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IULTCS IUC 27-2

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Leather — Chemical determination of metal content —

Part 2: Total metal content

Cuir — Dosage chimique des métaux —

Partie 2: Teneur totale en métaux

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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 of 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 www.iso.org/iso/foreword.html.

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 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).

This third edition cancels and replaces the second edition (ISO 17072-2:2019), which has been technically revised. The main changes are as follows:

— Clause 1, Clause 6 and 8.1 have been editorially and technically modified;

— a new Annex B has been added describing the digestion procedure for the determination of aluminium and titanium.

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 2:
Total metal content

1 Scope

This document specifies a method for the determination of the total metal content in leather using digestion of the leather 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 the total metal content in leather. It is not compound-specific or specific to the oxidation state of the metals.

The method is applicable for determining the following metals:

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

This method is also suitable for determining Boron (B) in leather.

In the case of chromium-tanned leathers, it is often more relevant to use ISO 5398-1, ISO 5398-2, ISO 5398-3 or ISO 5398-4.

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

For the determination of Al and Ti in leather, a digestion procedure is given in Annex B.

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 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 terminology databases for use in standardization at the following addresses:

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

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

4 Principle

Digestion of the sample of leather (see ISO 4044) is carried out using a ternary acid mixture or microwave digestion until complete mineralization is achieved. The residue is redissolved with water and analysed by AAS, ICP or SFA (for mercury).

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 oxidizing liquids, which can raise the possibility of fire in the event of contact with ignitable materials and promote an existing fire considerably or can decompose explosively with warming. They can also cause acute or chronic health dangers. Moreover, they are hazardous to water. Suitable safety measures are therefore necessary.

Analytical grade chemicals shall be used for digestion with the Kjeldhal method. Ultrapure acid shall be used for microwave digestion. All solutions are aqueous solutions.

5.1 Nitric acid, 60 % to 70 % mass fraction, CAS Registry Number®[1)] (CAS RN®): 7697-37-2.

5.2 Sulfuric acid, 98 % mass fraction, CAS RN: 7664-93-9.

5.3 Perchloric acid, 60 % to 70 % mass fraction, CAS RN: 7601-90-3.

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

5.5 Hydrochloric acid, 37 % mass fraction, CAS RN: 7647-01-0.

5.6 Water, grade 3 in accordance with ISO 3696.

1) CAS Registry Number® (CAS RN®) is a trademark of CAS corporation. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5.7 Hydrofluoric acid, CAS RN: 7664-39-3.

6 Apparatus and materials

All glassware, analytical devices and the materials, including filters, shall be suitable for analysis of trace metals. In particular, it is known that aluminium can be released from some types of glassware. Do a blank test to select the most suitable glassware.

Normal laboratory apparatus and, in particular, the following shall be used.

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

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

6.3 Heating apparatus for Kjeldahl flasks, equipped with fume extraction.

6.4 Long-necked Kjeldahl digestion flask, 1 l volume, with reflux condenser.

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

6.6 Vacuum filter system for membrane filters.

6.7 Magnetic stirrer.

6.8 Glass boiling beads.

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

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

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

6.12 Atomic fluorescence spectrometer (SFA) (see ISO 17852), for mercury analysis.

6.13 Volumetric flasks, with a capacity of 50 ml and 100 ml.

6.14 Microwave-assisted digestion (MAD) apparatus.

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 such as 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 dry matter content in accordance with ISO 4684.

8 Procedure

8.1 Acid digestion

WARNING — It is imperative that the leather sample is not in direct contact with perchloric acid because of the possible explosive reaction.

Weigh accurately 1 g of the prepared leather to the nearest 0,001 g using an analytical balance (6.2) and place in a long-necked Kjeldahl digestion flask (6.4). Add, using a measuring cylinder, 10 ml to 20 ml of a ternary mixture of nitric acid (5.1), sulfuric acid (5.2) and perchloric acid (5.3) in a ratio of 3:1:1, and a few glass boiling beads (6.8). Place a funnel or splash bulb in the neck of the flask and heat to boiling. Leave to react on the heating apparatus (6.3) until digestion is complete and the red vapours of nitrogen dioxide have disappeared. Stop warming after the digestion is complete.

In the event of incomplete digestion, allow the flask to cool, add a further 10 ml to 20 ml of the ternary acid mixture and repeat the procedure.

If the digestion is still incomplete, use alternative methods of digestion (see 8.2) or transfer the solution into a plastic flask (or other inert material) and slowly add 1 ml of hydrofluoric acid (5.7), ensuring solution agitation.

In the case of high volatile metals determination, check that this open acid digestion does not cause a partial loss of these elements.

For lead (Pb) and barium (Ba) determination, the digestion procedure shall be carried out separately, replacing sulfuric acid (5.2) with hydrochloric acid (5.5).

For aluminium (Al) and titanium (Ti) determination, the digestion described is normally not complete. For a complete digestion, the procedure in Annex B shall be carried out separately,

Allow to cool, redissolve with 30 ml of distilled water, filter if necessary, then transfer the filtrate to a 100-ml volumetric flask. With 30 ml of distilled water, thoroughly wash the flask used for digestion and the filter, transfer the water to the volumetric flask and make up to volume.

To control the contaminants in the mixture of acids, it is necessary to carry out a blank procedure. An aliquot of the mixture of acids is placed in a sample container and treated as a sample in all respects, including all analytical process steps.

8.2 Microwave digestion

The sample for analysis can also be prepared through application of MAD (6.14) or other validated digestion techniques. If this is to be used, then the procedure and the sample quantity shall be adapted. Weigh 0,1 g to 1,0 g of the prepared leather to the nearest 0,001 g.

For aluminium (Al) and titanium (Ti) determination, the digestion described is normally not complete. For a complete digestion, the procedure in Annex B shall be carried out separately,

8.3 Analysis by ICP, AAS and SFA

8.3.1 General

Prepare standard reference solutions of the required metals in accordance with ISO 11885 or ISO 15586 by ensuring that the acid concentration in the standard reference solutions is of the same order as that

of the sample. For calibration, prepare at least four standard reference solutions plus a calibration blank.

8.3.2 ICP

8.3.2.1 General

The solution obtained in 8.1 or 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.

8.3.2.2 ICP-OES

Set up the ICP-OES spectrometer (6.9) in accordance with the manufacturer's instructions and use the recommended settings indicated in ISO 11885. If required, carry out the determination of As, Sb, Sn, Se and Hg using a hydride generator following the manufacturer's instructions.

Analyse the solution obtained in 8.1 or 8.2 against the reference solutions of metals with known concentration using ICP-OES (6.9) at the characteristic wavelength of each individual element.

8.3.2.3 ICP-MS

Set up the ICP-MS (6.11) in accordance with the manufacturer's instructions and use the recommended settings indicated in ISO 17294-2.

Analyse the solution obtained in 8.1 or 8.2 against the reference solutions of metals with known concentration using the ICP-MS (6.11) at the characteristic ion mass of each individual element.

8.3.3 AAS

Prepare the atomic absorption spectrometer (6.10) following the manufacturer's instructions and use the recommended settings in accordance with ISO 15586. If required, carry out the determination of As, Sb, Sn, Se and Hg by using a hydride generator following the manufacturer's instructions.

Analyse the solution obtained in 8.1 or 8.2 against the reference solutions of metals with known concentration using AAS (6.10), with a suitable hollow-cathode-lamp for each individual element.

8.3.4 Analysis by SFA technique

Set up the SFA (6.12) 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 with known concentration using SFA (6.12).

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 shown by Formula (1):

$$w_x \quad \frac{w_{x,\mathrm{i}}}{m} \times V_1 \times F_\mathrm{d} \tag{1}$$

where

w_x is the mass fraction of the metal in the leather, expressed in milligrams per kilogram (mg/kg) of analysed product, and rounded to two significant figures;

$w_{x,i}$ 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 volumetric flask used for the digestion, expressed in millilitres (ml);

F_d is the dilution factor, if the solution obtained in 8.1 needs to be diluted.

If required, the results can be given based on the dry, degreased mass of the leather sample. Details shall be noted in the test report.

10 Test report

The test report shall include at least the following information:

a) the name of the laboratory;

b) a reference to this document, i.e. ISO 17072-2:2022;

c) a description of the applied analytical system;

d) a description of the leather sample tested;

e) results of the dry matter determination;

f) results obtained for the amount of total metal, expressed in milligrams per kilogram of dry leather (mg/kg);

g) details of any deviations from this standard test method;

h) the date of the test.