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Leather — Chemical tests — Determination of tan content in synthetic tanning agents

Cuir — Essais chimiques — Détermination de la teneur en tanin dans les agents de tannage synthétiques

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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).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

ISO 17489 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 289, *Leather*, in collaboration with the Chemical Tests 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).

IULTCS, originally formed in 1897, is a worldwide 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.

Introduction

The ISO Standard, ISO 14088, is the traditional method for analysing the tanning component in a tanning agent. It uses chromium (III) tanned hide powder and determines the proportion of tanning agent that is adsorbed onto the hide powder. The non-adsorbable fraction, consisting largely of inorganic salts, remains in the tanning solution. By determining the dry content of the initial tanning solution and the non-adsorbable fraction, one calculates the adsorbable fraction – this is the tanning component in the tanning agent.

In this manner, the traditional hide powder method is used to determine the tanning strength of tanning agents. However, for reproducible test results this method requires considerable expertise from the operator in the preparation and packing of hide powder filter cartridges. In addition, the time for the filtration can be very long and the method is not suitable when results are needed in a short period of time, such as during the production quality control when manufacturing synthetic tanning agents.

The co-polymers of vinylimidazole and vinylpyrrolidone are used for removing polyphenolic compounds and metals from wine. This polymer-based powder offers a simple and practical alternative to hide powder for the routine testing of synthetic tanning agents, such as in a manufacturing facility. Interlaboratory trials (given in Table A.1) show the reproducibility and precision of repeat testing is better for the polymer-based powder.

The two procedures, ISO 14088 and ISO 17489, use different adsorbing substrates; consequently, the value for the adsorbable fraction will be different and the results obtained can only be compared when made with the same adsorbing substrate.

Leather — Chemical tests — Determination of tan content in synthetic tanning agents

1 Scope

This International Standard specifies a simple and practical method of determining the adsorbable fraction of synthetic tanning agents using a polymer-based product. It is particularly suitable for measuring the batch-to-batch consistency of synthetic tanning agents.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
tan content
adsorbable fraction of a synthetic tanning agent when mixed in a water solution with a crosslinked, insoluble vinylimidazole/ vinylpyrrolidone copolymer product

4 Principle

An acidified synthetic tanning agent solution and an insoluble copolymer product are mixed at room temperature. The insoluble copolymer absorbs polyphenols from the tanning agent. The dry content of the solution before and after mixing with the absorbing copolymer are measured. The difference is the adsorbable fraction, called the tan content.

5 Reagents

5.1 Crosslinked, insoluble vinylimidazole/vinylpyrrolidone copolymer (see Annex B).

5.2 Formic acid solution, a mass fraction of 50 %.

5.3 Gelatine, pure, AR grade.

5.4 Sodium chloride, AR grade.

5.5 Deionised or distilled water, in compliance with grade 3 in ISO 3696:1987.

6 Apparatus

Normal laboratory equipment and the following items:

6.1 Analytical balance, accurate to ± 0,1 mg.

6.2 Drying oven, ventilated, capable of maintaining a temperature of 105 °C ± 2 °C.

6.3 Magnetic stirrer.

6.4 pH meter with suitable combination electrode.

6.5 Stainless steel, (e.g. AISI 316), **or aluminium dishes** for evaporating aqueous solutions.

6.6 Dessicator, with drying agent.

6.7 Membrane filter, 50 mm diameter, 0,45 µm pore size or a suitable analytical grade filter paper.

6.8 Glass beakers, 1 000 ml and 600 ml.

6.9 Volumetric flasks, 500 ml.

6.10 Pipette, 50 ml, analytical grade.

7 Procedure

7.1 Preparation of the synthetic tanning agent solution for analysis

In a 1 000 ml beaker (6.8), add the following.

For powder synthetic tanning agents:

— accurately weigh 1,300 0 g to 1,700 0 g of the powder and record the mass (m);

— add approximately 400 ml of warm deionised water (5.5).

For liquid synthetic tanning agents:

— accurately weigh 2,700 0 g to 3,300 0 g of the liquid and record the mass (m);

— add approximately 400 ml of deionised water (5.5) at room temperature.

Stir so that the synthetic tanning agent is dissolved, cool the solution to 23 °C ± 1 °C. While stirring, bring the pH to 2,0 to 2,3 by adding dropwise a 50 % formic acid solution (5.2). Transfer the solution quantitatively to a 500 ml volumetric flask (6.9) and fill to the mark with deionised or distilled water (5.5). This is the tanning solution to be used for the following test steps.

Prepare duplicate tanning solutions for each synthetic tanning agent to be tested.

7.2 Determination of the total residual dry content (duplicate determination)

Weigh (T_1) the clean, dry stainless steel dish (6.5). With an analytical 50 ml pipette (6.10), add 50 ml of the tanning solution into the dish and evaporate most of the water carefully on a suitable hotplate or water bath. Place the dish in an oven at 105 °C ± 2 °C until constant mass is reached. Allow the dish to cool for approximately 2 h in a desiccator (6.6) with a drying agent, for example, either calcium chloride ($CaCl_2$) or silica gel. Weigh the dish (P_1).

The % dry content is calculated as follows:

$$\% \text{ dry content} = \frac{(P_1 - T_1) \times 10 \times 100}{m}$$

7.3 Determination of the non-tanning agent content (i.e. non-adsorbable fraction)

7.3.1 In a 600 ml beaker, add the following:

— 40,0 g ± 0,1 g insoluble vinylimidazole/vinylpyrrolidone copolymer powder (5.1);

— 300 g ± 1 g of the tanning solution.

Stir the suspension for approximately 30 min and leave it to settle for approximately 90 min. If the upper aqueous phase is not clear, it can be centrifuged for 15 min at approximately 3 000 rpm. Decant the upper aqueous phase through a 0,45 µm membrane filter (6.7) or a suitable analytical grade filter paper if no membrane filter is available. Keep the filtrate, to make a double determination approximately 130 ml of filtrate is needed.

7.3.2 To check the absence of tanning agent in the filtrate (7.3.1), prepare a gelatine solution by dissolving approximately 10 g gelatine (5.3) and approximately 100 g NaCl (5.4) in 1 000 ml deionised or distilled water (5.5). To an approximately 5 ml sample of the filtrate (7.3.1), add approximately 2 ml of the gelatine solution. The test solution must remain clear. If precipitation or cloudiness occurs, then there is still residual tanning agent in the filtrate. Repeat the procedure in 7.3.1 using a larger amount of the insoluble vinylimidazole/vinylpyrrolidone copolymer powder (5.1) until the test solution is clear.

7.3.3 Carry out this section in duplicate. Weigh the clean, dry stainless steel dish (T_2). With an analytical 50 ml pipette (6.10), add 50 ml of the tanning solution into the dish and evaporate most of the water carefully on a suitable hotplate or water bath. Place the dish in an oven at 105 °C ± 2 °C until constant mass is reached. Allow the dish to cool for approximately 2 h in a desiccator (6.6) with a drying agent, for example either calcium chloride ($CaCl_2$) or silica gel. Weigh the dish (P_2).

The non-tanning agent content is calculated as follows:

$$\% \text{ Non-tanning agent content} = \frac{(P_2 - T_2) \times 10 \times 100}{m}$$

8 Calculation and expression of results

8.1 Calculate the tan content (i.e. the adsorbable fraction) as follows:

% tan content = (% dry content) – (% non-tanning agent content)

8.2 Make two determinations per synthetic tanning agent sample. For the final result, calculate the mean value from the two single results. If the difference in the two results for % tan content exceeds 0,5 %, repeat the determination.

9 Test report

The test report shall include the following:

a) reference to this International Standard (i.e. ISO 17489);

b) a description of the synthetic tanning agent sample tested;

c) the % tan content result obtained (average of two determinations, to one decimal place in %);

d) any deviations from the procedure specified in this International Standard.