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Third edition
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Leather — Chemical tests — Determination of pH and difference figure

Cuir — Essais chimiques — Détermination du pH et de l'indice de différence

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Foreword

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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 on 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 the following URL: www.iso.org/iso/foreword.html

This document was prepared by the Chemical Tests 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).

It is based on IUC 11 published in *J. Soc. Leather Tech. Chem.*, **49**, pp. 25–29, 1965, and declared an official method of the IULTCS in 1965.

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 4045:2008), which has been technically revised as follows:

— change in title to include “and difference figure”;

— 5.1 and 6.1 have been revised;

— Clause 7 has been revised: two samples are only prepared when there is enough leather;

— 8.1 has been revised to more clearly explain the procedure;

— 8.2 has been added (previously part of the next clause);

— 8.3 and 8.4 (previously 8.2 and 8.3) have been revised and the previous title of 8.4 deleted;

— Clause 9 b) and d) have been revised.

Leather — Chemical tests — Determination of pH and difference figure

1 Scope

This document specifies a method for determining the pH value and the difference figure of an aqueous leather extract. It is applicable to all types of leather.

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

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

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

3 Terms and definitions

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

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/

3.1
difference figure
difference between the pH value of a solution and that of its tenfold dilution

Note 1 to entry: The difference figure is a measure of the strength of acids and bases and can never exceed a value of 1. The difference figure amounts to 0,70 to 1,00 when a solution contains a free strong acid (or a free strong base). The ionization of weak acids and bases increases with greater dilution, and therefore the difference figure can only act as a criterion for the presence of free strong acid or base in aqueous extracts with pH values below 4 or above 10.

4 Principle

Preparation of an aqueous extract from a test portion of the leather and measurement of the pH of the extract, using a pH meter. In cases where the pH value obtained is below 4,00 or above 10,00, the pH value of a tenfold dilution of the aqueous extract is also determined.

5 Reagents

5.1 **Water**, minimum grade 3 in accordance with ISO 3696.

5.2 Buffer solution, for calibrating the electrode system.

It is preferable to purchase a commercially available standard buffer solution for measurement as recommended by the pH meter manufacturer. The length of time for which buffer solutions will keep depends on their composition and the method of use. Control of the accuracy of the buffer solution is therefore indispensable. Used buffer solution shall be discarded.

6 Apparatus

6.1 Suitable shaker, rotating shaker with a frequency of (50 ± 10) r/min or other suitable shaker after validation.

6.2 pH meter, with glass electrode, with a measuring range of 0 pH units to 14 pH units, graduated in 0,01 pH units. The electrode system shall be calibrated prior to each series of measurements against the buffer solution (5.2).

Aqueous extracts of heavily fat-liquored leather may in time make the electrode membrane dirty. In such cases, the membrane shall be lightly rubbed with a piece of cotton wool dipped in acetone or the electrode should be suspended in a 1:1 water:acetone mixture. After cleaning, the membrane should again be thoroughly soaked in water.

6.3 Analytical balance, capable of weighing to an accuracy of 0,01 g.

6.4 Wide mouthed flask, with leak-proof stopper, capacity 250 ml.

6.5 Measuring cylinder, capacity 100 ml, graduated in 1 ml divisions.

6.6 Volumetric flask, capacity 100 ml.

6.7 Pipette, capacity 10 ml.

7 Sampling and preparation of the samples

Sample in accordance with ISO 2418. If sampling in accordance with ISO 2418 is not possible, then details about sampling shall be given in the test report. Prepare the leather sample in accordance with ISO 4044.

Samples should be analysed in duplicate, if it is possible.

8 Procedure

8.1 Preparation of the extract

Weigh (5,0 ± 0,1) g of the test sample into the wide-mouthed flask (6.4) and using the measuring cylinder (6.5) add (100 ± 1) ml of water (5.1) at (20 ± 2) °C. Shake well by hand for about 30 s so that the test portion is uniformly wet. Shake mechanically in the shaker (6.1) for between 6,0 h and 6,5 h. Allow the extract to settle. Decant and take the liquid phase for pH measurement, ensuring that there are no suspended solids.

If difficulty is experienced in decanting the extract from the slurry, it may be strained through a clean, dry, non-absorbent mesh (for example, nylon cloth or a coarse sintered glass filter) or centrifuged.

8.2 Calibration of the pH meter

Calibrate the pH meter with a minimum of two buffer solutions: at least one below the expected value and at least one above the expected value. Both these buffer readings shall be within 0,02 pH units of the correct reading when the pH meter is standardized.

8.3 Determination of the pH value

Ensure that the extract (8.1) is at (20 ± 2) °C. Immediately after stirring, immerse the electrode in the extract solution and determine the pH value with the pH meter (6.2), to the nearest 0,05 pH unit, as soon as a steady reading has been reached. The reading should be taken within 30 s to 60 s.

The result of the pH value is either

— the mean value of the two individual determinations, or

— the value for a single sample.

The figures shall be given to the nearest 0,05 pH unit.

8.4 Determination of the difference figure

If the pH value is below 4,00 or over 10,00, the difference figure shall be determined, except if precluded by the client or specification. For this determination, using the pipette (6.7), transfer 10 ml of the extract solution into the volumetric flask (6.6) and fill up to the mark with water (5.1). Rinse the electrodes with approximately 20 ml of the diluted solution and then measure the pH value, as in 8.3.

The result of the difference figure value is either

— the mean value of the two individual determinations, or

— the value for a single sample.

The figures shall be given to the nearest 0,05 pH unit.

The difference figure is calculated by subtracting the pH value obtained in 8.4 from that obtained in 8.3. The difference figure result is quoted to the nearest 0,05 pH unit.

9 Test report

The test report shall include the following:

a) a reference to this document (i.e. ISO 4045);

b) a description of the sample;

c) a reference to any instability of the pH reading of the extract which prevents an unequivocal statement of the pH value or difference figure;

d) pH and, if requested, difference figures as measured in 8.3 and 8.4;

e) details of any deviations from the prescribed test conditions.

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