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Wool — Determination of dichloromethane-soluble matter in combed sliver

Laine — Détermination de l'extrait dichlorométhanique dans un ruban de peigné

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FOREWORD

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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.

International Standard ISO 3074 was drawn up by Technical Committee ISO/TC 38, *Textiles*, and circulated to the Member Bodies in October 1974.

It has been approved by the Member Bodies of the following countries :

Australia	Germany	Poland
Belgium	Hungary	Romania
Bulgaria	India	South Africa, Rep. of
Canada	Iran	Spain
Chile	Ireland	Sweden
Czechoslovakia	Israel	Turkey
Denmark	Japan	U.S.A.
Finland	Netherlands	U.S.S.R.
France	New Zealand	Yugoslavia

No Member Body expressed disapproval of the document.

This International Standard is based on Test Method IWTO-10-66, drawn up by the International Wool Textile Organization (IWTO).

Printed in Switzerland

Wool — Determination of dichloromethane-soluble matter in combed sliver

0 INTRODUCTION

Wool textiles may contain solvent-extractable oils and fats. These are derived mainly from

a) the wool grease occurring naturally in raw wool;

b) oils added to assist textile processing;

c) detergents picked up during washing and scouring processes;

d) special finishing agents.

The amount of these substances present depends on the stage of manufacture and its estimation is important for determining the clean wool content of a sample.

These different materials connot be estimated individually by solvent extraction methods, since there are no known solvents that are specific for each component. Hence, it is only possible to determine the amount of these substances extracted by a given solvent under specified conditions, any additional information being obtained by detailed analysis of the extracted material. Dichloromethane is recognized as a suitable solvent for extracting oils and fats.

The method described in this International Standard is based on the results of inter-laboratory trials organized by the Technical Committee of the International Wool Textile Organization.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the dichloromethane-soluble matter in combed wool sliver. Its use may be extended to wool in other forms.

It should be recognized that extraction with dichloromethane under the prescribed conditions does not completely remove all the fatty material present in a sample of wool. A further amount, possibly material of similar character, will usually be extracted by the use of solvents that cause greater swelling of the wool fibres.

The method is applicable only to 100 % wool products. It may give misleading results if applied to products in which fibres other than wool are present.

2 REFERENCES

ISO 139, *Textiles — Standard atmospheres for conditioning and testing.*

ISO 1130, *Textile fibres — Some methods of sampling for testing.*

3 DEFINITION

dichloromethane-soluble extract : The material extracted from wool by dichloromethane under prescribed conditions.

4 PRINCIPLE

Extraction of a known amount of wool in a Soxhlet apparatus with dichloromethane. Filtration of the dichloromethane solution, evaporation of the solvent and determination of the mass of the residue.

5 REAGENTS

5.1 Dichloromethane (methylene chloride), boiling range 39 to 41 °C.

When 100 ml of the solvent is evaporated, the residue shall not exceed 1 mg.

WARNING. Dichloromethane is toxic : the room in which extractions are made shall be adequately ventilated.

5.2 Acetone, analytical reagent quality.

6 APPARATUS

6.1 Soxhlet extraction apparatus assembled with ground glass joints and protected against the entry of moisture. The extractor (barrel) of the Soxhlet shall conveniently have a capacity of about 200 to 300 ml and the flask 250 ml. (Note of the volume of the extractor shall be made in the test report.)

6.2 Water-bath or other suitable means of low temperature heating.

6.3 Balance, with an accuracy of 0,05 g, preferably with large scale-pan.

6.4 Analytical balance, accurate to 0,000 1 g.

6.5 Desiccator.

6.6 Drying oven, capable of being controlled at a temperature of 105 ± 3 °C.

6.7 Conical flasks, 100 ml capacity.

6.8 Funnel.

6.9 Distillation unit.

6.10 Fat-free filter papers.[1)]

7 CONDITIONING AND TESTING ATMOSPHERE

The atmospheres for pre-conditioning, conditioning and testing shall be those specified in ISO 139.

8 SAMPLING

The laboratory sample shall be representative of the bulk of material and shall be sufficient to provide two test specimens each of mass approximately 20 g.

Useful information on sampling is given in ISO 1130.

9 PROCEDURE

9.1 Pre-condition the laboratory sample as specified in ISO 139, and then bring it to constant mass by exposing it for not less than 24 h in the standard atmosphere for testing (see clause 7).

9.2 In the standard atmosphere for testing, prepare two test specimens each of mass 20 ± 0,05 g. For each duplicate test, introduce the test specimen into the Soxhlet barrel in such a way that the extract will not carry wool fibres into the siphon tube and that the level of the top of the test specimen is below that of the end of the siphon tube. A particle-free extract may be secured by one of the following methods :

a) Insert a glass wool plug at the bottom of the Soxhlet barrel, effectively covering the exit tube.

b) Pack the test specimen into a Soxhlet thimble covering with a loose plug of dichloromethane-extracted cotton wool.

c) Enclose the test specimen in a lightweight woven or knitted dichloromethane-extracted fabric.

If a water-bath is used, heat it to approximately 45 °C. Assemble the flask and Soxhlet barrel. Pour into the barrel sufficient dichloromethane (5.1) to cause a first siphoning, together with a small excess. Complete the assembly of condenser, Soxhlet barrel, flask and heating device. Check that all joints are tight. Adjust the heating so that satisfactory siphoning occurs at the rate of not less than 6 cycles per hour. Allow 20 to 24 siphonings, adding additional solvent if desired. Reject any test in which the siphoning does not function correctly.

9.3 Filter the contents of the extraction flask, previously concentrated (if necessary) to approximately 25 ml, through filter paper (6.10) into a tared 100 ml conical flask (6.7) which is heated on a boiling water-bath, but not in direct contact with the water. A sintered glass Büchner filter may be used in lieu of filter paper. Wash the extraction flask and filter with three separate 10 ml portions of dichloromethane. Before the last washing, cut off the edge of the paper and place it at the bottom of the cone to facilitate washing. Check that the extraction flask and filter are free from fatty matter by washing with a further 10 ml portion of dichloromethane, which is collected separately and then evaporated on a watch glass. If any fatty residue appears, continue washing until the extraction flask and filter are free from fatty matter.

9.4 When the distillation of the dichloromethane is completed, detach the conical flask and heat on the water-bath for a further 5 min. If droplets of water are present in the flask, add 2 to 5 ml of acetone (5.2) and heat on the water-bath, repeating the process if necessary until no water is visible.

9.5 Heat the conical flask for 30 min in the drying oven (6.6) at 105 ± 3 °C or in a vacuum oven and then introduce, for a few seconds, a tube connected to a pump so as to suck out vapour from the flask.

9.6 Finally, heat for a further 5 min in the oven and place in the desiccator (6.5). Determine the mass of the flask and contents and hence the mass of the dichloromethane-soluble extract.

9.7 If the result is to be expressed on the dry mass of the test specimen, determine the dry mass of the extracted test specimen by heating to constant mass at a temperature of 105 ± 3 °C, preferably in a ventilated enclosure.

1) Schleicher No. 597 or Whatman No. 2 are suitable.

10 EXPRESSION OF RESULTS

10.1 The dichloromethane-soluble extract is given, as a percentage of the conditioned mass of the de-greased specimen, by the formula :

$$\frac{100\,m_1}{20 - m_1}$$

where m_1 is the mass, in grams, of the dichloromethane-soluble extract.

10.2 The dichloromethane-soluble extract is given, as a percentage of the dry mass of the de-greased specimen, by the following formula :

$$\frac{100\,m_1}{m_2}$$

where

m_1 is the mass, in grams, of the dichloromethane-soluble extract;

m_2 is the dry mass, in grams, of the extracted test specimen.

11 TEST REPORT

The test report shall include the following information :

a) reference to this International Standard;

b) the individual results and their mean, stating whether the results are expressed as a percentage of the dry mass or the conditioned mass of the de-greased wool;

c) the volume of the Soxhlet extractor;

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