INTERNATIONAL STANDARD

ISO 10195

IULTCS/IUC 41

First edition
2018-05

Leather — Chemical determination of chromium(VI) content in leather — Thermal pre-ageing of leather and determination of hexavalent chromium

Cuir — Détermination chimique de la teneur en chrome(VI) du cuir — Vieillissement thermique du cuir et détermination de la teneur en chrome hexavalent

Reference numbers
ISO 10195:2018(E)
IULTCS/IUC 41:2018(E)

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Published in Switzerland

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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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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 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, and the European Committee for Standardization (CEN) Technical Committee CEN/TC 309, *Footwear*, the secretariat of which is held by AENOR, 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.

Introduction

More than 80 % of leather is tanned using chromium(III) salts. The industry recommends manufacturing procedures to avoid oxidative conditions that could allow the formation of traces of hexavalent chromium [chromium(VI)] in the leather.

It is difficult to reproduce the natural ageing process. Therefore, in order to predict the tendency for trace levels of hexavalent chromium to develop in chromium(III) tanned leather, a number of pre-ageing tests have been proposed, some of which are being used in commercial leather specifications.

Following an inter-laboratory trial (see Annex A), thermal pre-ageing was selected as a suitable pre-ageing test procedure. The method is presented in this document.

Information on the development of hexavalent chromium during the natural ageing of chromium(III) tanned leather is given in Annex B.

Leather — Chemical determination of chromium(VI) content in leather — Thermal pre-ageing of leather and determination of hexavalent chromium

1 Scope

This document specifies a thermal pre-ageing procedure for leather to obtain indications about the tendency to the formation of hexavalent chromium under specified conditions and the determination of hexavalent chromium according to ISO 17075-1 or ISO 17075-2.

This thermal pre-ageing procedure does not simulate any real condition in leather production or use.

It is applicable to all types of chromium tanned 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 4044:2017, *Leather — Chemical tests — Preparation of chemical test samples*

ISO 17075-1:2017, *Leather — Chemical determination of chromium(VI) content in leather — Part 1: Colorimetric method*

ISO 17075-2:2017, *Leather — Chemical determination of chromium(VI) content in leather — Part 2: Chromatographic method*

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

A test specimen of leather is heated in an oven for a specified period of time. The sample is then cooled and the chemical determination of hexavalent chromium [chromium(VI)] is carried out according to ISO 17075-1 or ISO 17075-2.

5 Apparatus

5.1 Static oven, capable of maintaining the required temperature within a tolerance of ±2 °C. The oven shall not have a fan or any other air circulating system, nor openings that allow active air exchange. The oven should have an interior made of inert materials. An oven with natural convection should be used.

5.2 250 ml Erlenmeyer glass flask with stopper, for containing the specimen, able to be maintained at 80 °C for 24 h and cooled at ambient temperature. This vessel allows the procedure of ISO 17075-1 or ISO 17075-2 to start from 7.2 (see 6.9).

5.3 Apparatus for temperature and humidity measurement.

5.4 Analytical balance, with an accuracy of 0,1 mg.

6 Procedure

6.1 Pre-heat the oven (5.1) to the desired temperature, see Table 1.

Table 1 — Definition of the pre-ageing temperature

Method	Conditions	Recommended use
A1	At (60 ± 2) °C and relative humidity less than 20 %	General purpose ageing for leathers not subjected to critical conditions in manufacturing (gloves, garments, leather goods, handmade and fashion articles) and/or transportation
A2	At (80 ± 2) °C and relative humidity less than 10 %	Ageing for leathers subjected to critical conditions in manufacturing (footwear, automotive, upholstery) and/or transportation

6.2 Cut a leather sample in accordance with ISO 2418. If sampling in accordance with ISO 2418 is not possible (e.g. leathers from articles like shoes, garments), details about sampling shall be given in the test report.

6.3 Prepare the leather test specimen according to ISO 4044:2017, 6.3. Cut it into pieces of 3 mm to 5 mm with a sharp blade.

6.4 Weigh accurately (2,0 ± 0,1) g of leather test specimen, to 0,1 mg accuracy, into the vessel (5.2). Record the mass, *m*.

6.5 The vessel shall be closed immediately after weighing.

6.6 Open the vessel and place it in the oven.

6.7 The oven door shall not be opened during the ageing time. The temperature and humidity shall be checked (5.3) at least three times during the ageing process.

NOTE If the oven with natural convection is kept closed:

— at a temperature of 60 °C, the relative humidity will be physically lower than 20 %;

— at a temperature of 80 °C, the relative humidity will be physically lower than 10 %.

6.8 After (24 ± 1) h, remove the Erlenmeyer flask (5.2) from the oven, close it immediately and allow the leather test specimen to cool at ambient room temperature for 2 h ± 15 min.

6.9 Immediately after cooling, determine the hexavalent chromium content in the aged leather test specimen according to either ISO 17075-1 or ISO 17075-2.

Using the Erlenmeyer flask (5.2) allows the procedure to start from ISO 17075-1:2017, 7.2 or ISO 17075-2:2017, 7.2. Add by pipetting 100 ml of degassed solution (ISO 17075-1:2017, 5.1 or ISO 17075-2:2017, 5.1) into the flask and then displace the oxygen according to ISO 17075-1:2017, 7.2 or ISO 17075-2:2017, 7.2.

6.10 Calculate hexavalent chromium content in accordance with ISO 17075-1 or ISO 17075-2.

7 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 10195;

b) a description of the type of leather tested and details about sampling if necessary (6.2);

c) the pre-ageing method selected;

d) the amount of hexavalent chromium as defined in 6.10;

e) the test method used for hexavalent chromium determination.

Annex A
(informative)

Accuracy

Results obtained from an international inter-laboratory trial carried out in June 2016.

The purpose of this inter-laboratory trial was to compare two pre-ageing conditions (thermal and light) and their effect on the concentration of hexavalent chromium in leathers.

Seven laboratories were involved in this study. Five chromium(III) tanned leather samples were tested:

1) **Leather A**: bovine, crust leather without finishing that was prepared following recommended procedures to minimize hexavalent chromium, including the use of an antioxidant.

2) **Leather B**: bovine, crust leather without finishing that was prepared following recommended procedures to minimize hexavalent chromium, but without an antioxidant.

3) **Leather C**: sample of black leather with a finish coat.

4) **Leather D**: bovine, crust leather that was prepared using procedures not recommended for minimising hexavalent chromium, but with vegetable retanning.

5) **Leather E**: bovine, crust leather that was prepared using procedures not recommended for minimising hexavalent chromium.

Samples were sent to the laboratories carrying out the testing during the same week of 2016 (week 29).

The hexavalent chromium analyses were performed according to ISO 17075-2 (i.e. with cut leather samples and ionic chromatography determination).

The results of the thermal pre-ageing study are given in Table A.1.

Table A.1 — Results of the inter-laboratory test for thermal ageing (Method A2, Table 1) and the determination of hexavalent chromium in leather

Leathers	Before thermal ageing					After thermal ageing				
	Number of laboratories		AV	SD	UM	Number of laboratories		AV	SD	UM
	Participating	With acceptable values				Participating	With acceptable values			
A	7	6	0,24	0,13	0,07	7	6	0,51	0,22	0,11
B	7	5	0,21	0,14	0,08	7	6	0,71	0,26	0,13
C	7	7	6,62	1,17	0,84	7	7	13,08	3,60	1,70
D	7	7	0,37	0,24	0,11	7	7	1,57	0,73	0,34
E	7	6	9,88	1,16	0,59	7	7	25,95	8,09	2,82

AV Average value of hexavalent chromium in mg/kg.

SD Standard deviation in mg/kg.

UM Uncertainty of measurement in mg/kg.

In this study the light pre-ageing of the five leathers gave very similar results to those of the thermal pre-ageing, so it was decided to only keep the thermal pre-ageing as a unique test method.

The full report of this inter-laboratory study is available in the e-committee document CEN/TC 309/WG 2 N333.

Annex B
(informative)

Natural ageing and development of hexavalent chromium

The leathers A, B, D and E, as given in Annex A, were prepared during a research program and their hexavalent chromium content has been followed during more than 12 months of natural ageing. For information, Figure B.1 gives the evolution of the hexavalent chromium in these four leathers stored in ambient conditions in the CTC tannery, France.

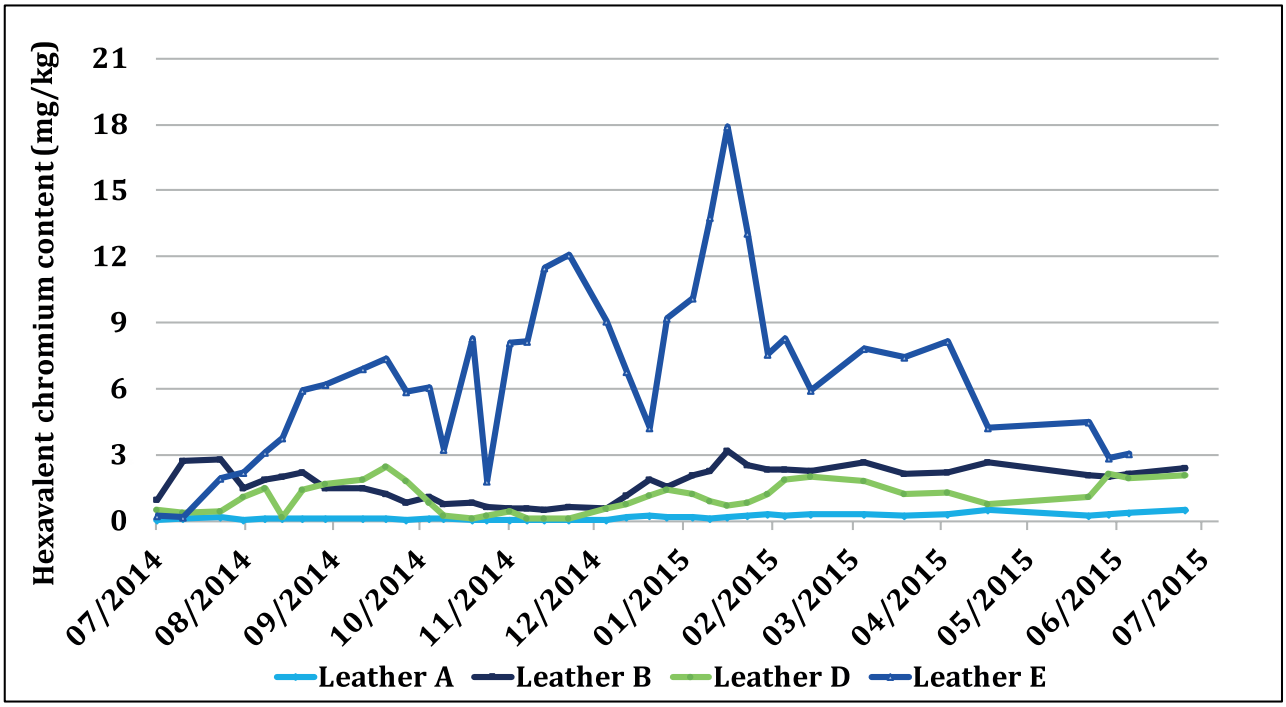

Figure B.1 — Evolution of hexavalent chromium in leathers A, B, D and E during a period of more than 12 months in ambient conditions

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