INTERNATIONAL STANDARD

ISO 5398-2

IULTCS/IUC 8:2

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Leather — Chemical determination of chromic oxide content —

Part 2: Quantification by colorimetric determination

Cuir — Dosage chimique de l'oxyde de chrome —

Partie 2: Quantification par dosage colorimétrique

Reference number
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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.

ISO 5398-2 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 289, *Leather*, in collaboration with the Chemical Tests Commission of the International Union of Leather Technologists and Chemists Societies (IUC Commission, IULTCS), in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement). It is based on IUC 8 originally 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.

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 5398-2 describes a colorimetric technique that requires the use of a spectrophotometer.

Leather — Chemical determination of chromic oxide content —

Part 2:
Quantification by colorimetric determination

1 Scope

This part of ISO 5398 describes the determination of chrome by colorimetric means. It is applicable to leathers which are expected to have chromic oxide contents in excess of 0,05 %.

This is an analysis for total chromium in leather; it is not compound specific or specific to its oxidation state.

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*

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 percentage by mass, based on dry matter.

4 Principle

Ash obtained from leather is fused by an alkaline mixture. The chromium present in the leather is solubilized in the hexavalent state followed by analysis of the solution after reaction with 1,5-diphenyl carbazide to diphenyl carbazone by photometric means.

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 (e.g. leathers from finished products like shoes, garments), details about sampling shall be given together with the test report.

Weigh accurately 0,75 g of the ground leather, depending on the expected content of chrome, to the nearest 0,001 g. From each leather, make a minimum of two determinations using separate leather portions.

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.

6.1 Alkaline fusion method

6.1.1 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 Photometric determination

6.2.1 Sulfuric acid, 98 %.

6.2.2 Potassium dichromate stock solution (0,2 mg Cr_2O_3/ml), prepared from 0,387 1 g potassium dichromate (6.2.7) in 1 l distilled water.

6.2.3 Potassium dichromate standard solution, prepared from 50 ml of stock solution (6.2.2) pipetted into a 250 ml volumetric flask and made to volume with distilled water. (1 ml of this solution contains 0,04 mg Cr_2O_3.)

6.2.4 Diphenyl carbazide solution, consisting of 1,00 g of 1,5-diphenyl carbazide dissolved in 100 ml acetone with one drop of glacial acetic acid added. This can be stored for up to two weeks at 4 °C.

6.2.5 Acetone.

6.2.6 Glacial acetic acid, 100 %.

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

7 Apparatus

Usual laboratory apparatus is required and, in particular, the following.

7.1 Crucible, glazed porcelain or platinum.

7.2 Spectrophotometer, capable of reading absorbance to a minimum of 3 decimal places at a wavelength of 540 nm.

7.3 Cuvettes, of 1 cm optical path length.

7.4 Muffle furnace.

7.5 Filtration device, using ash-free paper.

8 Methods

8.1 Alkaline fusion method

Pre-ash the accurately weighed sample of leather (see Clause 5) using an open flame prior to ashing and afterwards ash at 750 °C ± 50 °C for 4 h.

In the crucible (7.1) containing the leather ash, carefully add 5 g of fusion mixture (6.1.1) and mix well using a platinum wire or thin glass rod. Initially heat 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 approximately 30 min may be used for heating the melt.) After cooling, place the crucible in a beaker containing 100 ml to 150 ml of hand-warm water and continue to heat in the water until the fusion mixture has completely dissolved. No losses of the solution due to splashing shall occur.

Filter (7.5) the solution obtained into a 1 000 ml volumetric flask. Thoroughly wash the beaker, crucible and filter with hot water, collecting the washings in the volumetric flask. If a green residue is observed in the filter, this shall be ashed as described above with the filtrate being added to the 1 000 ml flask.

Carefully add at least enough sulfuric acid to the flask until pH 1 is reached, allow to cool down to room temperature and make up to volume with distilled water.

The resulting solution is stable for 4 days if stored in the dark for samples in excess of 0,5 % Cr_2O_3. For lower concentrations, analysis shall be done immediately.

8.2 Measurement of the aqueous solution

Add 10 ml, up to 50 ml from leather with an expected low chromium content, of the solution (8.1) to a 100 ml volumetric flask. Add 1 ml of diphenyl carbazide solution (6.2.4). Make up to volume with distilled water and invert several times to mix thoroughly. The colorimetric measurement should be carried out (15 ± 5) min after the addition of the diphenyl carbazide solution.

Measure the absorbance of the test solution against a blank solution prepared using 25 ml distilled water rather than the sample, 1 ml sulfuric acid and 1 ml diphenyl carbazide solution (6.2.4) at 540 nm using a 1 cm cuvette.

If the absorbance is in excess of that observed from the highest calibration standard, the analysis should be repeated using either smaller sample size, or with appropriate dilution of the solution obtained from 8.1.

8.3 Preparation of the calibration solution

From the standard solution of potassium dichromate (6.2.3), add the volumes given in the Table 1 to separate 100 ml volumetric flasks.

Table 1 — Volumes needed

Concentration mg Cr_2O_3/100 ml	$V_{standard}$ ml
0,02	0,5
0,04	1
0,08	2
0,16	4
0,24	6
0,28	7

Add 1 ml of sulfuric acid (6.2.1) to each flask followed by 1 ml of diphenyl carbazide solution (6.2.4). Make up each flask to volume with distilled water and inverted several times to mix thoroughly.

Prepare a calibration graph by plotting the absorbance against the Cr_2O_3 content in milligrams per 100 ml (mg/100 ml).

9 Check of the analytical system

Upon each set of analysis or on a daily basis (whichever is the least frequent), a solution prepared from 5 ml standard solution (6.2.3) should be evaluated according to the photometric method (8.2) to ensure the accuracy of the method.

10 Calculation and expression of results

10.1 General

Calculate the chromic oxide content, w_{Cr}, in the leather, as a percentage by mass on dry matter according to the following equation.

$$w_{Cr} = \frac{\rho_{sample}}{m_{sample}} F \frac{V_1}{V_2} D$$

where

ρ_{sample} is the concentration of Cr_2O_3 obtained from the calibration graph, in milligrams per 100 ml (mg/100 ml);

m_{sample} is the mass of the original leather sample, in grams (g), (use for the fusion; fusion mixture is dissolved in 1 000 ml);

D is the dilution factor of the samples from 8.1 (usually = 1);

V_1 is the volume of the volumetric flask (ml) used for the colorimetric determination;

V_2 is the volume, in millilitres (ml), of the solution (8.1) (10 ml to 50 ml depending on the chromium content);

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 percentage by mass.

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

10.2 Repeatability

The results of the duplicate determination should not differ by more than 0,1 % calculated on the original mass of the leather.

The results of the investigation concerning repeatability of the procedure are given in Table 2.

Table 2 — Repeatability results

Amount g	Extinction	Cr_2O_3 (dry matter) %
0,748 3	0,585 8	3,371
0,746 7	0,591 3	3,410
0,749 6	0,592 5	3,403
0,752 1	0,590 4	3,380
0,750 2	0,595 9	3,420
0,751 1	0,595 1	3,412

number of values: 6

average content [% Cr_2O_3 (dry matter)]: 3,399

standard derivation [% Cr_2O_3 (dry matter)]: 0,019

variation coefficient (%): 0,56

coefficient interval of average ($n = 6$, $P = 95$ %): 0,02

content [% Cr_2O_3 (dry matter)]: 3,40 ± 0,02

11 Test report

The test report shall include the following:

a) a reference to this part of ISO 5398 (ISO 5398-2:2009);

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 based on dry sample mass to 0,01 % for samples containing less than 1 % Cr_2O_3 and to 0,1 % for samples containing 1 % or more of Cr_2O_3;

f) details of any deviations from the described procedures.