



SLOVENSKI STANDARD
oSIST prEN ISO 17072-2:2021
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Usnje - Kemijsko določevanje kovin - 2. del: Celotni delež kovin (ISO/DIS 17072-2:2021)

Leather - Chemical determination of metal content - Part 2: Total metal content (ISO/DIS 17072-2:2021)

Leder - Chemische Bestimmung des Metallgehaltes - Teil 2: Gesamtmetallgehalt (ISO/DIS 17072-2:2021)

Cuir - Détermination chimique de la teneur en métal - Partie 2: Teneur totale en métaux (ISO/DIS 17072-2:2021)

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Leather — Chemical determination of metal content —
Part 2:
Total metal content

Cuir — Détermination chimique de la teneur en métal —
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ISO/DIS 17072-2:2021(E)
IUC 27-2:2021(E)**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).

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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. (standards.iteh.ai)

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 third edition cancels and replaces the second edition (ISO 17072-2:2019), which has been technically revised as follows:

- the Scope and [Clauses 6](#) and [8.1](#) have been editorially and technically modified;
- a new normative [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^[1], ISO 5398-2^[2], ISO 5398-3^[3] or ISO 5398-4^[4].

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

For the determination of Al and Ti in leather the extraction procedure given in [Annex B](#) shall be used.

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*

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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 <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 re-dissolved with water and analysed by AAS, ICP or SFA (for mercury).

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

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.

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

5.3 Sulfuric acid, 98 % concentration (by mass).

5.4 Perchloric acid, 60 % to 70 % concentration (by mass).

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

5.6 Hydrochloric acid, 37 %.

5.7 Water, grade 3 in accordance with ISO 3696.

6 Apparatus and materials

6.1 General

All glassware, analytical devices and the 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 Heating apparatus for Kjeldahl flasks, equipped with fume extraction.

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

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

6.7 Vacuum filter system for membrane filters.

6.8 Magnetic stirrer.

6.9 Glass boiling beads.

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

6.11 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.

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

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

6.14 Volumetric flasks, capacity 50 ml and 100 ml.

6.15 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 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.

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7.3 Determine the dry matter content in accordance with ISO 4684.

8 Procedure

8.1 Acid digestion

Weigh accurately 1 g of the prepared leather to the nearest 0,001 g using an analytical balance (6.3) and place in a long-necked Kjeldahl digestion flask (6.5). Add, using a measuring cylinder, 10 ml to 20 ml of a ternary mixture of nitric acid (5.2), sulfuric acid (5.3) and perchloric acid (5.4) in a ratio of 3:1:1, and a few glass boiling beads (6.9). Place a funnel or splash bulb in the neck of the flask and heat to boiling. Leave to react on the heating apparatus (6.4) until digestion is complete and the red vapours of nitrogen dioxide have disappeared. Stop warming after 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.

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

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

For lead (Pb) determination, the digestion procedure shall be carried out separately, replacing sulfuric acid (5.3) with hydrochloric acid (5.6).

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

Allow to cool, re-dissolve 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, it will be 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 procedures.

8.2 Microwave digestion

The sample for analysis can also be prepared through application of microwave-assisted digestion (MAD) (6.15) or other validated digestion technics. 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.

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.