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Leather — Chemical tests — Determination of free formaldehyde in process auxiliaries

Cuir — Essais chimiques — Dosage du formaldéhyde libre dans les auxiliaires de traitement

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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 27587:2009), which has been technically revised. The main changes to the previous edition are as follows:

— the wording in 5.6, 5.7, 5.12, 7.2, 7.3, 7.4 and Clause 8 has been modified;

— a new Figure 1 has been inserted and the previous Figure 1 changed to Figure 2;

— the recommended HPLC conditions previously in Clause 8 are now given in a new Annex B.

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 — Determination of free formaldehyde in process auxiliaries

1 Scope

This document specifies a method for the determination of free formaldehyde, which is released under dynamic conditions when the sample is heated in an inert dry atmosphere, in process auxiliaries for leather. The analytical result obtained according to this procedure is expressed in milligrams per kilogram (mg/kg) sample.

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

The sample is heated in an inert atmosphere for a defined period of time. The released formaldehyde is captured and derivatized using a dinitrophenylhydrazine (DNPH) cartridge. The analyte is eluted with acetonitrile and analysed by high-performance liquid chromatography (HPLC) using an ultraviolet (UV) or diode array detector (DAD).

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise stated.

5.1 Sulfuric acid, 3 mol/l.

5.2 Sodium hydroxide, 2 mol/l.

5.3 Sodium thiosulfate, 0,1 mol/l.

5.4 Iodine solution, 0,05 mol/l, i.e. 12,68 g iodine per litre of water.

5.5 Starch solution, 1 g/100 ml water.

5.6 Formaldehyde-2,4-DNPH analytical standard.

5.7 Calibration standards. Prepare solutions by adequate dilution of the formaldehyde-2,4-DNPH analytical standard (5.6).

5.8 Formaldehyde solution, a mass fraction of approximately 37 %.

5.9 Distilled water, quality 2 in accordance with ISO 3696.

5.10 Formaldehyde solution S1, prepare by pipetting 5 ml of formaldehyde solution (5.8) in a 1 000 ml volumetric flask and filling to the mark with distilled water.

5.11 Formaldehyde solution S2 in an adequate dilution for procedure control [e.g. 2,5 ml of the formaldehyde solution S1 (5.10) in a 100 ml volumetric flask, fill up with distilled water. The concentration of this solution should be adapted to ensure that the formaldehyde content is in the middle of the calibrated range.]

5.12 2,4-Dinitrophenylhydrazine cartridges (DNPH cartridges), suitable for fixing a total of 6 400 µg of carbonyls per cartridge.

For lower quantities of free formaldehyde, a smaller capacity cartridge can be used.

For higher quantities of free formaldehyde, increase the number of cartridges to be loaded in series.

It is recommended that the cartridge has an excess capacity of at least 20 % more than the predicted formaldehyde amount.

5.13 Acetonitrile, HPLC grade.

5.14 Water, HPLC grade.

6 Apparatus and materials

The usual laboratory apparatus is required and, in particular, the following.

6.1 Thermostatic oil bath with stirring facilities, capable of maintaining a constant temperature of (90 ± 3) °C. The oil bath shall be deep enough to allow the U-tube to be dipped in up to the side tubing.

6.2 Silicone oil, suitable for oil bath (6.1).

6.3 Nitrogen, purity 4,6.