

SLOVENSKI STANDARD oSIST prEN ISO 21135:2023

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Kemikalije za industrijo strojenja usnja - Določevanje celotne vsebnosti nekaterih bisfenolov (ISO/DIS 21135:2023)

Chemicals for the leather tanning industry - Determination of the total content of certain bisphenols (ISO/DIS 21135:2023)

Chemikalien für die Gerbereiindustrie - Bestimmung des Gesamtgehalts von einzelnen Bisphenolen (ISO/DIS 21135:2023)

Produits chimiques pour l'industrie du tannage du cuir - Détermination de la teneur totale en certains bisphénols (ISO/DIS 21135:2023)

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DRAFT INTERNATIONAL STANDARD ISO/DIS 21135

IULTCS

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Chemicals for the leather tanning industry - Determination of the total content of certain bisphenols

ICS: 59.140.30

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Foreword

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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 document 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).

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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.

This document 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*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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 <u>www.iso.org/members.html</u>.

Introduction

This document includes a procedure for analysing certain bisphenols in leather tanning chemicals using liquid chromatography (LC) equipment. With this analytical method, bisphenol A, bisphenol AF, 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 a synthetic organic chemical primarily used as a monomer in the manufacture of highperformance plastics, other polymers, such as resins, and in the colour developer for thermoprint paper. Bisphenol AF is a fluorinated organic compound that is an analogue of bisphenol A in which the two methyl groups are replaced with trifluoromethyl groups. Bisphenol B is similar to bisphenol A and is used in the manufacture of plastics and resins.

At present, the official European Chemicals Agency (ECHA) classification recognised in the European Union (EU) is the following:

- Bisphenol A as toxic to reproduction, skin sensitiser and Endocrine Disruptor^[1].
- Bisphenol B as Endocrine Disruptor^[2].
- Bisphenol S as toxic to reproduction and Endocrine Disruptor^[3].

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DRAFT INTERNATIONAL STANDARD

Chemicals for the leather tanning industry - Determination of the total content of certain bisphenols

1 Scope

This document specifies a method for determining the total content (solvent extractable) of the following bisphenols in chemicals for the leather tanning industry:

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

This method requires the use of liquid chromatography (LC) with either a single quadrupole mass spectrometer (MS), a triple quadrupole mass spectrometer (MS/MS), an ultraviolet (UV) detector, a diode array detector (DAD) or a fluorescence detector (FLD) to identify and quantify the bisphenols.

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

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2 Normative references

There are no normative references in this document.

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3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at <u>https://www.electropedia.org/</u>

4 Principle

The sample of the chemical is extracted in methanol using an ultrasonic bath. Subsequently, an aliquot of the solution can be directly analysed, using LC-MS, LC-MS/MS or LC with a UV detector (LC-UV), DAD (LC-DAD) or FLD (LC-FLD).

5 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used:

- **5.1** Ultrasonic bath, with controllable heating capable of maintaining a temperature of (60 ± 5) °C.
- 5.2 Glass container with a screw cap, e.g. volume of 20 ml.
- **5.3** Suitable syringe membrane filters, e.g. polyamide with pore size 0,2 μm.

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- **5.4** Volumetric flasks, e.g. volume of 10 ml and 100 ml.
- 5.5 LC vials, with cap, e.g. volume of 2 ml.
- **5.6** Analytical balance, with a resolution of 0,1 mg.
- **5.7 Pipettes**, various sizes, e.g. volume of 1 ml to20 ml.
- 5.8 Instrumental equipment, LC-MS/MS.
- 5.9 Alternative instrumental equipment, LC-MS, LC-UV, LC-DAD or LC-FLD.
- NOTE If two detectors are used, they can be arranged in series on the same LC system.

6 Reagents

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

6.1 Methanol, CAS Registry Number[®] (CAS RN[®])¹) 67-56-1, for LC-MS/MS it is necessary to have LC-MS quality. HPLC quality methanol is suitable for LC-UV, LC-DAD or LC-FLD.

6.2 Water, deionised or distilled, ultra-pure quality for LC-MS and LC-MS/MS, HPLC grade for LC-DAD, UV and FLD.

6.3 Bisphenol A, CAS RN® 80-05-7, minimum 98,0 %. S. itch. ai)

- 6.4 Bisphenol AF, CAS RN® 1478-61-1, minimum 98,0 %. 135:2023
 https://standards.iteh.al/catalog/standards/sist/f1bbac22-e34f-4918-88666.5 Bisphenol B, CAS RN® 77-40-7, minimum 98,0 %. p-iso-21135-2023
- **6.6 Bisphenol F,** CAS RN® 620-92-81, minimum 98,0 %.
- **6.7 Bisphenol S,** CAS RN® 80-09-1, minimum 98,0 %.
- **6.8** Stock solutions of a mix of bisphenol A, AF, B, F and S, $\rho = 1 \text{ mg/l}$, 10 mg/l and 50 mg/l.

EXAMPLE 10 mg of each of the respective bisphenols, A (6.3), AF (6.4) B (6.5), F (6.6) and S (6.7), is dissolved in separate 100 ml volumetric flasks (5.4) with methanol (6.1). Mixed stock solutions are prepared to obtain, respectively, concentrations of 1 mg/l, 10 mg/l and 50mg/l in methanol.

6.9 Internal standard, $\rho = 50 \text{ mg/l}$.

When using LC-MS or LC-MS/MS, the use of internal standards for each type of bisphenol is highly recommended to avoid matrix effects.

Examples of suitable mass-labelled internal standards:

¹⁾ CAS Registry Number[®] (CAS RN[®]) is a trademark of CAS Corporation. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

for bisphenol A:	bisphenol A-D8, CAS RN® 92739-58-7;		
	bisphenol A-D16, CAS RN® 96210-87-6;		
for bisphenol B:	bisphenol B-D8, CAS RN® to be assigned;		
for bisphenol F:	bisphenol F-D10, CAS RN® 620-92-8;		
for bisphenol S:	bisphenol S-D8, CAS RN® ¹ 80-09-1.		

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

6.10 Calibration solutions of bisphenols.

Prepare at least four calibration solutions of $\rho = 20 \ \mu g/l$ to $\rho = 1 \ 000 \ \mu g/l$ of bisphenols using the stock solutions (6.8), see Table 1 for LC-MS/MS or LC/MS. Internal standard is only added if the MS technique is used for detection. In other cases, the internal standard volume is replaced by methanol. For LC-DAD or UV or FLD techniques prepare at least 4 calibration solutions of $\rho = 2 \ mg/l$ to $\rho = 50 \ mg/l$ using the stock solutions (6.8), see Table 2.

Concentration μg/l	Volume methanol eh S ^{µl} _(6.1) ANI (stand	Volume of mix of bisphenols 1 mg/l μl (6.8)	Volume of mix of bisphenols 10 mg/l (6.8)	Volume of inter- nal standard at 50 mg/l μl (<u>6.9</u>) (only for MS detection)
20	960	20		20
50	930 <u>08181 p</u>	rEN ISO 50 135:202	$\frac{3}{20}$	20
100 100 100 100 100 100 100 100 100 100	880		aczz-co41-4910-000 5 0002	20
200	960	r0313t-p1011-130-2113	20202020	20
500	930		50	20
1 000	880		100	20

Table 2 — Example of calibration solutions for LC-DAD or UV or FLD

Concentration	Volume methanol	Volume of mix of	Volume of mix of
mg/l	μl	bisphenols 10 mg/l	bisphenols 50 mg/l
	(<u>6.1</u>)	μΙ	μΙ
		(<u>6.8</u>)	(<u>6.8</u>)
2	800	200	
5	500	500	
10		1000	
20	600		400
50			1000

7 Sampling and sample preparation

The sample should be thoroughly mixed to get a representative test portion for analysis.

In the case of a powdered sample, if the particles are heterogeneous, manually or mechanically mill to homogenize the size of the particles.