INTERNATIONAL STANDARD 4048

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

Leather — Determination of matter soluble in dichloromethane

Cuir — Dosage des matières solubles dans le dichlorométhane

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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 4048 was developed by Technical Committee ISO/TC 120, *Leather*, and was circulated to the member bodies in August 1976.

It has been approved by the member bodies of the following countries :

Australia	Hungary	Poland
Brazil	India	Romania
Chile	Israel	South Africa, Rep. of
Czechoslovakia	Mexico	Turkey
France	Netherlands	United Kingdom
Germany	New Zealand	

The member body of the following country expressed disapproval of the document on technical grounds :

U.S.S.R.

This International Standard is based on method IUC/4 of the International Union of Leather Technologists' and Chemists' Societies.

Leather — Determination of matter soluble in dichloromethane

0 INTRODUCTION

Not all fatty and similar substances can be extracted from leather with organic solvents; they may be partly soluble and partly bound to the leather. On the other hand, the solvent may dissolve non-fatty substances, for example sulphur and impregnants, both of which cause difficulty in the determination of the acid and saponification values of the fats.

NOTE — The apparatus and procedure specified in this method are also suitable for the extraction of leather by solvents other than dichloromethane. If, for any purpose, other solvents are used, the solvent or solvents used should be stated in the test report.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the substances in leather which are soluble in dichloromethane.

This method is applicable to all types of leather.

2 REFERENCES

ISO 2418, *Leather — Laboratory samples — Location and identification.*

ISO 2588, *Leather — Sampling — Number of items for a gross sample.*

ISO 4044, *Leather — Preparation of chemical test samples.* [1]

ISO 4098, *Leather — Determination of water-soluble matter, water-soluble inorganic matter, and water-soluble organic matter.* [1]

3 DEFINITION

For the purposes of this International Standard, the following definition applies :

extractable substances : Fats and other soluble matter which can be extracted from leather with dichloromethane.

4 PRINCIPLE

Continuous extraction of a sample of the prepared leather with dichloromethane. Evaporation of the solvent from the extract. Drying of the extract at 102 ± 2 °C and weighing.

5 REAGENTS

During the analysis, use only reagents of recognized analytical grade.

5.1 Dichloromethane, boiling point 38 to 40 °C, freshly distilled and kept in a dark flask over calcium oxide.

WARNING — Dichloromethane has toxic properties and should be used with caution.

NOTES

1 Dichloromethane that has stood for a long time should be tested for the presence of any hydrochloric acid which may have formed, as follows :

Shake 10 ml of dichloromethane with 1 ml of 0,1 N silver nitrate solution. If the silver nitrate solution becomes turbid, the dichloromethane should be redistilled and kept in a dark flask over calcium oxide.

2 Dichloromethane which has been used for this analysis can be recovered and reused after distillation.

6 APPARATUS

Ordinary laboratory apparatus and in particular

6.1 Soxhlet extraction apparatus, including an extraction flask of suitable capacity and a condenser.

6.2 Filter paper thimbles, of suitable sizes and manufacture, or **suitable glass filter bells.**

6.3 Oven, capable of being maintained at 102 ± 2 °C.

7 SAMPLING

7.1 Whole pieces of leather

In the absence of any other agreement on sampling between the interested parties, the procedure specified in ISO 2588 for sampling from a lot shall be followed. Samples shall be taken from the pieces as specified in ISO 2418.

7.2 Other applications

Sampling shall be carried out as required by the relevant specification or contract.

1) At present at the stage of draft.

8 PROCEDURE

Prepare the sample as specified in ISO 4044.

Weigh 10 ± 0,1 g of the prepared sample and press evenly into a filter paper thimble or glass bell (6.2). Cover the leather with a thin layer of cottonwad, previously extracted with the dichloromethane (5.1).

Dry the extraction flask (see 6.1) with two glass beads in it by heating for 30 min at 102 ± 2 °C. Weigh after cooling in a desiccator.

Begin the continuous extraction with the dichloromethane (see note 1); then, after at least 30 changes of solvent, distil the dichloromethane from the flask containing the extract (see note 2).

Dry the extract for 4 h in the oven (6.3), maintained at 102 ± 2 °C (if drops of water are visible before drying, add 1 to 2 ml of ethanol). Weigh after cooling for 30 min in a desiccator.

Repeat the drying, cooling and weighing operations at least three times, but with drying periods of 1 h, until either the further loss in mass does not exceed 0,01 g, or the total drying time equals 8 h (see note 3).

NOTES

1 Dichloromethane can also dissolve non-fatty materials from the leather, for example sulphur (the presence of sulphur is recognizable by a yellow precipitate in the flask). As sulphur causes difficulty, it can be removed in the following way :

Dissolve the extract in the smallest possible quantity of diethyl ether and filter through a little cottonwad into a previously weighed flask. After thoroughly washing out the cottonwad filter with ether, remove the ether from the extract in the flask by distillation over a hot water bath from which any flame has previously been removed. If sulphur should again precipitate, repeat the procedure. After the diethyl ether has been distilled off, dry the flask and residue and weigh.

2 The extract can be used for analysis, for example to determine acid and saponification values of the fats, or to determine the free fatty acid content of the leather.

3 After removal of the solvent, the extract may be used for determination of water-soluble substances in accordance with ISO 4098.

9 EXPRESSION OF RESULTS

9.1 Calculation

The matter extractable in dichloromethane [or other specified solvent(s)] is given, as a percentage by mass, by the formula

$$\frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the extract.

9.2 Repeatability

The results of duplicate determinations carried out by the same operator in the same laboratory should not differ by more than 0,2 %, calculated on the original mass of leather.

9.3 Reproducibility

The results of two determinations carried out by different operators in different laboratories on the same sample should not differ by more than 0,5 %, calculated on the original mass of leather.

10 TEST REPORT

The test report shall include the following particulars :

a) a reference to this International Standard;

b) complete identification of the sample;

c) the characteristics of the solvent;

d) the results obtained, to one decimal place, and the mean value;

e) details of any special circumstances which may have affected the results.