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ISO 5398-3

IULTCS/IUC 8:3

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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

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

This part of ISO 5398 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 originally published in *J. Soc. Leather Trades Chemists* **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.

ISO 5398 consists of the following parts, under the general title *Leather — Chemical determination of chromic oxide content*:

— *Part 1: Quantification by titration*

— *Part 2: Quantification by colorimetric determination*

— *Part 3: Quantification by atomic absorption spectrometry*

— *Part 4: Quantification by inductively coupled plasma — optical emission spectrometer (ICP-OES)*

Introduction

ISO 5398 has been split into 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.

ISO 5398-3 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 part of ISO 5398 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 dispute, the wet oxidation technique is to be used.

2 Normative references

The following referenced documents are indispensable for the application 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 3696:1987, *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.

3.1
chromic oxide content
amount of chromium in leather, determined by this method and reported as chromic oxide

NOTE 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

If possible, sample in accordance with ISO 2418 and grind 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, garments), details about sampling shall be given together with the test report.

Weigh 2 g of the ground leather to the nearest 0,001 g. From every leather, 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 3696:1987. 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.

6.2 Atomic absorption spectrometry

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

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.

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

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

The sample for analysis can also be prepared through application of microwave-assisted digestion (MAD). If this is to be used, then 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.

Where it is noted that the setting is as recommended by the manufacturer, then the settings used should be those described by the manufacturer for chromium.

Lamp current	as recommended by manufacturer
Slit width/band pass	0,5 nm
Wavelength	357,9 nm
Burner head	single slot nitrous oxide or high solids nitrous oxide to give red cone 10 mm to 20 mm high
Fuel flow	as recommended by manufacturer
Oxidant flow	as recommended by manufacturer
Photomultiplier voltage	as required to give optimum signal/noise ratio

Before carrying out the spectrometric measurements, set up the spectrophotometer according to the manufacturer’s instructions by aspirating a 4,0 µg/ml calibration solution. Optimize the aspiration and flame conditions (aspiration rate, nature of the flame, positions of the optical beam in the flame).

Aspirate distilled water and adjust controls to give a steady zero (base-line) reading.

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 by use of 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.

If the absorbance is outside 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 the following equation:

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

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, based on ISO 4684, 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 part of ISO 5398 (ISO 5398-3:2007);

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 percentage;

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.

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