
INTERNATIONAL STANDARD



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Leather — Determination of sulphated total ash and sulphated water-insoluble ash

Cuir — Dosage des cendres sulfatées totales et des cendres sulfatées insolubles dans l'eau

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4047 was developed by Technical Committee ISO/TC 120, *Leather*, and was circulated to the member bodies in July 1976.

It has been approved by the member bodies of the following countries :

Australia	Hungary	Poland
Brazil	India	Romania
Canada	Iran	South Africa, Rep. of
Chile	Israel	Turkey
Czechoslovakia	Italy	United Kingdom
Egypt, Arab Rep. of	Mexico	Yugoslavia
France	Netherlands	
Germany	New Zealand	

No member body expressed disapproval of the document.

This International Standard is based on method IUC/7 of the International Union of Leather Technologists' and Chemists' Societies.

Leather — Determination of sulphated total ash and sulphated water-insoluble ash

0 INTRODUCTION

The amount of mineral substances found by ashing leather may differ from the actual content owing to decomposition, reduction, or the escape by volatilization of certain salts. By treating the ash with sulphuric acid, the salts and oxides are converted into sulphates, but some salts will again be transformed into oxides at the selected temperature of ignition.

To determine the total mineral content, for example within the framework of a complete leather analysis, the water-soluble and water-insoluble inorganic substances can be ascertained by calculation or determined separately.

Ammonium salts are not determined by this method (compare with ISO 4098).

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the sulphated total ash and the sulphated water-insoluble ash of leather.

The method is applicable to all types of leather.

The determination may be inaccurate by the extent to which the leather contains organo-metallic compounds, for example silicone (see note 1 in 8.1).

2 REFERENCES

ISO 2418, *Leather — Laboratory samples — Location and identification*.

ISO 2588, *Leather — Sampling — Number of items for a gross sample*.

ISO 4044, *Leather — Preparation of chemical test samples*.¹⁾

ISO 4098, *Leather — Determination of water-soluble matter, water-soluble inorganic matter and water-soluble organic matter*.¹⁾

3 DEFINITIONS

For the purposes of this International Standard, the following definitions apply :

3.1 sulphated total ash : The residue obtained from carbonized leather at 800 °C after sulphating the ash under the specified conditions.

3.2 sulphated water-insoluble ash : The residue obtained when leather, previously extracted with water as specified in ISO 4098, is carbonized after sulphating the ash under the specified conditions.

4 PRINCIPLE

Carbonization of the leather followed by treatment with sulphuric acid and ashing in an open crucible.

5 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Sulphuric acid, approximately 2 N solution.

5.2 Ammonium nitrate, approximately 100 g/l solution.

6 APPARATUS

Ordinary laboratory apparatus and in particular

6.1 Crucibles and dishes of glazed porcelain, platinum or quartz.

6.2 Muffle furnace, capable of being maintained at a temperature close to, but not exceeding, 800 °C (see note 4 in 8.1).

1) At present at the stage of draft.

7 SAMPLING

7.1 Whole pieces of leather

In the absence of any other agreement on sampling between the interested parties, the procedure specified in ISO 2588 for sampling from a lot shall be followed. Samples shall be taken from the pieces as specified in ISO 2418.

7.2 Other applications

Sampling shall be carried out as required by the relevant specification or contract.

8 PROCEDURE

8.1 Sulphated total ash

Prepare the sample as specified in ISO 4044 (see note 1). Weigh, to the nearest 0,001 g, 2 to 5 g of the sample and carefully carbonize it over a low flame in a crucible (6.1) which has been previously heated to 800 °C, cooled and weighed, so that the leather burns with a small flame. Carbonize fat-liquored leather particularly carefully so that the grease burns very slowly. Then thoroughly moisten with the sulphuric acid solution (5.1) and heat over a low flame until sulphur trioxide fumes are no longer visible. Heat more vigorously, and then ignite in the furnace (6.2), maintained at 800 °C, until completely ashed (see notes 2, 3 and 4). Cool in a desiccator and weigh. Repeat the addition of acid and the heating, cooling and weighing operations until the mass of the residue is constant (see note 5).

NOTES

1 It is advisable to extract leather impregnated with volatile organo-metallic compounds with dichloromethane before determining the sulphated total ash.

2 If a carbon-free residue cannot be obtained in spite of heating at 800 °C, the residue should be moistened with a little ammonium nitrate solution (5.2) and the heating repeated until the ash is free from carbon.

3 If complete ashing is not possible, even with the aid of ammonium nitrate, extract the contents of the crucible with hot water and filter through an ash-free filter paper. Ash the carbon residue together with the filter paper, add the filtrate to the contents of the crucible, evaporate on a water bath, again heat at 800 °C until the last traces of visible carbon are removed, cool in a desiccator and weigh.

4 At temperatures above 800 °C, some loss of mass from the residue is possible due to volatilization of certain inorganic salts. For this reason, close control is essential to prevent the maximum furnace temperature from exceeding 800 °C.

5 The sulphated total ash should be retained, if required for the determination of its components (for example chromic oxide, aluminium oxide, iron oxide) and for the determination of the cations present in the mineral impurities.

8.2 Sulphated water-insoluble ash

Extract the water-soluble substances from the prepared ground leather as specified in ISO 4098. Air-dry the extracted sample (see note). Continue as specified in 8.1.

NOTE — For the separate determination of sulphated water-insoluble ash, it is not absolutely necessary for the whole of the extracted and dried leather left after determination of the water-soluble matter, in accordance with ISO 4098, to be ashed. The residual air-dried leather can be mixed and weighed and an adequate part of it ashed.

9 EXPRESSION OF RESULTS

9.1 Calculation

9.1.1 The sulphated total ash is given, as a percentage by mass, by the formula

$$\frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the original sample of leather;

m_1 is the mass, in grams, of sulphated total ash.

9.2.1 The sulphated water-insoluble ash is given, as a percentage by mass, by the formula

$$\frac{m_2}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the original sample of leather;

m_2 is the mass, in grams, of the sulphated water-insoluble ash.

NOTE — The percentage of sulphated water-insoluble ash may alternatively be calculated by deducting from the percentage of total sulphated ash the percentage of sulphated ash from the water-soluble matter, if this has been separately determined.

9.2 Repeatability

The results of duplicate determinations by the same operator should not differ by more than 0,1 %, calculated on the original sample mass. If the difference is greater, the determination shall be carried out once more.

10 TEST REPORT

The test report shall include the following particulars :

- a reference to this International Standard;
- complete identification of the sample;
- the results obtained, to one decimal place, and the mean value;
- details of any special circumstances which may have affected the results.