INTERNATIONAL STANDARD

ISO 3074

Second edition
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Wool — Determination of dichloromethane-soluble matter in combed sliver

Laine — Méthode de détermination de l'extrait dichlorométhanique dans un ruban de laine peignée

Reference number
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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).

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

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 38, *Textiles*, Subcommittee SC 23, *Fibres and yarns*.

This second edition cancels and replaces the first edition (ISO 3074:1975), which has been technically revised.

This second edition to ISO 3074 is based on the test method IWTO-10-03, drawn up by the International Wool Textile Organization (IWTO).

Introduction

Wool textiles can 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 cannot 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 (IWTO).

Wool — Determination of dichloromethane-soluble matter in combed sliver

1 Scope

This International Standard specifies a method for determining the dichloromethane-soluble matter in combed wool sliver. Its use can 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 can give misleading results if applied to products in which fibres other than wool are present.

2 Normative references

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

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

3 Terms and definitions

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

3.1
dichloromethane-soluble extract
material extracted from wool by dichloromethane under prescribed conditions

4 Principle

Once the appropriate specimen has been selected, it is then extracted in a Soxhlet extraction apparatus, using dichloromethane as the extraction solvent. The solvent is evaporated and both the residue and the extracted wool sample are oven-dried and weighed after cooling. The extractable matter is calculated by expressing the oven-dry mass of the residue as a percentage of the oven-dry mass of the extracted test specimen.

5 Reagents

5.1 Dichloromethane (methylene chloride), boiling range 39 °C 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 ml to 300 ml and the flask 250 ml.

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

6.3 Balance, with an accuracy of 0,01 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 °C ± 3 °C.

6.7 Distillation unit.

6.8 Fat-free filter papers.

7 Conditioning and testing atmosphere

Pre-conditioning, conditioning, and testing shall be carried out in the standard atmosphere 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 10 g.

Useful information on sampling is given in ISO 1130.

9 Procedure

9.1 Precondition the laboratory sample as specified in ISO l39, 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 10 g ± 0,5 g [use large scale-pan (6.3)]. 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 fat-free filter paper (6.8).

If a water-bath (6.2) 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 the 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 more dichloromethane if desired. Reject any test in which the siphoning does not function correctly.

9.3 Remove the Soxhlet apparatus (6.1) from the heat source. Remove the extracted test specimen from the barrel; allow it to air-dry in a fume cupboard.

9.4 Carefully boil off the dichloromethane by placing the extraction flask on the distillation unit (6.7). If droplets of water are present in the flask, add 2 ml to 5 ml of acetone (5.2) and heat on the water-bath (6.2), repeating the process if necessary until no water is visible.

9.5 Heat the extraction flask and the extracted test specimen in a ventilated oven (6.6) for 120 min at 105 °C ± 3 °C, then move them into a desiccator (6.5), cool for 30 min, then weigh on a balance (6.4) reading to 0,000 1 g and determine the oven-dry mass of the residue and the extracted test specimen.

10 Expression of the results

10.1 For each individual test specimen, calculate the percentage of extractable matter (residue) using the following formula:

$$C = \frac{m_1}{m_2} \times 100$$

where

C is the percentage of dichloromethane-soluble extract (residue), in %;

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

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

10.2 Calculate the mean of the individual determinations and report to the nearest 0,1 %.

11 Test report

The test report shall include the following information:

a) reference to this International Standard, i.e. ISO 3074;

b) the individual results and their mean;

c) the volume of the Soxhlet extractor.