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Leather — Chemical determination of formaldehyde content —

Part 1:
Method using high-performance liquid chromatography

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Contents

Page

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO/FDIS 17226-1
https://standards.iteh.ai/catalog/standards/sist/34fa525c-4f6d-46cc-8a64-c720410f69a1/iso-fdis-17226-1

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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).

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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.

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ISO/FDIS 17226-1
https://standards.iteh.ai/catalog/standards/sist/34fa525e-4f6d-46cc-8a64-c720410f69a1/iso-fdis-17226-1

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). This document is technically similar to the Colorimetric Section of the method IUC 19 which was declared an official method at the IULTCS Delegates meeting on 31st May 2003 in Cancún, Mexico.

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 third edition cancels and replaces the second edition (ISO 17226-1:2018), which has been technically revised.

The main changes to the previous edition are as follows:

— The listing of reagents in Clause 6 has been reorganised.

— The composition of the dinitrophenylhydrazine (DNPH) solution (6.10) has changed. It no longer contains concentrated *o*-phosphoric acid. Under acid conditions some extracted synthetic tanning agents and resins can continue to release formaldehyde over time, giving incorrect high results.

— With the change in composition of the DNPH solution (6.10), the reaction time limits in the previous edition are no longer necessary. In 9.2.2 the reaction time and temperature have been increased to 180 min and 50 °C, respectively. Consequently, the text in 9.2.2, 9.2.3.1 and 9.2.3.2 has been modified.

— A new Clause 10 has been added.

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— In Annex A, results of a new collaborative interlaboratory trial are presented.

— Annex B has been technically revised.

A list of all parts in the ISO 17226 series can be found on the ISO website.

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.

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(standards.iteh.ai)
ISO/FDIS 17226-1
https://standards.iteh.ai/catalog/standards/sist/34fa525c-4f6d-46cc-8a64-c720410f69a1/iso-fdis-17226-1

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ISO/FDIS 17226-1
https://standards.iteh.ai/catalog/standards/sist/34fa525c-4f6d-46cc-8a64-c720410f69a1/iso-fdis-17226-1

Leather — Chemical determination of formaldehyde content —

Part 1:
Method using high-performance liquid chromatography

1 Scope

This document specifies a method for the determination of free and released formaldehyde in leathers. This method, based on high-performance liquid chromatography (HPLC), is selective and not sensitive to coloured extracts and is intended to be used for precise quantification of formaldehyde.

The formaldehyde content is taken to be the quantity of free formaldehyde and formaldehyde extracted through hydrolysis contained in a water extract from the leather under standard conditions of use.

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.

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ISO 2418, *Leather — Chemical, physical and mechanical and fastness tests — Sampling location*

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

ISO/FDIS 17226-1
https://standards.iteh.ai/catalog/standards/sist/34fa523c-4f6d-46cc-8a64-c720410f69a1/iso-fdis-17226-1

ISO 4044, *Leather — Chemical tests — Preparation of chemical test samples*

ISO 4684, *Leather — Chemical tests — Determination of volatile matter*

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 Conformity

When compared with ISO 17226-2, the two analytical methods should give similar trends but not necessarily the same absolute result. Therefore, in cases of dispute, the method in this document shall be used in preference to ISO 17226-2.

5 Principle

The process is selective. Formaldehyde is separated and quantified as a derivative from other aldehydes and ketones by HPLC. Detected is the free formaldehyde and formaldehyde which is hydrolysed during extraction to yield free formaldehyde.

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The sample is eluted with a detergent solution at 40 °C. The eluate is mixed with 2,4-dinitrophenylhydrazine (DNPH), whereby aldehydes and ketones react to give the respective hydrazones. These are separated by means of a reversed-phase HPLC with ultraviolet (UV) detector or diode array detector (DAD).

6 Reagents

Use only reagents of recognized analytical grade, unless otherwise stated. All solutions are aqueous solutions.

6.1 Water, grade 3, in accordance with ISO 3696.

6.2 Formaldehyde solution, CAS no. 50-00-0, approximately 37 % (mass fraction).

Certified solutions of formaldehyde or formaldehyde-2,4-DNPH are commercially available. When these solutions are used, the procedure in 9.1 is not required.

6.3 Iodine solution, CAS no. 7553-56-2, 0,05 mol/l, i.e. 12,68 g iodine per litre.

6.4 Sodium hydroxide solution, CAS no. 1310-73-2, 2,0 mol/l.

6.5 Sulfuric acid solution, CAS no. 7664-93-9, 2,0 mol/l.

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6.6 Sodium thiosulfate solution, CAS no. 10102-17-7, 0,1 mol/l.

6.7 Starch solution, CAS no. 9005-84-9, 1 %, i.e. 1 g in 100 ml water.

ISO/FDIS 17226-1
https://standards.iteh.ai/catalog/standards/sist/34fa525c-4f6d-46cc-8a64-c720410f69a1/iso-fdis-17226-1

6.8 Acetonitrile, CAS no. 75-05-8, LC grade.

6.9 Sodium dodecylsulfonate, CAS no. 2386-53-0, or **sodium dodecylsulfate,** CAS no. 151-21-3 (detergent solution), 0,1 %, 1 g in 1 000 ml water.

6.10 Dinitrophenylhydrazine (DNPH) solution, CAS no. 119-26-6, consisting of 0,3 g DNPH (2,4-dinitrophenylhydrazine) dissolved in 100 ml acetonitrile (6.8).

7 Apparatus

Use the usual laboratory equipment and, in particular, the following:

7.1 Volumetric flasks, of capacities 10 ml, 500 ml and 1 000 ml.

7.2 Conical flasks with stopper or screw cap, of capacities 100 ml and 250 ml.

7.3 Strainer with glass fibre filter, GF8 (or glass filter strainer G 3, diameter 70 mm to 100 mm).

7.4 Water bath, thermostatically controlled to (40 ± 2) °C, fitted with a flask shaker, frequency (50 ± 10) r/min.

7.5 Water bath or oven, thermostatically controlled to (50 ± 2) °C.

7.6 HPLC system with UV or DAD detection.

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7.7 Membrane filter, for example polyamide, 0,45 µm.

7.8 Analytical balance, weighing to an accuracy of 0,1 mg.

8 Sampling

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), provide details about sampling together with the test report. Glue residuals shall be mechanically removed from leather samples.

Cut the leather sample into pieces in accordance with ISO 4044.

If the result is to be presented on the basis of dry substance, then test a further sample of the same leather in accordance with ISO 4684 so that the moisture content can be calculated.

9 Sample preparation and analysis

9.1 Procedure for the determination of formaldehyde in the stock solution

9.1.1 Preparation of the formaldehyde stock solution

Pipette 5 ml of the formaldehyde solution (6.2) into a 1 000 ml volumetric flask (7.1) containing approximately 100 ml water (6.1) and then fill the flask with water (6.1) up to the mark. This solution is the formaldehyde stock solution.

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9.1.2 Determination

ISO/FDIS 17226-1
https://standards.iteh.ai/catalog/standards/sist/34fa525c-4f6d-46cc-8a64-c720410f65a1/iso-fdis-17226-1

Pipette 10 ml from this solution into a 250 ml conical flask (7.2) and mix with 50 ml iodine solution (6.3). Add sodium hydroxide (6.4) until it turns yellow. Allow it to react for (15 ± 1) min at 18 °C to 26 °C and then add 15 ml of sulfuric acid (6.5) while swirling.

After adding 2 ml of starch solution (6.7), titrate the excess iodine with sodium thiosulfate (6.6) until the colour changes. Make three individual determinations.

Titrate at least two blank solutions in the same manner.

The concentration of formaldehyde stock solution is calculated according to Formula (1).

$$\rho_{FA} = \frac{(V_0 - V_1) \times c_1 \times M_{FA}}{2} \quad (1)$$

where

ρ_{FA} is the concentration of the formaldehyde stock solution, in milligrams per 10 ml (mg/10 ml);

V_0 is the titre of the thiosulfate solution for the blank solution, in millilitres (ml);

V_1 is the titre of the thiosulfate solution for the sample solution, in millilitres (ml);

M_{FA} is the relative molecular mass of formaldehyde, 30,02 g/mol;

c_1 is the concentration of the thiosulfate solution, in moles per litre (mol/l).

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