TECHNICAL REPORT 5090

Published 1977-02-15

ISO Technical Reports are subject to review within three years of publication, with the aim of achieving the agreements necessary for the publication of an International Standard.

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION · МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ · ORGANISATION INTERNATIONALE DE NORMALISATION

# Textiles — Methods for the removal of non-fibrous matter prior to quantitative analysis of fibre mixtures

*Textiles — Méthodes d'élimination des matières non fibreuses, préalablement à l'analyse quantitative des mélanges de fibres*

Technical Report 5090 was drawn up by Technical Committee ISO/TC 38, *Textiles*, and approved by the majority of its members. The reasons which led to the publication of this document in the form of a Technical Report are the following :

- the methods proposed are sometimes imprecise, and cannot, in the present state of knowledge, be improved;
- some methods are missing for the removal of certain non-fibrous materials;
- since methods for identification of non-fibrous materials are not available at present, it is not possible to decide whether removal has been total or not;
- certain methods can damage textile fibres without it being possible, at present, to assess the degree of damage.

Nevertheless, the necessity for making these methods available in the form in which they are given in this document has been unanimously recognised as urgent, notably for use with ISO 1833 and ISO 5088. On the other hand, experiments on these methods may ultimately remove the technical deficiencies cited above.



CONTENTS



## 0 INTRODUCTION

It is common practice for various additions to be made to fibres, yarns and fabrics for the purposes of assisting processing and manufacture or modifying the properties of the finished material. These usually result in appreciable increases in mass and often affect the solubility of the fibres. It must also be borne in mind that fibres generally contain a small proportion of naturally occurring non-fibrous substances. The removal of these non-fibrous substances is therefore necessary before conducting the procedure for quantitative chemical analysis specified in ISO 1833 and ISO 5088.

UDC 677.014.23/.24 : 543.05 Ref. No. ISO/TR 5090-1977 (E)

**Descriptors** : textiles, fibres, mixtures, chemical analysis, impurities, test specimen conditioning.



Printed in Switzerland

Price based on 7 pages

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 technical report do not pretend to be complete, and it should not be assumed that those described in the 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 technical report, 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 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 clause A.1 of the annex is conducted, it is not necessary to repeat this procedure as required by 1.6.2 of ISO 1833.

The contents of this document are intended for experimentation.

**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.**

## 1 SCOPE AND FIELD OF APPLICATION

This technical report 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 the table, in relation to the non-fibrous substances to be removed. The names of these fibres are defined in ISO 2076. Identification of the non-fibrous matter and of the fibres present is not covered by this technical report.



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.



## 2 REFERENCES

ISO 1833, *Textiles — Binary fibre mixtures — Quantitative chemical analysis.*

ISO 2076, *Generic names for man-made fibres.*

ISO 5088, *Textiles — Ternary fibre mixtures — Quantitative analysis.*

## 3 DEFINITION

For the purpose of this technical report, the following definition applies :

**non-fibrous matter** : Processing aids such as lubricants and sizes (but excluding jute-batching oils), and naturally occurring non-fibrous substances.

## 4 PRINCIPLE

Where possible, removal of 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.

## 5 APPARATUS

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

TABLE — **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* |
|---|---|---|---|---|
| | | No. | Reagents | |
| Oils, fats and waxes | Most fibres | A.1 | Light petroleum, Soxhlet | Elastane |
| Soaking oils | Nett silk | A.2 | Toluene/methanol, Soxhlet | — |
| Starch | Cotton[1)]<br>Linen[2)]<br>Viscose<br>Spun silk<br>Jute[3)] and most other fibres | A.3 | Amylase then boiling water | — |
| Locust-bean gum and starch | Cotton[1)]<br>Viscose<br>Spun silk | A.4 | Boiling water then A.3 | — |
| Tamarind seed size | Cotton[1)]<br>Viscose | A.5 | Boiling water twice | — |
| Acrylic (size or finish) | Most fibres[4)] | A.6 | 2 g/l soap, 2 g/l NaOH, 70 to 75 °C, rinse in water | Protein, deacetylated acetate, acetate, triacetate, acrylic, modacrylic |
| Gelatin and polyvinyl alcohol | Most fibres | A.7 | 1 g/l non-ionic detergent,<br>1 g/l anionic detergent,<br>1 g/l $Na_2CO_3$ | Protein, deacetylated acetate, acetate, triacetate, |
| Starch and polyvinyl alcohol | Cotton<br>Polyester | A.8 | A.3 followed by A.7 | Protein, deacetylated acetate, acetate, triacetate |
| Polyvinyl acetate | Most fibres | A.9 | Acetone, Soxhlet | Deacetylated acetate, acetate, triacetate, chlorofibre |
| Linseed oil sizes | Viscose crêpe yarns | A.10 | A.1 followed by A.7 | Protein, deacetylated acetate, acetate, triacetate |
| Amino-formaldehyde resins | Cotton<br>Cupro<br>Viscose<br>Modal<br>Deacetylated acetate<br>Acetate<br>Triacetate<br>Polyester<br>Polyamide (nylon) | A.11 | Orthophosphoric acid/urea, 80 °C, 10 min, rinse in water, then $NaHCO_3$ | Asbestos |
| Bitumen, creosote and tar | Most fibres | A.12 | Dichloromethane (methylene chloride), Soxhlet | Deacetylated acetate, acetate, triacetate, modacrylic, chlorofibre |
| Cellulose ethers | Most fibres | A.13.1 | Soak in cold water | — |
| | Cotton | A.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.14 | Soaking in acetone, 1 h | Deacetylated acetate, acetate, triacetate |
| Polyvinyl chloride | Most fibres | A.15 | Soaking in tetrahydrofuran (do not recover by distillation) | Deacetylated acetate, acetate, triacetate, chlorofibre |
| Oleates | Most fibres | A.16 | 0,2 N HCl, extraction in dichloromethane, Soxhlet | Deacetylated acetate, acetate, triacetate, modacrylic, chlorofibre, polyamide (nylon), asbestos |



1) Grey cotton loses mass when treated by these methods. The loss amounts to approximately 3 % of the final oven-dry mass.

2) Linen loses mass when treated by this method. The loss depends on the types of yarn from which the fabric is produced.

Losses are approximately as follows :

bleached yarns 2 %, boiled yarns 3 % and grey yarns 4 %.

3) Jute loses mass by approximately 0,5 % when treated by this method.

4) 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 %.

TABLE (*concluded*)

| 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* |
|---|---|---|---|---|
| | | No. | Reagents | |
| Oxides of chromium, iron and copper | Cupro<br>Viscose<br>Modal<br>Deacetylated acetate<br>Acetate<br>Triacetate | A.17 | 10 g/l hydrated oxalic acid at 80 °C, neutralized with $NH_4OH$ | – |
| Pentachlorophenyl laurate | Most fibres | A.18 | Toluene, Soxhlet | Polyethylene, polypropylene |
| Polyethylenes | Most fibres | A.19 | Boiling toluene under reflux | Polypropylene |
| Polyurethanes | Polyamide (nylon)<br>Cupro<br>Viscose<br>Modal<br>Deacetylated acetate<br>Acetate<br>Triacetate | A.20 | Dimethyl sulphoxide 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<br>Viscose<br>Modal<br>Deacetylated acetate<br>Acetate<br>Triacetate<br>Glass | A.21 | Swell in benzene, scrape, heat in molten *p*-dichlorobenzene, after 45 min, add 1 part *tert*-butyl hydroperoxide per 4 parts of *p*-dichlorobenzene, cool to 60 °C, add benzene | All synthetic fibres |
| Silicones | Most fibres | A.22 | Hydrofluoric acid, 50 to 60 ml per litre, 65 °C | Polyamide (nylon), glass |
| Tin weighting | Silk | A.23 | 0,5 N hydrofluoric acid | – |
| Wax-based waterproof finishes | Cotton<br>Protein<br>Polyester<br>Polyamide (nylon) | A.24 | Dichloromethane, Soxhlet, if metallic complex : 1 g/l formic acid and 5 g/l soap | Deacetylated acetate, acetate, triacetate, modacrylic, chlorofibre |



## ANNEX

## PROCEDURES FOR REMOVAL OF NON-FIBROUS MATTER

### A.2 SOAKING OILS

Extract the specimen is a Soxhlet apparatus with light petroleum (distilling betwwen 40 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 1.6.2 of ISO 1833.

### A.2 SOAKING OILS

Extract the specimen in a Soxhlet 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.3 STARCH

Immerse the specimen in a freshly prepared solution containing 0,1 % (*m/m*) 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.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.3.

### A.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.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 to 75 °C. Give three 5 min rinses in distilled water at 85 °C, squeeze, mangle or centrifuge, and dry the specimen.

### A.7 GELATIN 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 to 75 °C. Wash the specimen and dry it.

### A.8 STARCH AND POLYVINYL ALCOHOL



Conduct the procedure described in A.3, followed by the procedure described in A.7, with intermediate drying.



### A.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.10 LINSEED OIL SIZES

Conduct the procedure described in A.1, followed by the procedure described in A.7.

### A.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 °C 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.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.13 CELLULOSE ETHERS

#### A.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.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 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.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.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.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.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.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.19 POLYETHYLENES

Extract the specimen in boiling toluene under reflux.

NOTE — The material must be completely immersed in the boiling solvent.

### A.20 POLYURETHANES

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

Some polyurethanes can be removed by dissolution in dimethyl sulphoxide 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.

NOTE — Dimethyl sulphoxide has toxic properties.

### A.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.

NOTES

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 to 160 °C for 2 h and then washing the specimen in benzene.

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

## A.22 SILICONES

Scour the specimen in a solution containing 50 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.

NOTE – Hydrofluoric acid is a dangerous product.

## A.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.

NOTE – Hydrofluoric acid is a dangerous product.



## A.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.

This page intentionally left blank