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IULTCS/IUC 8-3

Second edition
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Leather — Chemical determination of chromic oxide content —

Part 3: Quantification by atomic absorption spectrometry

Cuir — Dosage chimique de l'oxyde de chrome —

Partie 3: Quantification par spectrométrie d'absorption atomique

Reference numbers
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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 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 8, published in *J. Soc. Leather Tech. Chem.*, **49**, p. 17, 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 second edition cancels and replaces the first edition (ISO 5398-3:2007), which has been technically revised as follows:

— some editorial corrections;

— Clause 5 refers to ISO 4044 for preparing the sample rather than grinding the leather;

— the description of suitable AAS equipment, previously in 8.2.1, has been moved to a new informative Annex B.

A list of all parts in the ISO 5398 series can be found on the ISO website.

Introduction

The ISO 5398 series comprises four parts, each describing methods suitable for the determination of the chromic oxide content in leather. The different techniques have been described to reflect the variations in industrial practice compared with the more sensitive analytical equipment available for test laboratories. Variations also exist in the range of chromic oxide that the methods are deemed suitable to quantify.

This document describes a technique that is suitable for determining chromium more precisely than those described in ISO 5398-1 and ISO 5398-2. It requires the use of sophisticated analytical equipment, such as atomic absorption spectroscopy.

Leather — Chemical determination of chromic oxide content —

Part 3:
Quantification by atomic absorption spectrometry

1 Scope

This document describes a method for the determination of chromium in aqueous solution obtained from leather. This is an analysis for total chromium in leather; it is not compound specific or specific to its oxidation state.

This method describes the determination of chromium by atomic absorption spectrometry and is applicable to leathers which are expected to have chromic oxide contents in excess of 5 mg/kg. Two techniques for the preparation of the solution to be analysed are included. In the case of disputes, the wet oxidation technique is to be used.

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*

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

EN 14602, *Footwear — Test methods for the assessment of ecological criteria*

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
chromic oxide content
amount of chromium in leather, determined by this method and reported as chromic oxide

Note 1 to entry: The chromic oxide content is expressed in milligrams per kilogram (mg/kg), based on dry matter.

4 Principle

The chromium present in the leather is solubilized in the hexavalent state followed by analysis of the solution by atomic absorption spectrometry.

5 Sampling and sample preparation

Sample in accordance with ISO 2418 and prepare the leather in accordance with ISO 4044. If sampling in accordance with ISO 2418 is not possible (as in the case of leathers from finished products such as shoes or garments), details about sampling shall be given together with the test report.

Weigh 2 g of the prepared leather to the nearest 0,001 g. From every leather sample, a minimum of two determinations shall be made.

6 Reagents

Unless otherwise stated, only analytical grade chemicals are to be used. The water shall be grade 3 in accordance with ISO 3696. All solutions are aqueous solutions.

6.1 Reagents for wet oxidation method

6.1.1 Nitric acid, 70 %.

6.1.2 Sulfuric acid, concentrated (98 %), and **perchloric acid** (60 % to 70 %), mixed together in the ratio of 1:3 by volume.

6.2 Reagents for atomic absorption spectrometry

6.2.1 Potassium dichromate ($K_2Cr_2O_7$), dried for 16 h ± 2 h at 102 °C ± 2 °C.

For safety reasons, it is advisable to use already prepared certified commercial solution. (For the concentration see 6.2.3.)

6.2.2 Potassium chloride (KCl).

6.2.3 Standard dichromate solution: dissolve 2,829 g of potassium dichromate (6.2.1) in water in a volumetric flask and make up to 1 000 ml with water. 1 ml of this solution contains 1 mg of chromium.

NOTE This solution is available commercially.

6.2.4 Potassium chloride solution: dissolve 2 g of potassium chloride (6.2.2) in 1 l of distilled water. Add 1 ml of nitric acid (6.1.1) to each litre prepared.

7 Apparatus

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

7.1 Conical flask, 500 ml, with ground glass stopper.

7.2 Atomic absorption spectrophotometer, with suitable hollow cathode lamp and nitrous oxide burner head or high solids nitrous oxide burner head.

7.3 Filtration device, using glass fibre (GFC) or membrane type filters.

7.4 Antibumping granules or similar, (wet oxidation method).

8 Methods

8.1 Preparation of analytical solution

8.1.1 Wet oxidation method

WARNING — It is imperative that nitric acid is added first because of the possible explosive reaction of perchloric acid with leather.

Accurately weigh a mass of leather (see Clause 5) into the conical flask (7.1). Add 10 ml of nitric acid (6.1.1) and allow to stand for 2 min. Add 15 ml of mixed sulfuric/perchloric acids (6.1.2) and a few antibumping granules (7.4). Place a funnel or splash bulb in the neck of the flask and heat to boiling on a wire gauze over a moderate flame. As soon as the reaction mixture begins to turn orange, lower the flame. After a complete change of colour, heat gently for at least 2 min. Allow to cool in air for 5 min and dilute to approximately 200 ml. Boil for 10 min to eliminate any chlorine.

The use of a sulfuric/perchloric acid mixture is preferred to the use of the individual acids as it prevents the accidental use of perchloric acid alone.

In the case of incomplete oxidation (i.e. the solution does not change to an orange colour), it is permissible to add further mixed sulfuric/perchloric acid to the sample.

8.1.2 Microwave digestion method

The sample for analysis can also be prepared through the application of microwave-assisted digestion (MAD). The procedure described in EN 14602 shall be followed.

8.2 Measurement of the aqueous solution

8.2.1 General

Prepare the atomic absorption spectrophotometer (7.2) by following the manufacturer's instructions for adjusting all instrument parameters. See Annex B for an example of suitable equipment.

8.2.2 Preparation of calibration graph

Prepare standard solutions by pipetting 10 ml of the standard dichromate solution (6.2.3) into a 100 ml volumetric flask and making up to volume with distilled water. Pipette 2,0 ml, 4,0 ml, 6,0 ml and 8,0 ml aliquots of this solution into 100 ml volumetric flasks and make up to volume with potassium chloride solution (6.2.4). These solutions contain 2,0 µg/ml, 4,0 µg/ml, 6,0 µg/ml and 8,0 µg/ml of chromium, respectively.

Aspirate the standard solutions and prepare a standard calibration curve. This calibration may be retained in the spectrophotometer's memory if preferred.

8.2.3 Analysis of the test solution

Transfer the contents from the analytical solution obtained from 8.1 into a 250 ml volumetric flask and make up to volume with the potassium chloride solution (6.2.4), mixing well.

This solution can be analysed directly following filtration (7.3), provided it does not contain more than 7,5 µg/ml of chromium. Otherwise, the solution should be diluted accordingly.

Aspirate the test solution and determine the absorbance obtained. Calculate the concentration of chromium in the solution using the standard calibration curve. Note that if the calibration is retained in the spectrophotometer's memory, then the reading may be given directly in terms of concentration.