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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Textiles — Fibres and yarns — Determination of commercial mass of consignments —

Part 3 : Specimen cleaning procedures

Textiles — Fibres et fils — Détermination de la masse commerciale d'un lot —

Partie 3: Méthode de nettoyage des éprouvettes

Reference number
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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6741-3 was prepared by Technical Committee ISO/TC 38, *Textiles*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Textiles — Fibres and yarns — Determination of commercial mass of consignments —

Part 3 :
Specimen cleaning procedures

0 Introduction

This document forms part 3 of a four-part International Standard prepared by ISO/TC 38, *Textiles*.

Part 1: Mass determination and calculations.

Part 2: Methods for obtaining laboratory samples.

Part 3: Specimen cleaning procedures.

Part 4: Values used for the commercial allowances and the commercial moisture regains. (Technical Report.)

The terminology used in this International Standard is in accordance with ISO 6348.

Most contracts of sale between buyer and seller specify either that the invoice mass of a consignment shall be determined by an independent third party, or that the seller's figure may be subject to an independent third-party check. This part of ISO 6741 describes the methods which are to be used by the independent third party in these cases. The figure for the commercial mass which results from the application of the procedures in this part of ISO 6741 either becomes the invoice mass of the consignment or is compared with the declared invoice mass plus or minus the tolerance agreed between the buyer and seller.

It is not intended that the methods in this part of ISO 6741 necessarily be used by the seller to establish his invoice mass.

The methods described in this part of ISO 6741 are, for the most part, destructive.

1 Scope and field of application

This part of ISO 6741 specifies specimen cleaning procedures to be used when the commercial mass is to be determined in accordance with ISO 6741-1 on a clean and dry basis. The procedure appropriate for a particular fibre is given in ISO 6741-4.

If the commercial mass is to be determined on a dry-only basis, cleaning is omitted.

2 References

ISO 675, *Textiles — Woven fabrics — Determination of dimensional change on commercial laundering near the boiling point.*

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

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading classification and designation.*

ISO 6348, *Textiles — Determination of mass — Vocabulary.*

ISO 6741, *Textiles — Fibres and yarns — Determination of commercial mass of consignements*

— *Part 1: Mass determination and calculations.*

— *Part 2: Methods for obtaining laboratory samples.*

— *Part 4: Values used for the commercial allowances and the commercial moisture regains.* (Technical Report.)

3 Principle

The test specimen, obtained as specified in ISO 6741-1, following the procedures given in ISO 6741-2, is cleaned by the method recommended for the particular fibre in ISO 6741-4.

4 Apparatus and reagents

Conventional laboratory apparatus is required. All reagents used shall be chemically pure.

5 Procedures

NOTE — See sub-clause 6.5 of ISO 6741-1.

5.1 Methods A1 and A2: Washing with soap

Enclose the test specimen in an open-textured bag large enough when closed with a draw-tape to contain the material in

a loose state. The bag and its draw-tape shall be made of filament polyamide or polyester yarn and shall have been previously boiled in several lots of distilled water. The oven-dry mass of the bag and its contents shall be determined to an accuracy of 0,05 %.

5.1.1 Method A1

Wash the test specimen (in the bag) at 70 to 75 °C in at least 25 times its own mass of a solution made by dissolving 5 g of soap flakes (the soap specified in ISO 675 is suitable) per litre of soft water, i.e. water of hardness not exceeding 5 mg of calcium carbonate per litre. Keep the specimen moving for 30 min in the solution, maintaining the temperature at 70 to 75 °C. Add more soft water at 70 to 75 °C, allowing the bath to overflow until all scum and foam have disappeared. Take the bag and its contents out of the bath and expel as much liquid as possible by centrifuging (preferably) or by squeezing. Rinse twice for 5 min in soft water at 85 °C, keeping the bag moving in the water. Finally, rinse for 5 min in demineralized water at 85 °C. Centrifuge (or squeeze) the specimen after each rinse.

5.1.2 Method A2

Wash the test specimen (in the bag) in a boiling solution at least 25 times its own mass of a solution made by dissolving 2 g of sodium carbonate per litre of soft water, i.e. water of hardness not exceeding 5 mg of calcium carbonate per litre. Keep the specimen moving for 45 min in the boiling solution. Add more boiling water, allowing the bath to overflow until all scum has disapperared. Take the bag and its contents out of the bath and expel as much liquid as possible by centrifuging (preferably) or by squeezing. Rinse twice for 5 min in boiling water, keeping the bag moving in the water. Finally, rinse for 5 min in boiling demineralized water. Centrifuge (or squeeze) the specimen after each rinse.

5.2 Method B: Washing with detergent

Proceed as in method A1 (5.1.1) but use a solution containing 5 g/l of the detergent specified in the annex in place of a soap solution.

5.3 Method C: Extraction with light petroleum

Enclose the test specimen in an open-textured bag large enough when closed with a draw-tape to contain the material in a loose state. The bag and its draw-tape shall be made of filament polyamide or polyester yarn and shall have been previously extracted with light petroleum. The oven-dry mass of the bag and its draw tape shall be determined to an accuracy of 0,05 %.

Place the bag containing the test specimen into either a tared glass Soxhlet thimble fitted with a sealed-in sintered disc of porosity grade P 40 (see ISO 4793) or into a tared alumina Soxhlet thimble of porosity grade P 40.

Carry out an extraction in a Soxhlet apparatus with light petroleum (boiling range 40 to 60 °C) for 1 h at a minimum rate of six extraction cycles per hour.

Remove the thimble and contents from the extractor, attach them to a filter flask and remove the light petroleum by suction.

Place the thimble and contents in a Soxhlet apparatus and extract with water for 2 h at a minimum rate of six extraction cycles per hour. Remove the sample and centrifuge to remove excess water.

(This method is taken from ISO 1833.)

5.4 Method D: Extraction with dichloromethane followed by size removal

Enclose the test specimen in an open-textured bag large enough when closed with a draw-tape to contain the material in a loose state. The bag and its draw-tape shall be made of unsized or fully desized cotton yarn and shall have been previously extracted with dichloromethane. The oven-dry mass of the bag and its draw tape shall be determined to an accuracy of 0,05 %.

Place the bag and its contents in a Soxhlet apparatus and extract with dichloromethane (methylene chloride) for at least ten cycles.

Remove the bag and test speciment and, after evaporation of the solvent, rinse by hand in two lots of soft water (hardness not exceeding 5 mg of calcium carbonate per litre) at 50 °C. Transfer to a solution of an enzyme preparation (a 0,05 % to 0,10 % diastatic solution at pH 6,0 to 7,5 is suitable) and maintain at 50 °C until all starchy matter has been shown to be hydrolysed by testing with a 0,5 % solution of iodine in potassium iodide.

Finally, wash the specimen in its bag by boiling for 5 min in soft water, cool it and squeeze it by hand. Repeat this last process to give six treatments in all. Centrifuge the sample in its bag to remove excess water.

5.5 Method E: Extraction with methanol

Enclose the test specimen in an open-textured bag large enough when closed with a draw-tape to contain the material in a loose state. The bag and its draw-tape shall be made of filament polyester yarn and shall have been previously extracted with methanol. The oven-dry mass of the bag and its draw tape shall be determined to an accuracy of 0,05 %.

Transfer the bag containing the test specimen to a 200 ml Soxhlet apparatus and extract with methanol. The amount of methanol shall be at least 1,5 times the amount of one Soxhlet charge. Continue the extraction for ten cycles. Remove the specimen and centrifuge to remove excess methanol.

5.6 Method F: Washing in soft or distilled water

Enclose the test specimen in an open-textured bag large enough when closed with a draw-tape to contain the material in a loose state. The bag and its draw-tape shall be made of filament polyester or polyamide yarn and shall have been previously boiled in several lots of distilled water. The oven-dry mass of the bag and its draw tape shall be determined to an accuracy of 0,05 %.

Wash the test specimen (in the bag) for 30 min with specimen agitation in at least 60 times its own mass of soft water (hardness not exceeding 5 mg of calcium carbonate per litre) at 60 to 70 °C. Remove the specimen, squeeze gently to expel as much

water as possible, place the specimen into a fresh supply of soft water at 60 to 70 °C, and agitate for a further 100 min. Rinse for 5 min in distilled water at 20 °C. Remove the specimen and squeeze gently to expel as much water as possible.

5.7 Method G : Cleaning by pyrolysis

WARNING — It is important that fumes do not enter the laboratory, causing a potential build-up of toxic volatile metals such as mercury, cadmium or antimony. Also, acrylic fibres on ashing produce the highly toxic gas hydrogen cyanide.

Place the test specimens on to specimen holders and put these assemblies into a muffle furnace maintained at a temperature of 625 ± 20 °C. Leave to burn for 5 min with the furnace door open (see the note) and then for 30 min with the door closed.

NOTE — The open door allows volatile gases to escape without depositing on to either the test specimen or its holder the residual solids which the specimen may contain.

Remove the assemblies from the muffle furnace and place them in a desiccator. Allow them to cool to the temperature of the weighing room (see ISO 139).

Handling precautions

Do not allow the sample to come into contact with the oven. Transfer the assemblies from the oven to the balance with great care, in order to avoid any loss of material.

Never touch the sample with the fingers; always use tweezers.

If the yarn has been given a special size which cannot be completely removed by combustion, other desizing methods may be used by agreement between the interested parties.

5.8 Method Z : Other cleaning procedure

A method agreed between the interested parties for use only when the above cleaning methods are not suitable.

Annex

Detergent to be used in connection with method B

(This annex forms an integral part of the Standard.)

IEC test detergent with perborate, type 1, of the following formulation:

	% (*m*/*m*)
Linear sodium alkyl benzene sulfonate (mean length of alkane chain: $C_{11,5}$)	6,4
Ethoxylated tallow alcohol (14 moles ethylene oxide)	2,3
Sodium soap (chain length C_{12} to C_{22})	2,8
Sodium tripolyphosphate	35,0
Sodium silicate (SiO_2/Na_2O = 3,3/1)	6,0
Magnesium silicate	1,5
Sodium carboxymethylcellulose	1,0
Ethylenediamine-tetra-acetic acid sodium salt	0,2
Sodium sulfate (as accompanying substance or added)	16,8
Water	8,0
Sodium perborate ($NaBO_2 \cdot 3H_2O \cdot H_2O_2$) (freshly added to the above, immediately before use)	20,0

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