INTERNATIONAL STANDARD

ISO 5398-4

IULTCS/IUC 8-4

Second edition
2018-12

Leather — Chemical determination of chromic oxide content —

Part 4: Quantification by inductively coupled plasma (ICP)

Cuir — Dosage chimique de l'oxyde de chrome —

Partie 4: Quantification par plasma à couplage inductif (ICP)

Reference numbers
ISO 5398-4:2018(E)
IULTCS/IUC 8-4:2018(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword ... **iv**
Introduction ... **vi**
1 Scope ... **1**
2 Normative references ... **1**
3 Terms and definitions ... **1**
4 Principle ... **2**
5 Sampling and sample preparation ... **2**
6 Reagents ... **2**
7 Apparatus ... **2**
8 Methods ... **2**
8.1 Preparation of analytical solution ... 2
8.1.1 Wet oxidation method ... 2
8.1.2 Microwave digestion or other method of digestion ... 3
8.2 Measurement of the aqueous solution ... 3
8.2.1 General ... 3
8.2.2 Preparation of calibration graph ... 3
8.2.3 Analysis of the test solution ... 3
9 Calculation and expression of results ... **4**
10 Test report ... **4**
Annex A (informative) **Determination of water and other volatile matter** ... **5**
Annex B (informative) **Suitable ICP equipment** ... **6**

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 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 5398-4:2007), which has been technically revised. The main changes compared to the previous edition are as follows:

— the title has changed to “inductively coupled plasma (ICP)” to include more than just the “ICP-OES” type equipment;

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

— the description of additional suitable ICP equipment (ICP-MS) has been included in a new subclause 7.3;

— the previous subclause 8.2.1 describing suitable ICP equipment has been moved to a new informative Annex B;

— the text has been revised for editorial corrections.

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

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.

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 inductively coupled plasma (ICP).

Leather — Chemical determination of chromic oxide content —

Part 4:
Quantification by inductively coupled plasma (ICP)

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 inductively coupled plasma (ICP) and is applicable to leathers which are expected to have chromic oxide contents in excess of 1 mg/kg. Two techniques for the preparation of the solution to be analysed are included. In the event of dispute, the wet oxidation technique is intended 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*

ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)*

ISO 17294-2, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of selected elements including uranium isotopes*

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 ICP.

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.

6 Reagents

Chemicals with at least analytical grade shall be used for wet oxidation method. Ultrapure acid shall be used for microwave digestion. The water shall be grade 3 in accordance with ISO 3696. All solutions are aqueous solutions.

6.1 Nitric acid, around 70 %.

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

6.3 Commercial solution of chromium, for example 1 000 mg/l.

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 Inductively coupled plasma-optical emission spectrometer (ICP-OES), as described in ISO 11885.

7.3 Inductively coupled plasma/mass spectrometer (ICP-MS), as described in ISO 17294-2.

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

7.5 Anti-bumping granules (or similar) (wet oxidation method).

7.6 Volumetric flask, capacity 50 ml and 250 ml.

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.

Weigh 2 g of the prepared leather to the nearest 0,001 g. If possible perform two determinations.

Accurately weigh a mass of leather (see Clause 5) into the conical flask (7.1). Add 10 ml of nitric acid (6.1) and allow to stand for 2 min. Add 15 ml of mixed sulfuric/perchloric acids (6.2) and a few anti-bumping 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 or on a hot plate. As soon as the reaction mixture begins to turn orange, lower the flame or the temperature. 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.

Transfer the analytical solution into a 250 ml volumetric flask and make up the volume with distilled water, mixing well.

With the wet oxidation method, the analysis should preferably be performed with ICP-OES, as there is a risk of interference with ICP-MS.

8.1.2 Microwave digestion or other method of digestion

The sample for analysis can also be prepared through application of microwave-assisted digestion (MAD) or other validated digestion methods. If this is to be used, then the procedure and the sample quantity shall be adapted. Weigh 0,1 g to 1 g of the prepared leather to the nearest 0,001 g. If possible perform two determinations.

8.2 Measurement of the aqueous solution

8.2.1 General

Prepare the ICP instrument (7.2) or (7.3) by following the manufacturer's instructions for adjusting all the instrument's parameters. See Annex B for an example of suitable equipment settings.

8.2.2 Preparation of calibration graph

Prepare standard reference solutions of chromium in accordance with ISO 11885 or ISO 17294-2 by ensuring that the acid concentration in the standard reference solutions is of the same order as that of the sample. For calibration, prepare at least four standard reference solutions plus a calibration blank.

8.2.3 Analysis of the test solution

This solution (8.1.1) can be analysed directly following filtration (7.4).

Aspirate the test solution and determine the concentration of chromium in the solution by use of the standard calibration curve.

NOTE If the calibration is retained in the spectrophotometer's memory, then the reading can be given directly in terms of concentration.

If the concentration is not in the range of the calibration standards, the analysis should be repeated either using a smaller sample size or with an appropriate dilution of the solution obtained from 8.1.1 or 8.1.2.

9 Calculation and expression of results

Calculate the chromic oxide content in the leather, w_{Cr}, expressed in milligrams per kilogram (mg/kg), using Formula (1):

$$w_{Cr} = \frac{\rho \times 1{,}462 \times V \times F}{m_0} \quad (1)$$

where

ρ is the concentration of chromium determined in 8.2.3, in micrograms per millilitre (µg/ml);

V is the total volume, in millilitres (ml) (if no additional dilution is required, V = 250 ml);

m_0 is the original mass of leather, in grams (g);

1,462 is the correction factor to convert Cr to Cr_2O_3;

F is the factor to correct to 0 % volatile matter; it is calculated as follows:

$$F = \frac{100}{100 - w_W}$$

where w_W is the volatile matter content, according to ISO 4684 (see Annex A), in percent by mass.

It is permissible, if required, to quote the results based on the dry, degreased mass of the sample.

10 Test report

The test report shall include the following:

a) a reference to this document, i.e. ISO 5398-4;

b) a description of the leather;

c) a reference to the method used for sample preparation, type of digestion and measurement;

d) the volatile matter content of the leather, in percent;

e) the results obtained, in milligrams per kilogram (mg/kg);

f) details of any deviations from the described procedures.

Annex A
(informative)

Determination of water and other volatile matter

The volatile matter content of leathers is determined according to ISO 4684. The volatile matter content of the leather is determined from a sample of ground leather prepared for the chromium determination. Wet leathers are dried before the determination of the volatile matter content according to ISO 4684. The loss in mass during initial drying is added to the loss in mass after drying according to ISO 4684.