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Leather — Chemical determination of chromic oxide content —

Part 1: Quantification by titration

Cuir — Dosage chimique de l'oxyde de chrome —

Partie 1: Quantification par titrage

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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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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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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.

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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 co-operation 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 5398-1:2007), 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.

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

Introduction

The ISO 5398 series consists of 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.

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This document describes a traditional technique applied in industry that does not require the use of advanced analytical equipment.

Leather — Chemical determination of chromic oxide content — Part 1:Quantification by titration

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 chrome by iodometric titration and is to be applicable to chromium-tanned leathers which are expected to have chromic oxide contents in excess of 0,3 %. Two different methods are described as alternatives for obtaining chromium in a suitable solution. It is appropriate to use either method.

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 4047, *Leather — Determination of sulphated total ash and sulphated water-insoluble ash*

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

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/

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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 per cent by mass, 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 iodometric titration.

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 like shoes or garments), details about sampling shall be given together with the test report.

Weigh accurately the prepared leather to the nearest 0,001 g. (Suggested masses are full chrome leather 1 g, semi-chrome leather 2 g, leather with low chrome content 2 g to 5 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 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.

6.1.3 Orthophosphoric acid, 90 %.

6.2 Alkaline fusion method

6.2.1 Fusion mixture, consisting of equal masses of **sodium carbonate** (Na_2CO_3), **potassium carbonate** (K_2CO_3) and **sodium tetraborate** ($Na_2B_4O_7$).

6.2.2 Hydrochloric acid, concentrated (37 %).

6.3 Iodometric titration

6.3.1 Potassium iodide solution, freshly prepared, 100 g/l.

6.3.2 Sodium thiosulfate, 0,1 mol/l standardized solution in water.

6.3.3 Starch indicator solution, 10 g/l (or soluble starch powder).

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 Crucible, glazed porcelain or platinum (required for alkaline fusion method only).

7.3 Burette, 50 ml.

7.4 Filtration device, using simple paper, glass fibre (GFC) or membrane type filters.

7.5 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 (7.5). 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. Allow to cool and add 5 ml of orthophosphoric acid (6.1.3) to mask any iron.

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 Alkaline fusion method

Ash the accurately weighed sample of leather (see Clause 5) in accordance with ISO 4047. In the crucible (7.2) containing the leather ash, carefully add 5 g of fusion mixture (6.2.1) and mix well using a platinum wire or thin glass rod. Start by heating the crucible gently on an open flame, then heat more fiercely for approximately 30 min. (A muffle furnace operating at 750 °C ± 50 °C for at least 30 min may be used to heat the melt.) After cooling, place the crucible in a beaker containing 100 ml to 150 ml of boiling water and continue to heat the water until the fusion mixture has completely dissolved. Filter (7.4) the solution obtained into the conical flask (7.1). Thoroughly wash the beaker, crucible and filter with hot water, collecting the washings in the flask. Carefully add at least 10 ml hydrochloric acid to the flask and allow to cool down to room temperature.

8.2 Measurement of the aqueous solution

Add to the solution obtained from 8.1.1 or 8.1.2 20 ml of potassium iodide solution (6.3.1), stopper the flask and leave to stand for 10 min in the dark. Titrate with 0,1 mol/l sodium thiosulfate solution (6.3.2) until the solution in the flask is either light green or blue using 5 ml of starch indicator solution (6.3.3) (or a small quantity of starch powder), added towards the end of titration. Note the millilitres of thiosulfate solution used.

If starch solution is used, it should either be freshly prepared or should have been prepared with the addition of a little mercuric iodide to preserve the solution for several months.

If the titre is in excess of 50 ml, the analysis should be repeated using either smaller sample size or with appropriate dilution of the solution obtained from 8.1.1 or 8.1.2.