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## Leather – Determination of total content of certain bisphenols

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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.

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](http://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](http://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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

ISO 11936 was prepared by the Chemical Test 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).

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.

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](http://www.iso.org/members.html).

## Introduction

This analytical method, ISO 11936, is a procedure for analysing certain bisphenols using liquid chromatography (LC) equipment. With this analytical method bisphenol A, bisphenol B, bisphenol F and bisphenol S can be determined.

In the leather industry, bisphenol F can be an impurity in synthetic tanning agents. Bisphenol S is a monomer that is used to manufacture synthetic tanning agents, which can lead to residues in the final product.

Bisphenol A is an endocrine disruptor for environmental organisms. Bisphenol A is a synthetic organic chemical primarily used as a monomer in the manufacture of high performance plastics, other polymers such as resins and in the color developer for thermoprint paper.

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# Leather – Determination of total content of certain bisphenols

## 1 Scope

This document is a method for determining the total content (solvent extractible) of the following bisphenols in leather:

- bisphenol A;
- bisphenol B;
- bisphenol F;
- bisphenol S.

This method requires the use of liquid chromatography (LC) with triple quadrupole mass spectrometer (MS/MS) or with ultraviolet (UV) detector or with Diode Array Detector (DAD) or with Fluorescence Detector (FLD) to identify and quantify the bisphenols.

NOTE 1 This method can also be used for other bisphenols if they are validated by the laboratory.

NOTE 2 The main difference between LC-MS/MS and LC-UV, LC-DAD or LC-FLD is the quantification limit (9.2).

## 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*

## 3 Terms and definitions

No terms and definitions are listed in this document.

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/>

## 4 Principle

The leather sample is extracted in methanol using an ultrasonic bath. Subsequently, an aliquot of the solution can be directly analysed, without further cleaning of the sample, using LC-MS/MS or LC with an ultraviolet detector (LC-UV), or diode array detector (LC-DAD), or fluorescence detector (LC-FLD).

## 5 Apparatus and materials

Normal laboratory apparatus and, in particular, the following:

- 5.1 **Ultrasonic bath**, with controllable heating capable of maintaining a temperature of  $(60 \pm 5)$  °C,
- 5.2 **Glass container with a screw cap**, for example, 22 ml volume has been found suitable,
- 5.3 **Polypropylene or polyethylene syringe**, 2 ml,
- 5.4 **Syringe membrane filters**, pore size 0,2 µm, for use with syringe (5.3),
- 5.5 **Volumetric flasks**, 10 ml and 100 ml,
- 5.6 **Analytical balance**, weighing to 1 mg,
- 5.7 **Pipettes**, various sizes, 1 ml to 5 ml,
- 5.8 **Instrumental equipment**, LC-MS/MS,
- 5.9 **Alternative instrumental equipment**, LC-UV, or LC-DAD, or LC-FLD.

NOTE If two detectors are used, they can be arranged in series on the same LC system.

## 6 Chemicals

If not otherwise defined, analytical reagent grade chemicals shall be used.

- 6.1 **Methanol**, CAS No. 67-56-1, for LC-MS/MS it is necessary to have LC-MS quality,

- 6.2 **Water**, deionised or distilled water, grade 3 according to ISO 3696,

- 6.3 **Bisphenol A**, CAS No. 80-05-7,

- 6.4 **Bisphenol B**, CAS No. 77-40-7,

- 6.5 **Bisphenol F**, CAS No. 620-92-8,

- 6.6 **Bisphenol S**, CAS No. 80-09-1,

- 6.7 **Stock solutions of a mix of bisphenol A, B, F, S**;  $\rho = 1$  mg/l and 10mg/l,

10 or 1mg of the respective bisphenols: A (6.3), B (6.4), F (6.5), S (&.6); are dissolved in different 100 ml volumetric flasks (5.5) with methanol (6.1) and filled up to the mark to obtain respectively the concentration of 1mg/l and 10mg/l.

- 6.8 **Internal standard**,  $\rho = 50$  mg/l,

When using LC-MS, the use of an internal standard is highly advisable to avoid matrix effects.

An example of a suitable mass-labelled internal standard is:

- Bisphenol A-D8, CAS No. 92739-58-7



Prepare a 50 mg/l solution of the internal standard by diluting the commercial solution with methanol.

## 6.9 Calibration solutions of bisphenols

Prepare four calibration solutions of  $\rho = 20 \mu\text{g/l}$  to  $\rho = 1000 \mu\text{g/l}$  of bisphenols using the stock solutions (6.7), see Table 1.

**Table 1 — Example of calibration solutions for LC-MS/MS**

Concentration ( $\mu\text{g/l}$ )	20	50	100	200	500	1000
Volume methanol ( $\mu\text{l}$ ) (6.1)	980	950	900	980	950	900
Volume of mix of bisphenols, 1 mg/l ( $\mu\text{l}$ ) (6.7)	20	50	100			
Volume of mix of bisphenols, 10 mg/l ( $\mu\text{l}$ ) (6.7)				20	50	100
Volume of internal standard at 50 mg/l ( $\mu\text{l}$ ) (6.8)	20	20	20	20	20	20

## 7 Leather sample

The leather sample shall be taken according to ISO 2418. If a sample according to ISO 2418 is not possible (e.g. in case of leather from finished products like shoes, clothing, etc.) the details of how the sample is taken shall be given in the test report. Glue residuals shall be mechanically removed from leather samples.

The leather samples shall be cut into small pieces according to ISO 4044.

## 8 Procedure

### 8.1 Extraction

Weigh  $(1,0 \pm 0,1)$  g of the leather sample with analytical balance (5.6) in a screw-top glass container (5.2) and add 20 ml methanol (6.1). Close the container and place it for  $(60 \pm 5)$  min in an ultrasonic bath (5.1) at  $(60 \pm 5)$  °C.

### 8.2 Analysis

After cooling down to room temperature, an aliquot of the extraction solution is removed using a disposable syringe (5.3) and transferred into a LC sample vial. The aliquot is now ready for the LC analysis.

The detection of the bisphenols is made using a LC-MS/MS (5.8) or alternatively with a LC-UV/LC-DAD/LC-FLD (5.9). Guidelines for suitable chromatographic conditions are given in Annex A and Annex B, respectively.

## 9 Expression of results

### 9.1 Calculation

The content of each bisphenol is calculated as mass portion,  $w$ , in milligrams per kilogram (mg/kg) of the leather sample according to the following [formula \(1\)](#):

$$w = \frac{(A_s - b) \cdot V}{a \cdot m_E} \quad (1)$$

where

$A_s$  is the peak area of each bisphenol in the extraction solution;

$b$  is the intercept of the calibration graph;

$a$  is the slope of the calibration graph;

$V$  is the final volume used (20 ml);

$m_E$  is the mass of the leather sample in grams (g).

## 9.2 Precision

With this method it is feasible to reach limits of quantification (LOQ) of 20 mg/kg for each bisphenol with LC-MS/MS and 100mg/kg with LC-UV, LC-DAD or LC-FLD.

## 10 Test report

The test report shall include at least the following information:

- a) a reference to ISO 11936;
- b) type, origin, and denomination of the leather sample;
- c) date of receipt and date of examination;
- d) the type LC equipment used;
- d) identification and quantification of each bisphenol expressed in milligrams per kilogram (mg/kg);
- e) any deviations from the procedure.