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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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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).

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.

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

1 Scope

This International Standard specifies a test method for the determination of tanning agents through filtration of 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 3696, *Water for analytical laboratory use — Specification and test methods*

3 Principle

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

4 Reagents

4.1 Distilled water, freshly prepared according to ISO 3696 (Water for analytical laboratory use, Grade 3).

The pH value of the water shall be between 5 and 6. When using methyl red, the water should not turn red. The evaporation residue of 100 ml should be less than 1 mg.

4.2 Hide powder[1)], containing not more than 0,5 % chromium oxide and with a humidity not more than 13 %.

The blank value of the hide powder shall be calculated according to Annex B.

4.3 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 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.

5.1 Desiccator, with an airtight cover and containing silica orange gel.

1) See Annex C.

5.2 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.

5.3 Water bath.

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

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

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

5.7 Procter bell (see Figure 1), composed of a cylindrical glass bell (length of the cylindrical part: 90 mm ± 1 mm; internal diameter of the cylindrical part: 28 mm ± 1 mm). A perforated rubber cork is inserted into the narrow part of the bell. A capillary glass tube (internal diameter 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

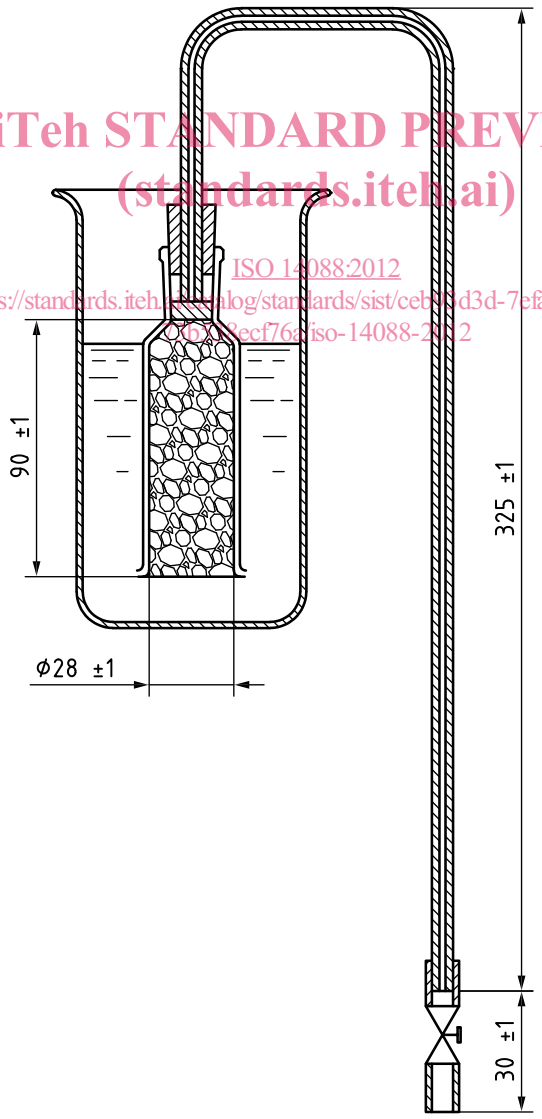

Figure 1 — Procter bell

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 1 000 ml volumetric flasks.

5.11 50 ml pipette.

5.12 Vacuum filter system (e.g. Figure 2).

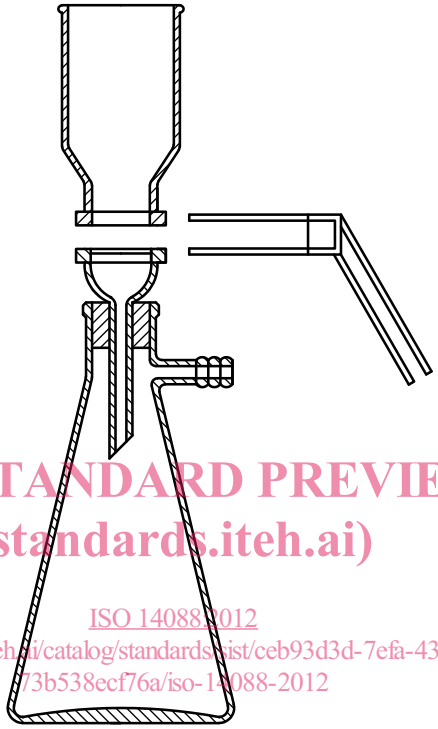

Figure 2 — Vacuum filter system

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

5.14 50 ml and 100 ml measuring cylinders.

6 Sampling and sample preparation

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

If the particles are heterogeneous, resort to manual or mechanical milling to homogenize the size of the particles. The particle size should not be smaller than 300 µm to avoid blocking the Procter bell.

7 Instrumental analysis

7.1 Preparation of the analytical solution

7.1.1 Vegetable tanning agents in powder/solid form

Weigh the appropriate quantity (see Table A.1) of vegetable tanning agents on an analytical balance (5.5). Add this to 800 ml of hot (60 °C to 80 °C) distilled water in a 1 000 ml volumetric flask (5.10). Shake the flask to fully dissolve the tanning agents. There may be some residue if there is any insoluble matter in the sample. Leave it to cool down in a water bath at (20 ± 2) °C and add distilled water (4.1) up to the mark.

The aim is to obtain an analytical solution containing between 3,75 g and 4,25 g of substances absorbed by the hide powder. 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 an analytical balance (5.5), taking into account the percentage of content in dry form. Add this to in a 1 000 ml volumetric flask (5.10) containing 800 ml of hot (60 °C to 80 °C) distilled water. Shake the flask to fully dissolve the tanning agents. There may be some residue if there is any insoluble matter in the sample. Leave it to cool down in a water bath at (20 ± 2) °C and add distilled water (4.1) up to the mark.

The aim is to obtain an analytical solution containing between 3,75 g and 4,25 g of substances absorbed in the hide powder. 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 an analytical balance (5.5). Add this to a 1 000 ml volumetric flask (5.10) containing 800 ml of hot (40 °C to 50 °C) distilled water. Shake the flask to fully dissolve the tanning agents. There may be some residue if there is any insoluble matter in the sample. Leave it to cool down in a water bath at (20 ± 2) °C and add distilled water (4.1) up to the mark.

In the case of breakthrough of the tanning agent (see 7.3), repeat the analysis using a lower mass. Record this deviation in the test report.

7.1.4 Synthetic tanning agents in liquid form

Weigh about (8 ± 0,1) g of tanning agents on an analytical balance (5.5). Add it to a 1 000 ml volumetric flask (5.10) containing 800 ml of hot (40 °C to 50 °C) distilled water. Shake the flask to fully dissolve the tanning agents. There may be some residue if there is any insoluble matter in the sample. Leave it to cool down in a water bath at (20 ± 2) °C and add distilled water (4.1) up to the mark.

In the case of breakthrough of the tanning agent (see 7.3), repeat the analysis using a lower mass. Record this deviation in the test report.

7.1.5 Vegetable tanning agents organic solvent extracted in powder form

Weigh about (4 ± 0,1) g of tanning agents on an analytical balance (5.5). Add this to a 1 000 ml volumetric flask (5.10) containing 800 ml of hot (40 °C to 50 °C) distilled water. Shake the flask to fully dissolve the tanning agents. There may be some residue if there is any insoluble matter in the sample. Leave it to cool down in a water bath at (20 ± 2) °C and add distilled water (4.1) up to the mark.

Select the amount in function of the quality of the extract desired. The final concentration of the analytical solution should contain about 4 g of tanning compound per litre.

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.

Weigh 7,0 g of hide powder (4.2) on a technical balance (5.6) and introduce it 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 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 to 10 drops of the de-tanned solution drip through per minute. The resulting solution shall be clear.

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 (5.14) and disposed of.

The next 60 ml should be collected in a perfectly dry 100 ml glass measuring cylinder (5.14) to determine the non-tanning agents. To control the breakthrough of tanning agents use 5 ml of the collected solution and add 0,5 ml gelatine solution (4.3). 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 °C and no more than 20 °C.

Use the pipette (5.11) to transfer 50 ml of the filtered solution into a previously dried and weighed silver dish (5.2).

Place the dish on the water bath (5.3) and wait for complete evaporation.

Put the dish (5.2) in the oven (5.4) at (102 ± 2) °C to attain constant mass (about 18 h ± 2 h).

Put the dish (5.2) in the silica gel dryer (5.1) and weigh it after 15 min on analytical balance (5.5).

7.4 Determination of soluble substances

To filter the analytical solutions, use the filter system (5.12) indicated in Figure 2.

Use the cellulose acetate membranes with 0,45 µm pores (5.13).

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

If the filtration is not possible, the solution should be centrifuged.

Collect about 100 ml of filtrate.

Use the pipette (5.11) to transfer 50 ml of the filtered solution into a previously dried and weighed silver dish (5.2).