INTERNATIONAL STANDARD

ISO 17072-2

IULTCS/IUC 27-2

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Leather — Chemical determination of metal content —

Part 2: Total metal content

Cuir — Détermination chimique de la teneur en métal —

Partie 2: Teneur totale en métaux

Reference number
ISO 17072-2:2011(E)
IULTCS/IUC 27-2:2011(E)

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 17072-2 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 289, *Leather*, in collaboration with the Chemical Test Commission of the International Union of Leather Technologists and Chemists Societies (IUC Commission, IULTCS), in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement). This method is technically similar to the method in IUC 27-2.

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.

ISO 17072 consists of the following parts, under the general title *Leather — Chemical determination of metal content*:

- *Part 1: Extractable metals*
- *Part 2: Total metal content*

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ISO 17072-2:2011(E)
IULTCS/IUC 27-2:2011(E)

Leather — Chemical determination of metal content —

Part 2:
Total metal content

1 Scope

This part of ISO 17072 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), or inductively coupled plasma/mass spectrometry (ICP/MS), or 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)

Antimony (Sb)

Arsenic (As)

Barium (Ba)

Cadmium (Cd)

Calcium (Ca)

Chromium (Cr) (except chromium-tanned leathers)

Cobalt (Co)

Copper (Cu)

Iron (Fe)

Lead (Pb)

Magnesium (Mg)

Manganese (Mn)

Mercury (Hg)

Molybdenum (Mo)

Nickel (Ni)

Potassium (K)

Selenium (Se)

Silicon (Si)

Sodium (Na)

Tin (Sn)

Titanium (Ti)

Zinc (Zn)

Zirconium (Zr)

The quantification limit of total lead is 8 mg/kg (see Annex A).

This part of ISO 17072 is not applicable to chromium-tanned leathers. In this case, ISO 5398-1, or ISO 5398-2, or ISO 5398-3, or ISO 5398-4 are used.

2 Normative references

The following referenced documents are indispensable for the application 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 3696:1987, *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 metals 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 62 elements*

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

EN 14602, *Footwear — Test methods for the assessment of ecological criteria*

3 Principle

Digestion of the sample of ground 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 or ICP.

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

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 together with the test report.

4 Reagents

WARNING — As concentrated acids are used, it is imperative to use all necessary personal protective equipment.

4.1 General

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

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

4.3 Sulfuric acid (H_2SO_4), 98 % concentration (by mass).

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

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

4.6 Hydrochloric acid (HCl), 37 %.

4.7 Water, grade 3 in accordance with ISO 3696:1987.

5 Apparatus and materials

5.1 General

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

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

5.2 Laboratory oven, capable of maintaining 102 °C ± 2 °C.

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

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

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

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

5.7 Vacuum filter system for membrane filters.

5.8 Magnetic stirrer.

5.9 Glass boiling beads.

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

5.11 Flame or graphite-furnace atomic absorption spectrometer (AAS) (see ISO 15586), with hydride-generator module and with a suitable hollow-cathode-lamp nitrous oxide burner head or high-solids nitrous oxide burner head. The gases used shall be of analytical grade.

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

5.13 Spectrometer of atomic fluorescence (SFA), for mercury analysis.

6 Sampling and sample preparation

6.1 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.

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

6.3 Determine the dry matter content in accordance with ISO 4684. The same sample of ground leather can be used for the digestion in 6.5.

6.4 Using the analytical balance (5.3), weigh accurately 1 g of the ground leather to the nearest 0,001 g and place in a long-necked Kjeldahl digestion flask (5.5).

6.5 Prepare the sample using the digestion method for the determination of Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Si, Sn, Ti, Zn, Zr, As, Se, Sb, Hg.

If using acid digestion (6.5.1) for Pb detection, the digestion procedure shall be carried out separately, replacing H_2SO_4 with HCl.

6.5.1 Acid digestion

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

Place the ground leather sample in the Kjeldahl flask (5.5), add, using a measuring cylinder, 10 ml to 20 ml of a ternary mixture of nitric acid (4.2), sulfuric acid (4.3) and perchloric acid (4.4) in a ratio of 3:1:1, and a few glass boiling beads (5.9). Place a funnel or splash bulb in the neck of the flask and heat to boiling on a wire gauze over a moderate flame. Leave to react on the heating apparatus (5.4) until digestion is complete and the red vapours of nitrogen dioxide have disappeared. In the event of incomplete digestion, allow the flask to cool, add a further 10 ml to 20 ml of the ternary mixture and repeat the procedure.

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, 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.

6.5.2 Microwave digestion

The sample for analysis can also be prepared through application of microwave-assisted digestion (MAD). If this is used, then a suitable procedure such as that described in EN 14602 shall be used.

7 Instrumental procedure

7.1 Analysis by ICP, AAS and SFA

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.

7.1.1 ICP

The filtrate solution can be analysed directly, provided it contains a concentration of analysed metals within detection limits. Otherwise, the solution should be diluted as appropriate.

7.1.1.1 ICP/OES

Set up the ICP/OES spectrometer (5.10) in accordance with the manufacturer's instructions and use the recommended settings indicated in ISO 11885.

Analyse the filtrate against the reference solutions of metals with known concentration using ICP/OES (5.10) at the characteristic wavelength of each individual element, either as it is or following an appropriate dilution.

7.1.1.2 ICP/MS

Set up the ICP/MS (5.12) in accordance with the manufacturer's instructions and use the recommended settings indicated in ISO 17294-2.

Analyse the filtrate against the reference solutions of metals with known concentration using the ICP/MS (5.12) at the characteristic ion mass of each individual element, either as it is or following an appropriate dilution.

7.1.2 AAS

Prepare the atomic absorption spectrometer (5.11) following the manufacturer's instructions and use the recommended settings in accordance with ISO 15586.

7.2 Analysis by hydride technique

The determination of As, Sb, Sn, Se and Hg can be carried out using a hydride generator, following the manufacturer's instructions.

7.3 Analysis by SFA technique

Hg analysis can be carried out using the SFA technique. If this is to be used, then the procedure described in ISO 17852 shall be followed.

8 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 in 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 detected 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.

It is possible, if required, to quote the results based on the dry, degreased mass of the leather sample. Details shall be noted in the test report.

9 Test report

The test report shall include at least the following information:

a) a reference to this part of ISO 17072 (i.e. ISO 17072-2:2011);

b) a description of the leather sample tested;

c) results of the dry matter determination;

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

e) details of any deviations from this standard test method.

Annex A
(informative)

Interlaboratory test results

Table A.1 — Lead interlaboratory test: statistical evaluation of results

Parameter	ISO 5725-2:1994	ISO 5725-5:1998
No. of considered laboratories	8	8
Overall mean value	162,31	163,48
Repeatability standard deviation	5,216	5,498
Relative repeatability standard deviation	3,21	3,36
Standard deviation between laboratories	27,5	64
Reproducibility standard deviation	28,053	29,093
Relative reproducibility standard deviation	17,28	17,80
Repeatability factor	14,76	15,56
Reproducibility factor	79,39	82,33

Bibliography

[1] ISO 5398-1, *Leather — Chemical determination of chromic oxide content — Part 1: Quantification by titration*

[2] ISO 5398-2, *Leather — Chemical determination of chromic oxide content — Part 2: Quantification by colorimetric determination*

[3] ISO 5398-3, *Leather — Chemical determination of chromic oxide content — Part 3: Quantification by atomic absorption spectrometry*

[4] ISO 5398-4, *Leather — Chemical determination of chromic oxide content — Part 4: Quantification by inductively coupled plasma — Optical emission spectrometer (ICP-OES)*

[5] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[6] ISO 5725-5:1998, *Accuracy (trueness and precision) of measurement methods and results — Part 5: Alternative methods for the determination of the precision of a standard measurement method*

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