



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

Cuir — Essais chimiques — Analyse quantitative des agents tannants par la méthode au filtre-cloche

ICS 59.140.30

ISO/CEN PARALLEL PROCESSING

This draft has been developed within the European Committee for Standardization (CEN), and processed under the **CEN-lead** mode of collaboration as defined in the Vienna Agreement.

This draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel five-month enquiry.

Should this draft be accepted, a final draft, established on the basis of comments received, will be submitted to a parallel two-month approval vote in ISO and formal vote in CEN.

This draft International Standard is submitted to all ISO member bodies for voting, as a standard prepared by an international standardizing body in accordance with Council Resolution 42/1999. The proposer, *Chemical Test Commission of the International Union of Leather Technologists and Chemists Societies (Commission IUC, IULTCS)*, has been recognized by the ISO Council as an international standardizing body for the purpose of Council Resolution 42/1999.

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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 14088 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).

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

1 Scope

This International Standard describes a test method for the determination of tanning agents in all vegetable and synthetic tanning products.

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*

3 Principle

Indirect gravimetric analysis of vegetable tanning agents with fixing of the absorbent compounds in low-chromed hide powder.

4 Reagents

4.1 Distilled water

4.2 Hide powder¹⁾

5 Equipment

5.1 The glass equipment shall be resistant to the action of distilled water. The flasks and tubes shall be Class A. Normal laboratory equipment, and in particular:

5.2 **Dryers**, shall have airtight covers and contain silica gel.

5.3 **Evaporation dishes**, shall be short with flat base and measure 7 to 8.5 centimetres in diameter.

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

¹⁾ You can purchase hide powder with certificate for analysis of substances released by hide powder in contact with distilled water (blank value) from FILK Postfach, 48 Freiberg, 09581, Germany, Phone: +49 (0)3731 366-165, ax: +49 (0)3731 366-130

5.4 Water bath

5.5 Thermostatic heater, which temperature shall be kept at the operating range of $(105 \pm 2)^\circ\text{C}$

5.6 Balances, analytical balance with precision of 0,2 mg at a load of 100g. Technical balance with precision of 0,1g at a load of 1000g.

5.7 Procter bell (see Figure 1), composed of a cylindrical glass bell (length of the cylindrical part is 90 ± 1 mm, internal diameter of the cylindrical part $28 \text{ mm} \pm 2 \text{ mm}$) and a narrow part in which is inserted a perforated rubber cork. The hole in the cork takes a capillary glass tube (internal diameter 1,5 mm) with two right-angled bends. The end of the shortest part can fit right down to the base of the cork.

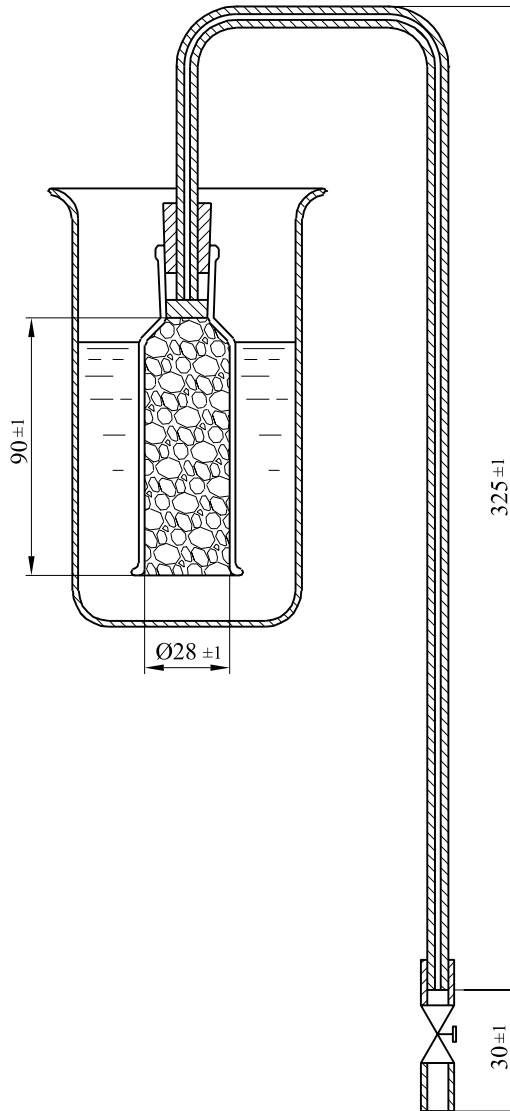


Figure 1 — Procter bell

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

5.9 Hoffman clamp

5.10 1000 ml calibrated flasks

5.11 50 ml pipette

5.12 Vacuum filter system

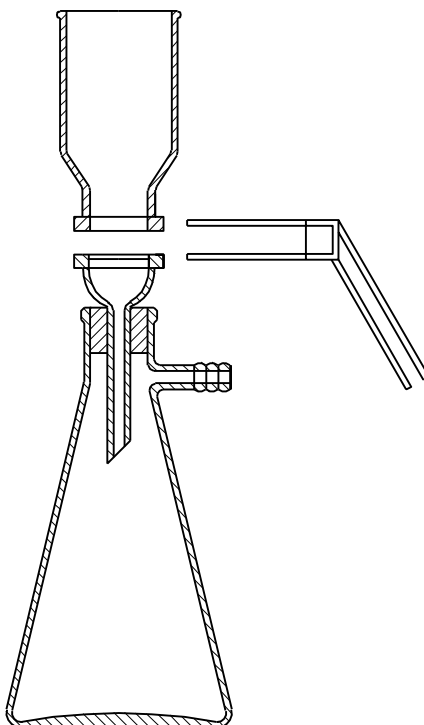


Figure 2 — Vacuum filter system

5.12 Cellulose acetate membrane filters, with pores of 0,45 μm and 3 μm .

5.13 50ml and 100 ml measuring cylinders.

6 Sampling and sample preparation

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

If the particles are any larger than 300 μm or are heterogeneous, resort to manual or mechanical milling to reduce the size of the particles as required.

7 Instrumental analysis

7.1 Preparation of the analytical solution

7.1.1 Vegetable tanning agents in powder/solid form

Weigh the appropriate quantity of vegetable tanning agents on a analytical balance (5.6) to obtain an analytical solution containing between 3,75 and 4,25 grams of substances absorbed by the hide powder (see Annex 1) and add it to 800 ml of hot distilled water (60-80) $^{\circ}\text{C}$ in a calibrated 1000 ml flask (5.10). Shake to fully dissolve the tanning agents. There may be some residue if there is any insoluble matter in the sample. Leave to cool down in water bath at (20 \pm 2) $^{\circ}\text{C}$ and add distilled water up to the mark (4.1).

If the tanning content in the solution goes beyond these limits, repeat the analysis with a sample of suitable quantity.

7.1.2 Vegetable tanning agents in liquid form

Weigh the tanning agents on a analytical balance (5.6), taking into account the percentage of content in dry form so as to obtain an analytical solution containing between 3,75 and 4,25 grams of substances absorbed in the hide powder. Add it to 800 ml of hot distilled water (60-80)°C in a 1000 ml calibrated flask (5.10). Shake to fully dissolve the tanning agents. There may be some residue if there is any insoluble matter in the sample. Leave to cool down in water bath at (20 ± 2)°C and add distilled water up to the mark (4.1).

If the tanning content in the solution goes beyond these limits, repeat the analysis with a sample of suitable quantity.

7.1.3 Synthetic tanning agents in powder form

Weigh about (4 ± 0,1)g of tanning agents on a analytical balance (5.6). Add it to 800 ml of hot distilled water (40-50)°C in a 1000 ml calibrated flask (5.10). Shake to fully dissolve the tanning agents. There may be some residue if there is any insoluble matter in the sample. Leave to cool down in water bath at (20 ± 2)°C and add distilled water up to the mark (4.1).

7.1.4 Synthetic tanning agents in liquid form

Weigh about (8 ± 0,1)g of tanning agents on a analytical balance (5.6). Add it to 800 ml of hot distilled water (40-50)°C in a 1000 ml calibrated flask (5.10). Shake to fully dissolve the tanning agents. There may be some residue if there is any insoluble matter in the sample. Leave to cool down in water bath at (20 ± 2)°C and add distilled water up to the mark (4.1).

7.1.5 Vegetable tanning agents organic solvent extracted in powder form

Weigh about (4 ± 0,1)g of tanning agents on a analytical balance (5.6). Add it to 800 ml of hot distilled water (40-50)°C in a 1000 ml calibrated flask (5.10). Shake to fully dissolve the tanning agents. There may be some residue if there is any insoluble matter in the sample. Leave to cool down in water bath at (20 ± 2)°C and add distilled water up to the mark (4.1).

7.2 Preparation of the bell

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

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

Weight on a technical balance (5.6) 7,0 g of hide powder (4.2) and introduce it uniformly in the bell pressing it down, up to the top of the rim.

Check it is all fully pressed down to ensure the analytical solution will all be tanned.

Put the polyethylene tube in the glass capillary tube and use the Hoffman clamp (5.9).

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

Place the bell containing hide powder in a beaker of suitable capacity. Fill the beaker with the unfiltered analytical solution up to the neck of the bell. When the hide powder is completely soaked, suck on the longer end of the capillary tube to create a slight depression and start siphoning the solution.

Use the Hoffman clamp (5.9) to adjust the flow of the solution so that about 8-10 drops of the de-tanned solution drip through per minute.

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

The first 30 ml of the filtrate should be collected in a 50 ml glass measuring cylinder (5.13) and disposed of.

The next 60 ml should be collected in a perfectly dry 100 ml glass measuring cylinder (5.13) to determine the non-tanning agents.

The solution should be at a temperature no less than 18 °C and no more than 25 °C.

Use the pipette (5.11) to transfer 50 ml of the filtered solution into a previously calibrated silver dish (5.3).

Place the dish on the water bath (5.4) and wait for complete evaporation.³

Put the dish (5.3) in the thermostatic heater (5.5) at (105 ± 2) °C to attain constant weight (about 18h \pm 2h).

Put the dish (5.3) in the silica gel dryer (5.2) and weight if after 15 min on analytical balance (5.6).

7.4 Determination of soluble substances

To filter the analytical solutions, use the filter system (5.9) indicated in figure 2.

Use the cellulose acetate membranes with 0,45 μ m pores.

If filtration proves awkward, use membranes with 3,0 μ m pores and then pass the pre-filtered solution through the 0,45 μ m membranes.

Collect about 100 ml of filtrate.

Use the pipette (5.11) to transfer 50 ml of the filtered solution into a previously calibrated silver dish (5.3).

Place the dish on the water bath (5.4) and wait for complete evaporation.

Put the dish (5.3) in the thermostatic heater (5.5) at (105 ± 2) °C to attain constant weight (about 18h \pm 2h).

Put the dish (5.3) in the silica gel dryer (5.2) and weight if after 15 min on analytical balance (5.6).

7.5 Determination of total solids

Calibrate a silver dish (5.3) in the thermostatic heater (5.5) at 105 ± 2 °C and then cool it off in the dryer (5.2), for about 15 min, down to ambient temperature.

Weigh the dish (5.3) with the analytical balance (5.6).

Add about 3g-5 g of the sample to the dish.

Put the dish (5.3) in the thermostatic heater (5.5) at (105 ± 2) °C to attain constant weight (about 18h \pm 2h).

Put the dish (5.3) in the silica gel dryer (5.2) and weight if after 15 min on analytical balance (5.6).

7.6 Expression of the results

Results are expressed as follows:

$$\% \text{ content of dry substance (\% total solids)} \quad \frac{g1 \times 100}{P^0}$$

$$\% \text{ soluble solids} \quad \frac{g2 \times 20 \times 100}{P1}$$

$$\% \text{ non-tanning solids} = \frac{g3 \times 20 \times 100}{P1}$$

where:

g1 is the dry residue as determined at clause 7.5

g2 is the dry residue of 50 ml of the filtered analytical solution

g3 is the dry residue of the non-tanning agent solution, deducted from the blank value.²

P⁰ is the product's weight (clause 7.5)

P1 is the initial weighing of the product (analytical solution)

$$\% \text{ Tanning agents (\% vegetable substances)} = \% \text{ soluble solids} - \% \text{ non-tanning solids}$$

$$\% \text{ Insoluble matter} = \% \text{ total solids} - \% \text{ soluble solids}$$

$$\% \text{ Water} = 100 - \% \text{ total solids}$$

$$\text{Tanning agent/non-tanning agent ratio} = \frac{\% \text{ Tanning agent}}{\% \text{ non - tanning agent}}$$

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

8 Test report

The test report shall include the following:

- a) reference to this document (i.e. ISO 14088);
- b) the origin and batch of the hide powder;
- c) any deviation from the analytical procedure.

² If you use hide powder provided by Filk, subtract the Blind value on the certificate provided with the purchased batch. If instead the powder comes from another supplier or is not certified, you need to calculate the blind value according to the method in Annex 2.

Annex A
(informative)

**Approximate quantity of vegetable tannic extract agent in
powder/solid form to be weighted**

TANNING AGENTS %	APPROX AMOUNT TO WEIGH g
50	8,0
55	7,3
60	6,5
65	6,1
70	5,7
75	5,3
80	5,0

Annex B (informative)

Determination of hide powder blank value

Place the bell containing hide powder in a beaker of suitable capacity. Fill the beaker with distilled water up to the neck of the bell.

When the hide powder is completely soaked, suck on the longer end of the capillary tube to create a slight depression and start siphoning the solution.

Use the Hoffman clamp (5.9) to adjust the flow of the solution so that about 8-10 drops of the solution drip through per minute.

Collect a total of 90 ml in 120 ± 10 min.

The first 30 ml of the filtrate should be collected in a 50 ml glass measuring cylinder (5.13) and disposed of.

The next 60 ml should be collected in a perfectly dry 100 ml glass measuring cylinder (5.13) to determine the blank value.

Distilled water should be at a temperature no less than 18 °C and no more than 25 °C.

Use the pipette to transfer 50 ml of the filtered solution into a previously calibrated silver dish.

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

Put it in the stove at $(105 \pm 2)^\circ\text{C}$ to attain constant weight (about $18\text{h} \pm 2\text{h}$).

To determine the blank value, the procedure needs to be repeated at least 5 times for each batch purchased.

Calculate the average of the values obtained. The result is the "blank value".