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Läder – Kemisk bestämning av krom (VI) innehåll i läder – Del 2: Kromatografisk metod (ISO 17075-2:2017)

Leather – Chemical determination of chromium(VI) content in leather – Part 2: Chromatographic method (ISO 17075-2:2017)

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Denna standard ersätter SS-EN ISO 17075:2007, utgåva 1.

The European Standard EN ISO 17075-2:2017 has the status of a Swedish Standard. This document contains the official version of EN ISO 17075-2:2017.

This standard supersedes the Swedish Standard SS-EN ISO 17075:2007, edition 1.

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EUROPEAN STANDARD

EN ISO 17075-2

NORME EUROPÉENNE

EUROPÄISCHE NORM

February 2017

ICS 59.140.30

Supersedes EN ISO 17075:2007

English Version

**Leather - Chemical determination of chromium(VI)
content in leather - Part 2: Chromatographic method (ISO
17075-2:2017)**

Cuir - Détermination chimique de la teneur en
chrome(VI) du cuir - Partie 2: Méthode
chromatographique (ISO 17075-2:2017)

Leder - Chemische Bestimmung des Chrom(VI)-Gehalts
in Leder - Teil 2: Chromatographie (ISO 17075-2:2017)

This European Standard was approved by CEN on 30 December 2016.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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European foreword

This document (EN ISO 17075-2:2017) has been prepared by Technical Committee IULTCS "International Union of Leather Technologists and Chemists Societies" in collaboration with Technical Committee CEN/TC 289 "Leather" the secretariat of which is held by UNI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2017 and conflicting national standards shall be withdrawn at the latest by August 2017.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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Endorsement notice

The text of ISO 17075-2:2017 has been approved by CEN as EN ISO 17075-2:2017 without any modification.

Leather — Chemical determination of chromium(VI) content in leather —

Part 2: Chromatographic method

1 Scope

This document specifies a method for determining chromium(VI) in solutions leached from leather under defined conditions. The method described is suitable to quantify the chromium(VI) content in leathers down to 3 mg/kg.

This document is applicable to all leather types.

The results obtained from this method are strictly dependent on the extraction conditions. Results obtained by using other extraction procedures (extraction solution, pH, extraction time, etc.) are not comparable with the results produced by the procedure described in this document.

If a leather sample is tested with both ISO 17075-1 and this document, the results obtained with this document are considered as the reference. The advantage of the method described in this document is that there are no interferences from the colour of the extract. Nevertheless, interlaboratory trials do not show significant differences (see [Annex D](#)) and the results are comparable between both methods.

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 4044:2017, *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 <http://www.iso.org/obp>

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

3.1

chromium(VI) content

amount of chromium(VI) in leather determined after extraction with an aqueous salt solution at pH 7,0 to 8,0

Note 1 to entry: The chromium(VI) content is reported as chromium(VI) in milligrams per kilogram (mg/kg), expressed as the dry mass of the sample.

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4 Principle

Extractable chromium(VI) is leached from the sample in phosphate buffer at pH 7,0 to 8,0. An aliquot of the filtered extract is analysed for Cr(VI) using ion-exchange chromatography with UV-VIS detection.

5 Chemicals

All reagents used shall have at least analytical grade purity.

5.1 Extraction solution

Dissolve 22,8 g dipotassium hydrogenphosphate $K_2HPO_4 \cdot 3H_2O$ in 1 000 ml water (5.7), adjusted to pH $8,0 \pm 0,1$ with phosphoric acid (5.2). Degas this solution with either argon or nitrogen (5.6) or ultrasonic bath.

Standard practice is to make up a fresh solution each day. However, the solution can be kept for up to one week in a refrigerator at $(4 \pm 3) ^\circ C$ but shall be warmed to room temperature and degassed prior to use.

5.2 Phosphoric acid solution

700 ml *o*-phosphoric acid, $\rho = 1,71$ g/ml, made up to 1 000 ml with water (5.7).

First add approximately 200 ml of deionised water (5.7) to a 1 000 ml volumetric flask, then add the 700 ml of *o*-phosphoric acid and dilute to the mark with deionised water.

5.3 Potassium dichromate ($K_2Cr_2O_7$), dried for (16 ± 2) h at $(102 \pm 2) ^\circ C$.

5.4 Chromium(VI) stock solution

Dissolve 2,829 g potassium dichromate ($K_2Cr_2O_7$) (5.3) in water (5.7) in a volumetric flask and make up to 1 000 ml with water (5.7). One millilitre of this solution contains 1 mg of chromium.

A stock solution at this concentration level of hexavalent chromium is an alternative available commercially.

5.5 Chromium(VI) standard solution

Pipette 1 ml of solution (5.4) into a 1 000 ml volumetric flask and make up to the mark with extraction solution (5.1). One millilitre of this solution contains 1 μg of chromium.

The solution can be kept for up to one week in a refrigerator at $(4 \pm 3) ^\circ C$ but shall be warmed to room temperature prior to use.

A stock solution of hexavalent chromium at this concentration level is an alternative available commercially.

5.6 Argon or nitrogen, oxygen-free

Preference should be given to argon as an inert gas instead of nitrogen because argon has a higher specific mass than air.

5.7 Distilled or deionised water, Grade 3 quality as specified in ISO 3696.

6 Apparatus and materials

Usual laboratory equipment and, in particular, the following.

- 6.1 **Suitable mechanical orbital shaker**, $(100 \pm 10) \text{ min}^{-1}$.
- 6.2 **Conical flask**, of capacity 250 ml, with stopper.
- 6.3 **Aeration tube** and **flow meter**, suitable for a flow rate of $(50 \pm 10) \text{ ml/min}$.
- 6.4 **Membrane filter**, $0,45 \mu\text{m}$ pore size [polytetrafluoroethylene (PTFE) or polyamide 66].
- 6.5 **Common laboratory glassware** and **pipettes**.
- 6.6 **Ion-exchange chromatograph, with UV detector** or **high performance liquid chromatograph (HPLC) with anion-exchange column and UV detector**. It is recommended a photo diode array detector (DAD).
- 6.7 **Analytical balance**, capable of weighing to 0,1 mg.
- 6.8 **Suitable vials for HPLC**.
- 6.9 **Sharp cutting tool** or **blade**, suitable for cutting the leather into 3 mm to 5 mm pieces.

7 Procedure

7.1 Sampling and preparation of samples

If possible, sample in accordance with ISO 2418. If sampling in accordance with ISO 2418 is not possible (e.g. leathers from finished products like shoes, garments), details about sampling shall be given in the test report.

Prepare the leather sample by cutting (6.9) into small pieces according to the method specified in ISO 4044:2017, 6.3.

7.2 Preparation of analytical solution

Weigh (6.7) approximately $(2 \pm 0,1) \text{ g}$ of leather pieces to the nearest 0,001 g. Pipette 100 ml of degassed solution (5.1) into a 250 ml conical flask (6.2). Displace oxygen by passing oxygen-free argon (or nitrogen) (5.6) into the flask for 5 min with a volume flow of $(50 \pm 10) \text{ ml/min}$. Remove the aeration tube (6.3), add the leather and close the flask with a stopper. Record the extract volume as V_0 .

Shake the conical flask with the leather pieces for $3 \text{ h} \pm 5 \text{ min}$ on a mechanical orbital shaker at $(100 \pm 10) \text{ min}^{-1}$ (6.1) at room temperature to extract the chromium(VI).

Shake the suspension in a smooth circular movement to keep the leather pieces from adhering to the wall of the flask and avoid shaking faster than specified.

Immediately after completing the 3 h of extraction, filter the contents of the conical flask through a membrane filter into a glass or plastic vessel with lid. Check the pH of the solution. The pH of the solution shall be between 7,0 and 8,0. If the pH of the solution is not within this range, start the complete procedure again.

Consider using a smaller sample mass, if the pH is not between 7,0 and 8,0. In this case, the quantification limit will be increased.

Transfer an aliquot of the filtered extract into a vial (6.8).