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Leather — Chemical tests — Determination of water-soluble matter, water-soluble inorganic matter and water-soluble organic matter

Cuir — Essais chimiques — Dosage des matières solubles dans l'eau, des matières inorganiques solubles dans l'eau et des matières organiques solubles dans l'eau

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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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 voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by the Chemical Tests Commission of the International Union of Leather Technologists and Chemists Societies (IUC Commission, IULTCS) in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 289, *Leather*, the secretariat of which is held by UNI, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

It is based on IUC 6, originally published in *J. Soc. Leather Tech. Chem.*, **49**, p. 13, 1965, and declared an official method of the IULTCS in 1965.

IULTCS, originally formed in 1897, is a world-wide organization of professional leather societies to further the advancement of leather science and technology. IULTCS has three commissions, which are responsible for establishing international methods for the sampling and testing of leather. ISO recognizes IULTCS as an international standardizing body for the preparation of test methods for leather.

This second edition cancels and replaces the first edition (ISO 4098:2006), which has been technically revised in 8.1 and 9.1 to allow a larger tolerance in the temperature of extraction.

Leather — Chemical tests — Determination of water-soluble matter, water-soluble inorganic matter and water-soluble organic matter

1 Scope

This document specifies a method of determination of water-soluble matter, water-soluble inorganic matter and water-soluble organic matter.

It is applicable to all leather types. The result obtained by this analysis depends on factors such as:

— the degree to which the leather is ground;

— the extraction temperature;

— the extraction period;

— the ratio of leather to water.

To obtain comparable results, it is consequently imperative that test conditions be accurately reproduced.

In all cases, any ammonium salts in the filtrate are included as part of the water-soluble matter and are then decomposed on ignition. Thus they contribute towards the result for water-soluble organic substances. The concentration of the ammonium salts can be determined in the filtrate separately if required.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2418, *Leather — Chemical, physical and mechanical and fastness tests — Sampling location*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4048, *Leather — Chemical tests — Determination of matter soluble in dichloromethane and free fatty acid content*

ISO 4044, *Leather — Chemical tests — Preparation of chemical test samples*

ISO 4684, *Leather — Chemical tests — Determination of volatile matter*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
water-soluble matter
substance which, under the conditions described in this method, is dissolved out of the leather by water

3.2
water-soluble inorganic matter
sulfated ash of the *water-soluble matter* (3.1) prepared in accordance with this method

3.3
water-soluble organic matter
difference between total *water-soluble matter* (3.1) and *water-soluble inorganic matter* (3.2)

4 Principle

Following aqueous extraction of a prepared sample under specified conditions, the water-soluble matter is quantified by evaporation and drying at (102 ± 2) °C. Sulfating and ashing of the residue at 700 °C yields the water-soluble inorganic matter. The water-soluble organic matter is derived by the difference.

5 Reagents

5.1 Sulfuric acid solution,1 mol/l.

5.2 Distilled or **deionized water**, conforming to the requirements of Grade 3 of ISO 3696.

6 Apparatus

The usual laboratory apparatus is required, in particular the following:

6.1 Flasks, with a wide neck and a close fitting stopper (650 ml to 750 ml capacity should be suitable).

6.2 Graduated measuring cylinder, capacity 500 ml.

6.3 Pipette, capacity 50 ml.

6.4 Evaporating basin, quartz, platinum or porcelain, with flat bottom, and a working capacity of at least 50 ml.

6.5 Appropriate shaking apparatus operating at (50 ± 10) cycles per minute.

6.6 Thermometer of range from 0 °C to 50 °C.

6.7 Fluted filter paper of fast qualitative grade.

6.8 Boiling water or **steam bath**.

6.9 Oven, capable of being maintained at (102 ± 2) °C.

6.10 Muffle furnace, capable of being maintained at a temperature close to but not exceeding 700 °C (see 9.4).

6.11 Desiccator.

6.12 Analytical balance weighing to an accuracy of 0,001 g.

7 Sampling and preparation of samples

Sample in accordance with ISO 2418 and grind leather in accordance with ISO 4044. If sampling in accordance with ISO 2418 is not possible (e.g. in the case of leathers from finished products, such as shoes or garments), details about sampling shall be given together with the test report.

Weigh accurately approximately 10 g of ground leather and record the mass (m_0). Extract the leather in accordance with ISO 4048 before determination of the water-soluble matter.

If the result is to be presented on the basis of dry substance, then a further sample of the same leather shall be tested in accordance with ISO 4684, so that the moisture content can be calculated.

8 Procedure

8.1 General

Quantitatively transfer the air-dried, ground, dichloromethane-extracted leather obtained from Clause 7 into a flask (6.1). Add (500 ± 10) ml of deionised water (5.2) at (23,5 ± 3,5) °C, close the stopper securely and shake mechanically (6.5) at (50 ± 10) cycles per minute for (120 min ± 10) min at (23,5 ± 3,5) °C (see 9.1).

Filter the contents of the flask through a fluted filter paper (6.7) until the filtrate is clear. Discard the first 50 ml. Determine the water-soluble organic matter and inorganic matter in subsequent 50 ml aliquot portions of the filtrate (see 9.2 and 9.3).

8.2 Water-soluble matter

Pipette 50,0 ml (6.3) of filtrate into a basin (6.4) which has previously been prepared by heating at 700 °C (6.10), cooled in a desiccator (6.11) and accurately weighed (6.12). Evaporate the filtrate over the water bath (6.8) and dry the residue at (102 ± 2) °C (6.9) for approximately 2 h. Cool in a desiccator, using only one basin at a time in a small desiccator, and no more than two in a large desiccator. Weigh quickly, and repeat the drying, cooling and weighing procedure either until the further reduction in mass does not exceed 2 mg, or the total drying time equals 8 h. Record the final mass and calculate the mass of dry residue (m_1).

8.3 Water-soluble inorganic matter

Thoroughly wet the residue obtained in 8.2 (see 9.2) in the basin (6.4) with just sufficient 1 mol/l sulfuric acid (5.1), and heat gently over a low flame until no more sulfur trioxide fumes are visible. Heat more strongly until the basin approaches red heat. Transfer to the muffle furnace (6.10) at 700 °C for 15 min (see 9.4). Cool in the desiccator and weigh as quickly as possible. Repeat the addition of acid, heating, cooling and weighing until the further reduction in mass does not exceed 2 mg, or the total drying time equals 8 h. Record the final mass and calculate the mass of the sulfated residue, (m_2).

9 Remarks on the procedure

9.1 If the specified extraction temperature of (23,5 ± 3,5) °C cannot be maintained in the test room, it is advisable to use a vacuum flask of 650 ml to 750 ml capacity. The range of values of the total water-soluble matter, $w_{T,ws}$, [see Clause 10 a)] is likely to differ by about 0,5 % over the permitted temperature range.

9.2 The water-soluble matter and the water-soluble inorganic matter can each be determined separately. Water-soluble matter can be determined by evaporating 50 ml portions of the filtrate in previously dried platinum, quartz, silver, porcelain or glass dishes at (102 ± 2) °C, in accordance with

8.2. Water-soluble inorganic matter can be determined by the evaporation of separate 50 ml portions in previously heated quartz, platinum or glazed porcelain dishes in accordance with 8.3.

9.3 If the mass of water-soluble inorganic matter is likely to be less than 2,0 % of the leather mass, it is recommended that a 100 ml or 200 ml aliquot portion should be used. In cases where the result is likely to be less than 1,0 %, a 100 ml or 200 ml portion should always be used.

9.4 At temperatures above 700 °C some loss of mass from the residue is possible, owing to volatilisation of certain inorganic salts. For this reason close control is essential to prevent the maximum furnace temperature from exceeding 700 °C.

10 Calculation and expression of results

Calculate the following percentages when the evaporated volume of water is 1/10 of the total volume. If other volumes are used then the factor 10 shall be changed appropriately.

a) Total water-soluble matter, $w_{T,ws}$ (mass fraction in per cent), see Formula (1):

$$w_{T,ws} = \frac{m_1 \times 10 \times 100}{m_0} \quad (1)$$

where

m_1 is the mass of the dry residue;

m_0 is the mass of the original sample of leather.

b) Water-soluble inorganic matter, $w_{I,ws}$, (mass fraction in per cent), see Formula (2):

$$w_{I,ws} = \frac{m_2 \times 10 \times 100}{m_0} \quad (2)$$

where

m_2 is the mass of sulfated residue;

m_0 is the mass of the original sample of leather.

c) Water-soluble organic matter, $w_{O,ws}$ (mass fraction in per cent). To obtain the percentage water-soluble organic matter, calculate the difference between the percentage total water-soluble matter and the percentage water-soluble inorganic matter, see Formula (3):

$$w_{O,ws} = \left(w_{T,ws}\right) - \left(w_{I,ws}\right) \quad (3)$$

If the results are to be reported on the basis of dry substance, the results above shall be multiplied by the factor 100/(100 − w), where w is the mass fraction of the volatile matter in per cent according to ISO 4684. If the results are presented on the basis of dry substance, this shall be clearly mentioned in the test report.

11 Test report

The test report shall include the following information:

a) reference to this document, i.e. ISO 4098;

b) the results obtained to one decimal place;

c) acknowledgement if the results are determined on the basis of the dry substance;

d) a description of the sample tested;

e) details of any deviations from the procedure, or special circumstances which may have affected the results.

12 Repeatability

The results of duplicate determinations should not differ by more than 0,2 %, calculated on the original mass of leather. If determinations differ by more than 0,2 %, then a further set of duplicate determinations shall be made and the results reported.