

**BRITISH STANDARD**

---

**BS EN ISO  
4047:1998**

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

The European Standard EN ISO 4047:1998 has the status of a  
British Standard

ICS 59.140.30

**NO COPYING WITHOUT BSI PERMISSION EXCEPT AS PERMITTED BY COPYRIGHT LAW**

---



\* S \*

## National foreword

This British Standard is the English language version of EN ISO 4047:1998. It is identical with ISO 4047:1977. It supersedes BS 1309:1974, clause 6, which is withdrawn by amendment.

The UK participation in its preparation was entrusted to Technical Committee TC1/69, Footwear and leather, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

### Cross-references

Attention is drawn to the fact that CEN and CENELEC standards normally include an annex which lists normative references to international publications with their corresponding European publications. The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

**Compliance with a British Standard does not of itself confer immunity from legal obligations.**

### Summary of pages

This document comprises a front cover, an inside front cover, the EN ISO title page, the EN ISO foreword page, the ISO title page, the ISO foreword page, pages 1 and 2, an inside back cover and a back cover.

This British Standard, having been prepared under the direction of the Sector Committee for Materials and Chemicals, was published under the authority of the Standards Committee and comes into effect on 15 November 1998

© BSI 1998

ISBN 0 580 30781 6

### Amendments issued since publication

| Amd. No. | Date | Text affected |
|----------|------|---------------|
|          |      |               |
|          |      |               |
|          |      |               |
|          |      |               |

EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

EN ISO 4047

September 1998

ICS 59.140.30

Descriptors: see ISO document

English version

Leather - Determination of sulphated total ash and sulphated  
water-insoluble ash (ISO 4047:1977)

Cuir - Dosage des cendres sulfatées totales et des cendres  
sulfatées insolubles dans l'eau (ISO 4047:1977)

Leder - Bestimmung der sulfatierten Gesamtasche und der  
sulfatierten wasserunlöslichen Asche (ISO 4047:1977)

This European Standard was approved by CEN on 9 August 1998.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

© 1998 CEN All rights of exploitation in any form and by any means reserved  
worldwide for CEN national Members.

Ref. No. EN ISO 4047:1998 E

**EN ISO 4047:1998****Foreword**

The text of the International Standard from Technical Committee ISO/TC 120 "Leather" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 289 "Leather", the secretariat of which is held by UNI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 1999, and conflicting national standards shall be withdrawn at the latest by March 1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

**Endorsement notice**

The text of the International Standard ISO 4047:1977 has been approved by CEN as a European Standard without any modification.

---

# INTERNATIONAL STANDARD 4047

---

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION · МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ · ORGANISATION INTERNATIONALE DE NORMALISATION

---

## 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*

First edition — 1977-11-01

UDC 675 : 543.82

Ref. No. ISO 4047-1977 (E)

**Descriptors :** leather, chemical analysis, determination of content, ash, mineral matter, sulphating analysis.

Price based on 2 pages

**EN ISO 4047:1998****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*.<sup>1)</sup>

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

## 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).

<sup>1)</sup> At present at the stage of draft.

## EN ISO 4047:1998

### 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.



**BS EN ISO  
4047:1998**

---

**BSI — British Standards Institution**

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

**Revisions**

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: 0181 996 9000. Fax: 0181 996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

**Buying standards**

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: 0181 996 7000. Fax: 0181 996 7001.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

**Information on standards**

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: 0181 996 7111. Fax: 0181 996 7048.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: 0181 996 7002. Fax: 0181 996 7001.

**Copyright**

Copyright subsists in all BSI publications. BSI also holds the copyright, in the UK, of the publications of the international standardization bodies. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI.

This does not preclude the free use, in the course of implementing the standard, of necessary details such as symbols, and size, type or grade designations. If these details are to be used for any other purpose than implementation then the prior written permission of BSI must be obtained.

If permission is granted, the terms may include royalty payments or a licensing agreement. Details and advice can be obtained from the Copyright Manager. Tel: 0181 996 7070.

BSI  
389 Chiswick High Road  
London  
W4 4AL