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Leather — Chemical determination of metal content —

Part 1: Extractable metals

Cuir — Détermination chimique de la teneur en métal —

Partie 1: Métaux extractibles

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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 Test 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).

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 17072-1:2011), which has been technically revised as follows:

— the Scope and Clauses 5, 6, 7, 8.3, 9 and 10 have been editorially modified;

— 7.2 and 8.2 have been technically modified. 7.2 refers to ISO 4044 for sample preparation, which requires the user to grind sufficiently large pieces of leather or cut small pieces of leather;

— the previous subclauses 6.3 and 7.3 from ISO 17072-1:2011 have been deleted.

A list of all parts in the ISO 17072 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Leather — Chemical determination of metal content —

Part 1:
Extractable metals

1 Scope

This document specifies a method for the determination of extractable metals in leather using extraction with an acid artificial-perspiration solution and subsequent determination with inductively coupled plasma optical emission spectrometry (ICP-OES), inductively coupled plasma mass spectrometry (ICP-MS), atomic absorption spectrometry (AAS) or spectrometry of atomic fluorescence (SFA).

This method determines extractable metals in leather; it is not compound-specific or specific to the oxidation state of the metals. This method is especially suitable for determining the extractable chromium in chromium-tanned leathers.

The method is applicable for the determination of many extractable metals, including:

Aluminium (Al)	Copper (Cu)	Nickel (Ni)
Antimony (Sb)	Iron (Fe)	Potassium (K)
Arsenic (As)	Lead (Pb)	Selenium (Se)
Barium (Ba)	Magnesium (Mg)	Tin (Sn)
Cadmium (Cd)	Manganese (Mn)	Titanium (Ti)
Calcium (Ca)	Mercury (Hg)	Zinc (Zn)
Chromium (Cr)	Molybdenum (Mo)	Zirconium (Zr)
Cobalt (Co)		

Interlaboratory test results and the quantification limits possible with ICP-OES are given in Table A.1 and Table A.2 of Annex A.

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 105-E04, *Textiles — Tests for colour fastness — Part E04: Colour fastness to perspiration*

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

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

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

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

ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)*

ISO 15586, *Water quality — Determination of trace elements using atomic absorption spectrometry with graphite furnace*

ISO 17294-2, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of selected elements including uranium isotopes*

ISO 17852, *Water quality — Determination of mercury — Method using atomic fluorescence spectrometry*

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Principle

A sample of leather is extracted at (37 ± 2) °C for 4 h ± 5 min in an acid artificial-perspiration solution. The extract solution is filtered, acidified and analysed by ICP, SFA or AAS.

The results are reported on the dry matter of the leather.

5 Reagents

WARNING — The concentrated acids used in this method are very corrosive and/or oxidising liquids, which could raise the possibility of fire in the event of contact with ignitable materials and promote an existing fire considerably, or could decompose explosively with warming. They might cause acute or chronic health dangers as well. Moreover, they are hazardous to water. Suitable safety measures are therefore necessary.

5.1 General

Unless otherwise stated, only analytical grade chemicals shall be used. All solutions are aqueous solutions.

5.2 Nitric acid, 60 % to 70 % concentration (by mass).

5.3 L-histidine monohydrochloride monohydrate.

5.4 Sodium chloride.

5.5 Sodium dihydrogen phosphate dihydrate.

5.6 Sodium hydroxide, 0,1 mol/l.

5.7 Element stock solutions, of the various metals with mass concentrations of 1 000 mg/l each.

5.8 Nitric acid, 0,1 mol/l.

5.9 Water, grade 3 in accordance with ISO 3696.

5.10 Solution of gold (Au), in hydrochloric acid or potassium permanganate, 1 000 µg/l.

6 Apparatus and materials

6.1 General

All glassware, analytical devices and materials, including filters, shall be suitable for analysis of trace metals.

Use normal laboratory apparatus and, in particular, the following.

6.2 Laboratory oven, capable of maintaining (102 ± 2) °C.

6.3 Analytical balance, with an accuracy of 0,1 mg.

6.4 Laboratory balance, with an accuracy of 0,01 g.

6.5 Membrane filters and holder, suitable for filtering aqueous solutions, with a pore size of 0,45 µm.

6.6 Filtration device, using glass fibre (GFC) or membrane type filters.

6.7 Volumetric flasks, of various sizes (50 ml, 100 ml, 1 000 ml).

6.8 Erlenmeyer flasks, of approximately 250 ml capacity, with stoppers.

6.9 Volumetric conical flasks, of various sizes.

6.10 Water bath, capable of maintaining (37 ± 2) °C, fitted with a horizontal flask shaker, frequency (60 ± 5) rpm, or orbital shaker, frequency (100 ± 10) rpm.

6.11 Inductively coupled plasma optical emission spectrometer (ICP-OES) (see ISO 11885), with a hydride-generator module. The gases used shall be of analytical grade.

6.12 Flame or graphite-furnace atomic absorption spectrometer (AAS) (see ISO 15586), with a hydride-generator module and suitable burner heads, and hollow-cathode-lamps. The gases used shall be of analytical grade.

6.13 Inductively coupled plasma mass spectrometer (ICP-MS) (see ISO 17294-2). The gases used shall be of analytical grade.

6.14 Atomic fluorescence spectrometer (SFA), for mercury analysis.

7 Sampling and sample preparation

7.1 If the leather piece available for testing is a whole hide or skin, then the test specimens shall be sampled in accordance with the standard procedures given in ISO 2418. If sampling in accordance with ISO 2418 is not possible (e.g. leathers are from finished products like shoes or garments), details about the sampling shall be given in the test report.

7.2 Prepare the leather sample in accordance with ISO 4044. Test pieces that are wet (in excess of 30 % moisture) should be pre-dried for at least 12 h, at a temperature not exceeding (50 ± 2) °C. The drying temperature should be selected while considering the influence of elevated temperature on the nature of the analyte.

7.3 Determine the volatile matter content in accordance with ISO 4684. The same sample of leather can be used for the extraction in 8.2.

8 Procedure

8.1 Preparation of acid artificial-perspiration solution

Preparation of acid artificial-perspiration solution, shall be in accordance with ISO 105-E04. The solution shall be freshly prepared each day, containing, per litre:

— 0,5 g of L-histidine monohydrochloride monohydrate (5.3);

— 5 g of sodium chloride (5.4);

— 2,2 g of sodium dihydrogen phosphate dihydrate (5.5).

The solution is brought to pH (5,5 ± 0,1) with 0,1 mol/l sodium hydroxide solution.

8.2 Extraction of leather sample

Weigh accurately 2 g of the prepared leather to the nearest 0,001 g using an analytical balance (5.3) and place in a 250 ml Erlenmeyer flask (5.8).

Add 100 ml of the acid artificial-perspiration solution (8.1) by pipette and, at (37 ± 2) °C, shake (6.10) in a water bath (6.10) for 4 h ± 5 min.

In case of insufficient sample, it is possible to sample 1 g of leather, but in this case the extraction shall be performed with 50 ml of the acid artificial-perspiration in a 150 ml Erlenmeyer flask.

Filter the extract with a filter paper (6.6), then filter with a membrane filter (6.5).

In the case of Sn, Sb, Pb determination, check that the filtration doesn't cause a partial loss of these elements.

For the direct measurement of the elements, take a suitable amount of extract for analysis and add 5 % (by volume) of nitric acid (5.2). Consider this addition in the dilution factor.

To control the contaminants, it will be necessary to carry out a blank procedure. An aliquot of acid perspiration is placed in a sample container and treated as a sample, in all respects, including all analytical procedures.

The above extraction conditions shall be followed exactly. Any deviation would lead to the wrong results.

In the case of mercury (Hg) determination, add 10 µl of gold solution (5.10) at the same time as the acid artificial-perspiration solution.

8.3 Analysis by ICP, AAS and SFA

8.3.1 General

The solution obtained in 8.2 can be analysed directly, provided it contains a concentration of analysed metals within calibration limits. Otherwise, the solution should be diluted as appropriate.

Prepare standard reference solutions of the required metals in accordance with ISO 11885, using perspiration solution (8.1), water or acidified water.

8.3.2 ICP

8.3.2.1 ICP-OES

Set up the ICP-OES (6.11) in accordance with the manufacturer's instructions and use the recommended settings indicated in ISO 11885.

Analyse the solution obtained in 8.2 against the reference solutions of metals with known concentration using ICP-OES (6.11) at the characteristic wavelength of each individual element.

8.3.2.2 ICP-MS

Set up the ICP-MS (6.13) in accordance with the manufacturer's instructions and use the recommended settings indicated in ISO 17294-2.

Analyse the solution obtained in 8.2 against the reference solutions of metals with known concentration using ICP-MS (6.13) at the characteristic ion mass of each individual element.

8.3.3 AAS

Prepare the atomic absorption spectrometer (6.12) following the manufacturer's instructions and use the recommended settings in accordance with ISO 15586.

Analyse the solution obtained in 8.2 against the reference solutions of metals with known concentration using AAS (6.12), with suitable hollow-cathode-lamp for each individual element.

8.3.4 Analysis by SFA technique

Set up the SFA (6.14) in accordance with the manufacturer's instructions and use the recommended settings indicated in ISO 17852.

Analyse the solution obtained in 8.2 against the reference solutions of mercury (Hg) with known concentration using SFA (6.14).

9 Calculation and expression of results

Express the result by stating the mass fraction (content) of the analysed metal, in milligrams per kilogram (mg/kg), calculated on the dry mass of the leather, as follows:

$$w_x = \frac{w_{x,\mathrm{i}}}{m} \times V_1 \times F_\mathrm{d}$$

where

w_x is the mass fraction of the metal extracted from the leather, expressed in milligrams per kilogram (mg/kg) of analysed product, and rounded to the nearest first decimal place;

$w_{x,\mathrm{i}}$ is the concentration of the metal in question determined by the instrument, expressed in milligrams per litre (mg/l);

m is the dry mass of the sample, expressed in grams (g), calculated in accordance with ISO 4684;

V_1 is the volume of the acid artificial-perspiration solution for the extraction, expressed in millilitres (ml);

F_d is the dilution factor eventually derived from the digestion procedure or acidification.