696).

## 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.

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

**6.2 Stoppered weighing bottles**, large enough to contain such crucibles.

**6.3 Vacuum flask**.

**6.4 Desiccator**, containing self-indicating silica gel.

**6.5 Ventilated oven**, for drying test specimens at (105 ± 3) °C.

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

**6.7 Soxhlet extraction apparatus**, or any other apparatus giving identical results.

NOTE Soxhlet size of a volume, in millilitres, equal to 20 times the mass, in grams, of the laboratory test sample has been found convenient.

## 7 Conditioning and testing atmosphere

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

## 8 Sampling and pre-treatment of laboratory test 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 test 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 laboratory test sample as described in 8.2.

### 8.2 Pre-treatment of laboratory test sample

Laboratory test sample shall be pretreated by a suitable method (as described in Annex A) which does not affect any of the fibre components.

NOTE 1 The purpose of the pretreatment is to remove non-fibrous matter, which is not to be taken into account in the percentage calculations.

If the pretreatment is not applied, it shall be reported with the reasons of this deviation of the given procedure.

NOTE 2 The non-application of certain pretreatments is known to have a negligible effect on the results.

# 9 Procedure

## 9.1 General instructions

### 9.1.1 Handling

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

### 9.1.2 Drying

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

If the drying period is less than 14 h, the test specimen or the residue shall be weighed to check that its mass has become constant.

The mass may be considered to have become constant if, after a further drying period of 60 min, its variation is less than 0,05 %.

### 9.1.3 Drying of test specimens

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

### 9.1.4 Drying of crucible and residue

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

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

### 9.1.5 Cooling

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

### 9.1.6 Weighing

After cooling, complete the weighing of the test specimen in a weighing bottle or crucible with the residue in a weighing bottle within 2 min of its removal from the desiccator.

Weigh to the nearest 0,000 2 g.

## 9.2 Testing execution

From the pre-treated laboratory test sample, take a test specimen weighing about 1 g. Cut the yarn or dissected fabric into lengths of about 10 mm. Dry the test specimen in a weighing bottle, cool it in the desiccator and weigh it (see 9.1).

Transfer the test specimen to the glass vessel specified in the appropriate part of ISO 1833, reweigh the weighing bottle with its stopper immediately, and obtain the dry mass of the test 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.

The analysis of at least two test specimens shall be made.

## 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. The percentage of soluble component is obtained by difference.

Calculate the results on the basis of clean dry mass using Formula (1), adjusted by the agreed moisture regain and the correction factors necessary to take account of loss of matter during pre-treatment and analysis by using Formula (2).

NOTE The agreed moisture regain of each fibre is specified in some regional legislation or after agreement between interested parties.

**10.2** Calculation of mass percentage of insoluble component on clean dry mass basis.

$$P=\frac{100\times m_1\times d}{m_0} \qquad (1)$$

where

$P$ is the mass percentage of clean dry insoluble component;

$m_0$ is the dry mass of the test specimen, expressed in g;

$m_1$ is the dry mass of the residue, expressed in g;

$d$ 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** Calculation of mass percentage of insoluble component on clean, dry mass basis (see 10.2), with percentage additions for moisture and non-fibrous matter, and/or percentage losses of fibrous matter by pre-treatment.

$$P_{1A}=\frac{100\times P\left[1+0{,}01\times\left(a_2+b_2\right)\right]}{P\times\left[1+0{,}01\times\left(a_2+b_2\right)\right]+\left(100-P\right)\times\left[1+0{,}01\left(a_1+b_1\right)\right]} \qquad (2)$$

where

$P_{1A}$ is the mass percentage of clean insoluble component with percentage additions for moisture and non-fibrous matter;

$P$ is the mass percentage of clean dry insoluble component [as calculated in Formula (1)];

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

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

$b_1$ is the mass percentage variation 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 mass percentage variation of insoluble fibrous matter caused by the pre-treatment, and/or the percentage addition to the insoluble component for non-fibrous matter.

Calculate the mass percentage of the second component using the Formula (3).

$$P_{2A} = 100 - P_{1A} \tag{3}$$

If the calculated value of $P_{1A}$ is greater than 100 % (leading to a negative percentage of $P_{2A}$), the correction factor $d$ used in Formula (1) should be reconsidered due to a potential over estimation. The change of the correction factor $d$ shall be reported. If technically possible, the mass percentage of the components shall be confirmed based on an alternative method (e.g. chemical analysis using another solvent, manual separation, microscopical analysis).

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 components to pre-treatment applied in the analysis. Pure fibres are those free from all non-fibrous matter except that which they normally contain (either naturally or because of the manufacturing process), in the state (unbleached, bleached) in which they are found in the material to be analysed.

Where no pre-treated separate fibre components used in the manufacture of the material to be analysed are available, average values of $b_1$ and $b_2$ as obtained from tests performed on clean fibres similar to those in the mixture under examination, could be used.

## 11 Precision of the methods

The precision indicated in individual parts of ISO 1833 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 on test 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 of any part shall specify the following:

a) a reference to the relevant part of ISO 1833;

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

c) identification (e.g. description) of the laboratory test sample;

d) qualitative fibre identification of the test specimens;

e) method(s) of pre-treatment used;

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

g) the individual results and the arithmetic mean, each to the nearest 0,1 %;

h) any deviation from the given procedure, especially if pretreatment is not applied (see 8.2)

i) any unusual features observed;

j) the date of the test.

# Annex A
(normative)

# 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 described 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 matter is 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 this annex can ensure adequate removal of oils, fat and waxes. With other non-fibrous matter, it is necessary, wherever possible, to check that removal is complete.

**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 Overview

This annex describes procedures for the removal of certain commonly found types of non-fibrous matter 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 matter to be removed. The names of these fibres shall be in accordance with ISO 2076 or ISO 6938. Identification of the fibres present is covered by ISO/TR 11827.

In certain cases, the elimination of all the added matter is impracticable. The quantity remaining should not affect 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 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/Apparatus | |
| Oils, fats and waxes | Most fibres | A.5.1.1 | Light petroleum, Soxhlet | Elastane, elastolefin, Man-made cellulose fibres dyed with reactive dyes and cotton |
| | Elastane | A.5.1.2 | Soap and water | — |
| | Elastolefin | A.5.1.3 | Acetone, Soxhlet | Acetate, modacrylic, chlorofibre |
| | Man-made cellulose fibres dyed with reactive dyes and cotton | A.5.1.4 | A.5.1.1 followed by sodium hydroxide solution | — |
| 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 followed by 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 sodium hydroxide, 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 sodium carbonate | 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 | A.5.11 | Orthophosphoric acid/urea, 80 °C, 10 min, rinse in water, then sodium hydrogen carbonate | Asbestos |
| Bitumen, creosote and tar | Most fibres | A.5.12 | Dichloromethane (methylene chloride), Soxhlet | 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 can undergo a loss in mass of fibre substance of up to 1 % when treated by this method. The loss in mass of polyamide 6 can vary between 1 % and 3 %.

**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/Apparatus** | |
| Cellulose ethers | Most fibres | A.5.13.1 | Soak in cold water | — |
| | Cotton | A.5.13.2 | 175 g/l sodium hydroxide solution at 10 °C, neutralized in 0,1 mol/l 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 |
| Oleates | Most fibres | A.5.16 | 0,2 mol/l hydrochloric acid, extraction in dichloromethane, Soxhlet | Deacetylated acetate, acetate, triacetate, modacrylic, chlorofibre, polyamide, 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 ammonium hydroxide | — |
| Pentachlorophenyl laurate | Most fibres | A.5.18 | Toluene, Soxhlet | Polyethylene, polypropylene |
| Polyethylenes | Most fibres | A.5.19 | Boiling toluene under reflux | Polypropylene |
| Polyurethanes | Polyamide, cupro, viscose, modal, deacetylated acetate, acetate, triacetate | A.5.20 | Dimethyl sulfoxide or dichloromethane, if possible 50 g/l sodium hydroxide, ethanol at 50 °C | Deacetylated acetate, acetate, triacetate, polyester, acrylic, modacrylic |
| Natural rubber and styrene-butadiene, polychloroprene, 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, glass |
| Tin weighting | Silk | A.5.23 | 0,5 mol/l hydrofluoric acid | — |
| Wax-based waterproof finishes | Cotton, protein, polyester, polyamide | 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 can undergo a loss in mass of fibre substance of up to 1 % when treated by this method. The loss in mass of polyamide 6 can vary between 1 % and 3 %.

**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/Apparatus | |
| Non-cellulosic components (such as pectin, hemicellulose, ligninetc) | Bast fibres (e.g. Flax) | A.5.25 | Sodium hydroxide | — |

[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 can undergo a loss in mass of fibre substance of up to 1 % when treated by this method. The loss in mass of polyamide 6 can vary between 1 % and 3 %.

## A.5 Procedures

### A.5.1 Oils, fats and waxes

#### A.5.1.1 Oils, fats and waxes using light petroleum

Extract the test specimen in 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.

For some unbleached, natural vegetable fibres (for example, jute, coir), pre-treatment with light petroleum and water does not remove all the natural non-fibrous matter. Nevertheless, additional pre-treatment is not applied unless the sample contains finishes which are insoluble in both light petroleum and water.

#### A.5.1.2 Oils, fats and waxes using soap and water

For test specimen containing elastane, pre-treatment with light petroleum and water is not applicable as it affects the elastane itself.

NOTE Usually the masses of oils, fats and waxes on elastane fibres are negligible when elastane is present in low mass percentage.

Immerse and agitate the test specimen in water containing 1 g/l of soap, as described in ISO 105-C10, during 1 h at 60 °C, then rinse with warm water (approximately at 40 °C).

#### A.5.1.3 Oils, fats and waxes using acetone

In the case of elastolefin or fibre mixtures containing elastolefin and other fibres (except acetate), the procedure described in A.5.1.1 shall be slightly modified in that light petroleum ether shall be replaced by acetone.

#### A.5.1.4 Oils, fats and waxes using light petroleum and then sodium hydroxide solution

In the case of dyeing of man-made cellulose fibre (viscose, modal, cupro, lyocell) based on reactive dyes, conduct the procedure described in A.5.1.1, followed by the following procedure.

Boil the test specimen for 1 h in a sodium hydroxide solution at 5 % (mass fraction) and rinse with hot water.

NOTE This procedure is intended to remove concerned reactive dyes in deep colour shades for which an application of a cuprammonium hydroxide solution (Schweizer's reagent) leads to the swelling of the fibre instead of its dissolution.

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

Extract the test 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 safer.

### A.5.3 Starch

Immerse the test 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/test 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 test 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 test specimen thoroughly in water, squeeze or mangle it, and dry it.

### A.5.4 Locust-bean gum and starch

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

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

### A.5.6 Acrylic size

Immerse and agitate the test 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 test specimen.

### A.5.7 Gelatine and polyvinyl alcohol

Treat the test specimen in a solution (using a minimum liquor/test 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 test 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 test 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 test specimen with a solution of 25 g/l of 50 % orthophosphoric acid and 50 g/l of urea at 80 °C for 10 min, using a liquor/test specimen ratio of 100/1. Wash the test specimen in water, drain, wash it in a 0,1 % sodium hydrogen carbonate 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 test specimen with dichloromethane (methylene chloride) in a Soxhlet apparatus. The duration of treatment depends on the amount of non-fibrous matter present, and it might be necessary to renew the solvent.

NOTE Extraction of jute with dichloromethane also removes the batching oil, possibly 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 test specimen in cold water for 2 h. Rinse the test specimen repeatedly in cold water, with vigorous squeezing.

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

Immerse the test 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 test specimen thoroughly in a fresh portion of reagent, rinse it well in water, neutralize it with approximately 0,1 mol/l acetic acid, rinse it again in water, and dry it.

### A.5.14 Cellulose nitrate

Immerse the test specimen in acetone at room temperature for 1 h, using a liquor/test specimen ratio of 100/1.

Drain, wash the test specimen in three portions of fresh acetone, and allow the entrained solvent to evaporate.

### A.5.15 Polyvinyl chloride

Immerse the test specimen in tetrahydrofuran at room temperature for 1 h, using a liquor/test specimen ratio of 100/1.

If necessary, scrape off the softened polyvinyl chloride. Drain, wash the test 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 test specimen in approximately 0,2 mol/l hydrochloric acid at ambient temperature until it is thoroughly wetted. Wash the test specimen well and dry it. Extract the test specimen in a Soxhlet apparatus with dichloromethane for 1 h at a minimum rate of six cycles per hour.

### A.5.17 Oxides of chromium, iron and copper

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

Immerse the test specimen in a solution containing 14 g/l of hydrated oxalic acid at 80 °C for 15 min, using a liquor/test 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 test specimen). Neutralize the test specimen with ammonium hydroxide and wash it thoroughly in water. Squeeze, mangle or centrifuge, and dry it.

### A.5.18 Pentachlorophenyl laurate (PCPL)

Extract the test 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 test 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 test specimen with fresh quantities of solvent.

When the fibre composition of the test 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, polychloroprene, nitrile and most other synthetic rubbers

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

Soak the test 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 can 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 test 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 of 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) might require the addition of the same volume of nitrobenzene as of tert-butyl hydroperoxide to speed up the dissolution process.

NOTE 1 Natural rubber dissolves 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 test specimen in benzene.

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

### A.5.22 Silicones

Scour the test 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 test 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 test specimen in 0,5 mol/l hydrofluoric acid in a polyethylene vessel at 55 °C for 20 min, stirring occasionally. Rinse in warm water. Immerse the test specimen in a 2 % solution of sodium carbonate at 55 °C for 20 min. Wash the test 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 test 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 test 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 test specimen thoroughly in water until it is free from acid.

### A.5.25 Non-cellulosic components of bast fibres

Place at least 1 g of the test specimen in a weighed filter crucible. Place and dry the weighed filter crucible and the test specimen in an oven, then cool down and weigh. Allow the sodium hydroxide solution (solution of 1,5 mol/l) to boil for at least 15 min in the flask equipped with a reflux condenser. The bath ratio of the solution to the test specimen shall be at 1/100. After removal of air from the solution (due to boiling), place the test specimen in the flask and continue boiling for 1 h. Constantly wet out the test specimen in the solution. Rinse the test specimen by continuous siphoning with water for 5 min. Immerse the test specimen in the acetic acid solution of 0,1 mol/l for 10 min. Filter the contents of the flask through the weighed filter crucible and wash any fibres from the flask into the crucible with 0,1 mol/l acetic acid solution. Drain the crucible with suction and wash with water until neutralization is achieved. Do not apply suction until the washing liquor has drained under gravity. Finally, drain the crucible with suction, dry the crucible and residue, cool and weigh them.

NOTE For example, based on the intensity of the flax retting and of the applied treatments, 10 % to 40 % of noncellulosic components (such as pectin, hemicellulose, lignin) of the flax fibres are removed prior to the dissolution of viscose or cupro or modal or lyocell.