

INTERNATIONAL
STANDARD

ISO
14088

IULTCS/IUC 32

Second edition
2020-03

**Leather — Chemical tests —
Quantitative analysis of tanning agents
by filter method**

*Cuir — Essais chimiques — Analyse quantitative des agents de
tannage par la méthode au filtre cloche*

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Reference numbers
ISO 14088:2020(E)
IULTCS/IUC 32:2020(E)

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Published in Switzerland

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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.

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 14088:2012), which has been technically revised.

The main changes to the previous edition are as follows:

- [Clause 8](#) (Clause 7 in the first edition) has been extensively revised;
- Annex A from the first edition has been deleted.

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 tests — Quantitative analysis of tanning agents by filter method

1 Scope

This document specifies a test method for the determination of tanning agents through filtration of all vegetable and synthetic tanning products.

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

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

Indirect gravimetric analysis of vegetable and synthetic tanning agents through fixing of the absorbent compounds on low-chromed hide powder.

5 Reagents

5.1 Distilled water, freshly prepared, grade 3 according to ISO 3696.

The pH value of the water shall be between 5 and 7. When using methyl red, the water should not turn red. The evaporation residue of 100 ml should be less than 1 mg.

5.2 Hide powder, containing less than 0,8 % chromium oxide and with a moisture content no more than 13 %.

The blank value of the hide powder shall be calculated according to [Annex A](#). Examples of commercial suppliers of certified hide powder are listed in [Annex B](#)

NOTE In addition, the bulk density (between 0,07 g/ml and 0,23 g/ml), ash content (max: 1,2 %) and particle size of the hide powder can be measured.

5.3 Gelatine solution, of 1 g gelatine and 10 g sodium chloride, filled up to 100 ml with distilled water, adjusted to pH = 4,7.

5.4 Formic acid, technical grade.

6 Apparatus

The glass equipment shall be resistant to the action of distilled water. The flasks and tubes shall be Class A.

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

6.1 Desiccator, with an airtight cover and containing silica gel.

6.2 Evaporation dishes, suitable for slowly evaporating water.

These shall be short with flat bases and measure 7 cm to 8,5 cm in diameter.

Use silver dishes. If this is not possible, preferably use dishes made of stainless steel or, if necessary, ceramic or glass.

6.3 Water bath.

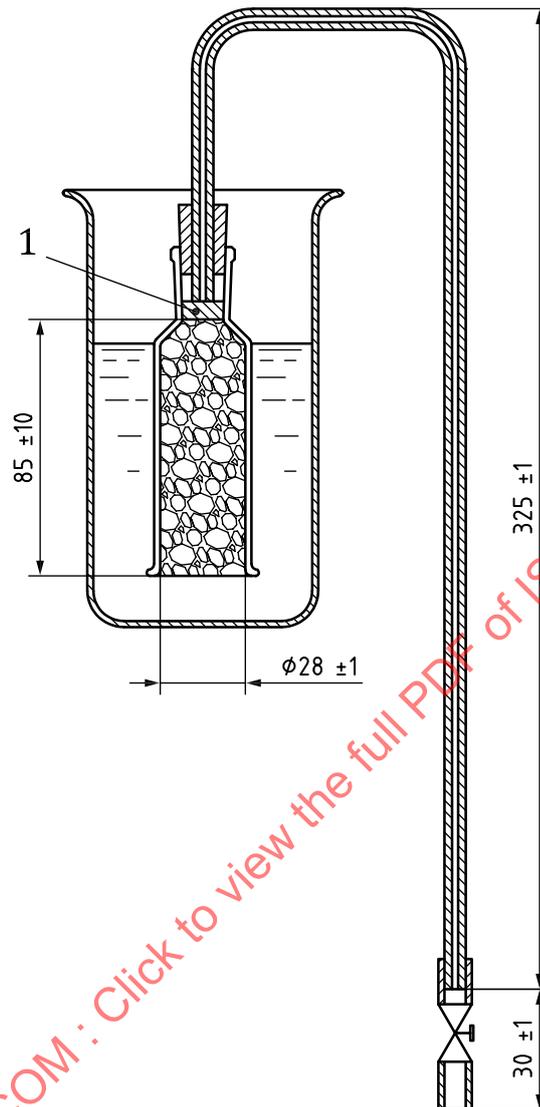
6.4 Drying oven, whose temperature shall be kept in the operating range of (102 ± 2) °C.

6.5 Analytical balance, with a precision of 0,2 mg at a load of 200 g.

6.6 Technical balance, with a precision of 0,1 g at a load of 1 000 g.

6.7 Procter bell (see [Figure 1](#)), composed of a cylindrical glass bell; length of the cylindrical part, (85 ± 10) mm; internal diameter of the cylindrical part, (28 ± 1) mm. A perforated rubber cork is inserted into the narrow part of the bell. A capillary glass tube [internal diameter (1,0 to 1,5) mm] with two right-angled bends is inserted into the hole in the cork as shown in [Figure 1](#). The end of the shortest part can fit right down to the base of the cork.

Dimensions in millimetres



Key

- 1 layer of cotton wool

Figure 1 — Procter bell

6.8 Polyethylene tube, the tube shall be the right size to fit onto the bell's capillary glass tube.

6.9 Hoffman clamp.

6.10 1 000 ml volumetric flasks.

6.11 50 ml pipette.

6.12 Vacuum filter system (for example, see [Figure 2](#)) including a 47 mm sintered glass vacuum filter holder with base, a glass funnel, clamp, stopper and the vacuum filtering flask.

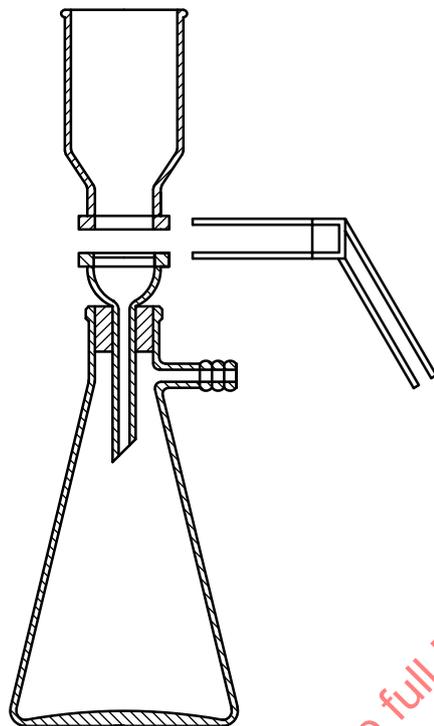


Figure 2 — Vacuum filter system

6.13 Cellulose acetate membrane filters, with pore sizes of 0,45 μm and 3 μm .

6.14 50 ml and 100 ml measuring cylinders.

6.15 Rubber pipette filler.

7 Sampling and sample preparation

There should be a generous, representative sample of the tanning agents for analysis. The sample should be thoroughly mixed.

If the particles are heterogeneous, resort to manual or mechanical milling to homogenize the size of the particles.

8 Instrumental analysis

8.1 Preparation of the analytical solution

8.1.1 Vegetable tanning agents

Weigh the appropriate quantity of vegetable tanning agent on an analytical balance ([6.5](#)), taking into account the expected tannin content in order to have the analytical solution containing $(4,00 \pm 0,25)$ g/l of tannin in water. If the tannin content in the solution goes beyond these limits, repeat the analysis with a sample of suitable quantity.

Dissolve the tanning material in a vessel with hot distilled water (60 °C to 80 °C) and transfer without lumps into the 1 000 ml flask. Rinse the vessel with water and transfer to the flask until approximately

800 ml to 900 ml is in the flask. Shake the flask manually to fully dissolve the tanning material. Leave the flask at room temperature for not more than 30 min and cool down in a water bath at $(21 \pm 3) ^\circ\text{C}$. Add distilled water (5.1) up to the mark. The freshly prepared analytical solution shall be used within two hours.

8.1.2 Synthetic tanning agents

Weigh $(4 \pm 0,1)$ g of tanning agents in powder form or $(8 \pm 0,1)$ g of tanning agents in liquid form on an analytical balance (6.5).

Dissolve the tanning material in a vessel with warm distilled water ($40 ^\circ\text{C}$ to $50 ^\circ\text{C}$) and transfer without lumps into the 1 000 ml flask. Rinse the vessel with water and transfer to the flask until approximately 800 ml to 900 ml is in the flask. Shake the flask manually to fully dissolve the tanning material. Leave the flask at room temperature for not more than 30 min and cool down in a water bath at $(21 \pm 3) ^\circ\text{C}$. Add distilled water (5.1) up to the mark. The freshly prepared analytical solution shall be used within two hours.

8.2 Preparation of the Procter bell

Place a layer of cotton wool at the top of the bell (6.7) to prevent the hide powder from entering the capillary tube.

Weigh 7,0 g of hide powder (5.2) on a technical balance (6.6). Remove lumps in the hide powder by separating them manually, and introduce the hide powder uniformly in the bell, pressing it down, up to the top of the rim. Check that the hide powder is fully pressed down to ensure that it will be completely tanned.

Put the rubber cork containing the glass capillary tube in the bell.

To adjust the flow, a polyethylene tube can be used with the glass capillary tube and the Hoffman clamp (6.9).

8.3 De-tanning the analytical solution (determination of the non-tanning agents)

Place the bell (6.7) containing hide powder in a beaker of suitable capacity. Fill the beaker with the unfiltered analytical solution up to the neck of the bell. Wait approximately 30 min to completely soak the hide powder, suck on the longer end of the capillary tube with a rubber pipette filler (6.15) to create a slight pressure difference and start siphoning the solution.

If using the polyethylene tube (6.8) and the Hoffman clamp (6.9) as shown in Figure 1, adjust the flow of the solution so that approximately 8 to 10 drops of the de-tanned solution siphon through per minute. The resulting solution shall be clear.

NOTE The Hoffman clamp does not correct eventual errors of preparation of the Procter bell.

Collect a total of approximately 90 ml solution in (120 ± 10) min.

The first approximately 30 ml of the filtrate solution should be collected in a 50 ml glass measuring cylinder (6.14) and disposed of.

The next approximately 60 ml should be collected in a perfectly dry 100 ml glass measuring cylinder (6.14) to determine the non-tanning agents. To control the possible breakthrough of tanning agents use 5 ml of the collected solution and add 0,5 ml gelatine solution (5.3). The pH of the total solution should be lower than 5. If necessary, use a few drops of formic acid to reduce the pH. A white precipitate is an indication of a breakthrough. In this case, repeat the analysis with a lower sample mass.

The solution should be at a temperature no less than $18 ^\circ\text{C}$ and no more than $24 ^\circ\text{C}$.

Use the pipette (6.11) to transfer 50 ml of the filtered solution into a previously dried and weighed silver dish (6.2).

Place the dish on the water bath (6.3) and wait for complete evaporation.

Put the dish (6.2) in the oven (6.4) at (102 ± 2) °C to attain constant mass (about 18 h \pm 2 h).

Put the dish (6.2) in the silica gel desiccator (6.1) and weigh it after 15 min on an analytical balance (6.5).

8.4 Determination of soluble substances

To filter the analytical solutions, use the filter system (6.12) indicated in Figure 2.

Use the cellulose acetate membranes with 0,45 µm pore size (6.13). The filtration shall last less than 5 min.

If filtration proves awkward, use membranes with 3,0 µm pores to pre-filter. If filtrations need more than 5 min, substitute the membrane with a new one.

If the filtration is not possible easily, the solution should be previously centrifuged using a stopper to prevent evaporation of the analytical solution.

In any case, the analytical solution shall be filtered through the membrane with 0,45 µm pore size.

Collect about 100 ml of filtrate.

Use the pipette (6.11) to transfer 50 ml of the filtered solution into a previously dried and weighed silver dish (6.2).

Place the dish on the water bath (6.3) and wait for complete evaporation.

Put the dish (6.2) in the oven (6.4) at (102 ± 2) °C to attain constant mass, for approximately (18 h \pm 2) h.

Put the dish (6.2) in the silica gel desiccator (6.1) and weigh it after 15 min on an analytical balance (6.5).

8.5 Determination of total solids

Use the pipette (6.11) to transfer 50 ml of the analytical solution into a previously dried and weighed silver dish (6.2).

Place the dish on the water bath (6.3) and wait for complete evaporation.

Take care to transfer homogeneously the analytical solution in the dish. If there is too much insoluble matter and if it is not possible to transfer it effectively and representatively with the 50 ml pipette, add 1 g to 3 g of the sample into the dish as an alternative.

Put the dish (6.2) in the oven (6.4) at (102 ± 2) °C to attain constant mass, approximately (18 \pm 2) h. In the case of product in powder form directly in the dish, the heating should be done without air circulation.

Weigh the dish (6.2) with the analytical balance (6.5).

Put the dish (6.2) in the silica gel desiccator (6.1) and weigh it after 15 min on an analytical balance (6.5).

9 Calculation and expression of the results

Calculate the percentage content of dry substances (S_{to}) (% total solid) using [Formula \(1\)](#).

$$S_{to} = \frac{m_1 \times 100}{m_{p0}} \quad (1)$$

where

S_{to} is the percentage content of dry substances (% total solid);

m_1 is the dry residue as determined at [8.5](#), in grams (g);

m_{p0} is the product's mass (see [8.5](#)), in grams (g).

Calculate the percentage of soluble solids (S_{so}) using [Formula \(2\)](#).

$$S_{so} = \frac{m_2 \times 20 \times 100}{m_{p1}} \quad (2)$$

where

S_{so} is the percentage content of soluble solids (%);

m_2 is the dry residue of 50 ml of the filtered analytical solution, in grams (g);

m_{p1} is the initial mass of the product (analytical solution), in grams (g).

Calculate the percentage of non-tanning solid (S_{nt}) using [Formula \(3\)](#).

$$S_{nt} = \frac{m_3 \times 20 \times 100}{m_{p1}} \quad (3)$$

where

S_{nt} is the percentage of non-tanning solid (%);

m_3 is the dry residue of the non-tanning agent solution, deducted from the blank value, in grams (g);

m_{p1} is the initial weighing of the product (analytical solution), in grams (g).

If certified hide powder is used, subtract the blank value on the certificate provided with the purchased batch. If the powder comes from another supplier or is not certified, the blank value shall be calculated according to the method in [Annex A](#).

Calculate the percentage of tanning agents (T) using [Formula \(4\)](#).

$$T = S_{s0} - S_{nt} \quad (4)$$

Calculate the percentage of insoluble matter (I_M) using [Formula \(5\)](#).

$$I_M = S_{t0} - S_{s0} \quad (5)$$

Calculate the percentage of water (W) using [Formula \(6\)](#).

$$W = 100 - S_{t0} \quad (6)$$

Calculate the ratio of tanning agent to non-tanning agent (R) using [Formula \(7\)](#).

$$R = \frac{T}{S_{nt}} \quad (7)$$

If there are reasons to doubt the tanning action of a product, one can avoid using the tanning agent on the test report by replacing “% tanning agent” with “% substances absorbed by hide powder”.

10 Test report

The test report shall include the following:

- a) a reference to this document, i.e. ISO 14088:2020;
- b) the origin and batch of the hide powder;
- c) any deviation from the analytical procedure;
- d) percentage content of dry substances (S_{t0}) (% total solid) to the nearest 0,1 %;
- e) percentage of soluble solids (S_{s0}) (%) to the nearest 0,1 %;
- f) percentage of non-tanning solid (S_{nt}) (%) to the nearest 0,1 %;
- g) percentage of tanning agents (T) (%) to the nearest 0,1 %;
- h) percentage of insoluble matter (I_M) (%) to the nearest 0,1 %;
- i) percentage of water (W) (%) to the nearest 0,1 %;
- j) ratio of tanning agent to non-tanning agent (R).