SLOVENSKI STANDARD
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Tekstilije - Določevanje ftalatov - Tetrahidrofuranska metoda (ISO/DIS 14389:2021)

Textiles - Determination of the phthalate content - Tetrahydrofuran method (ISO/DIS 14389:2021)

Textilien - Bestimmung des Phthalatanteils - Tetrahydrofuran-Verfahren (ISO/DIS 14389:2021)

Textiles - Détermination de la teneur en phtalates - Méthode au tétrahydrofurane (ISO/DIS 14389:2021)

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Textiles — Determination of the phthalate content — Tetrahydrofuran method

Textiles — Détermination de la teneur en phtalates — Méthode au tétrahydrofurane

ICS: 59.060.01

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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).

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT),see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC *38*, *Textiles*.

This second edition cancels and replaces the first edition (ISO 14389:2014), which has been technically revised.

The main changes compared to the previous edition are as follows:

— in clause 5, four detected substances have been added, the original internal standard (DCHP) has been replaced with Benzyl 2-ethyl-hexyl phthalate and DCHP has been regarded as detected substance;

— in 6.3, the frequency of thermostatic ultrasonic bath has been deleted;

— in 7.2.1, “in duplicate” has been deleted;

— in 7.2.3, extract temperature has been changed from (60 ± 5) °C to about 60 °C;

— in Annex A, the example of determining the mass of the plastic component (coating) has been added.

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.

Introduction

This document covers a test method for the determination of some phthalates in textile articles.

Phthalates are commonly used as plasticizers in polymers. Phthalates are an issue for textile manufacturers and retailers due to their use within motifs, coated fabrics, plastisol prints, buttons, etc.

Phthalates are controversial because high doses of many phthalates have shown hormonal activity in rodent studies. Studies on rodents involving large amounts of phthalates have shown damage to the liver, the kidneys, the lungs, and the developing testes.

Due to their potential effect as endocrine disruptors, some of the listed phthalates are toxic in reproduction. The listed phthalates are based on those which have been restricted in some regulations (e.g. in the European Union).

Textiles — Determination of the phthalate content — Tetrahydrofuran method

WARNING — This document calls for the use of substances and/or procedures that might be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage. It has been assumed in the drafting of this document that the execution of its provisions is entrusted to appropriately qualified and experienced operators.

1 Scope

This document specifies a method of determining phthalates in textiles with gas chromatography–mass spectrometry (GC-MS) with mass selective detector.

This document is applicable to textile products where there is a risk of the presence of some phthalates.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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 https://www.electropedia.org/

3.1
plasticized or softened material
plastic material that is treated with chemicals to make it more flexible

Note 1 to entry: For this specific document, the chemicals are phthalates.

EXAMPLE Examples of plastic material: coating, pigment print binder, etc.

3.2
overall treated textiles
textiles with a continuous finish, coating or print

3.3
locally treated textiles
textiles with a discontinuous finish, coating or print

3.4
representative specimen
specimen obtained by mixing pieces of all the different treated parts and colours

4 Principle

The phthalates are extracted from textile specimen by ultrasonic generator with tetrahydrofuran. As the plastic polymer is partially or completed dissolved, the phthalate extraction is followed by the

precipitation of the dissolved polymer using the appropriate solvent (acetonitrile, *n*-hexane, etc.). After centrifugation and dilution of the extract to volume, gas chromatography–mass spectrometry (GC-MS) is used to identify individual phthalates in the specimen and quantify them by using an internal standard (IS).

5 Reagents

Unless otherwise specified, use only reagents of recognized analytical grade.

5.1 Tetrahydrofuran (THF), CAS number: 109-99-9.

5.2 Solvents used for the precipitation. Examples are:

5.2.1 Acetonitrile, CAS number: 75-05-8.

5.2.2 *n*-Hexane, CAS number: 110-54-3.

5.3 List of phthalates determined by this document in Table 1

Table 1 — List of phthalates

No.	Substance[a]	Abbreviation	CAS RN®
5.3.1	Di-cyclo-hexyl phthalate	DCHP	84-61-7
5.3.2	Di-iso-nonyl phthalate	DINP	28553-12-0 or 68515-48-0
5.3.3	Bis-(2-ethylhexyl) phthalate	DEHP	117-81-7
5.3.4	Di-n-octyl phthalate	DNOP	117-84-0
5.3.5	Di-iso-decyl phthalate	DIDP	26761-40-0 or 68515-49-1
5.3.6	Butyl benzyl phthalate	BBP	85-68-7
5.3.7	Di-butyl phthalate	DBP	84-74-2
5.3.8	Di-iso-butyl phthalate	DIBP	84-69-5
5.3.9	Di-n-pentyl phthalate	DPP	131-18-0
5.3.10	1,2-benzenedicarboxylic acid; Di-C_{6-8} -branched alkyl esters, C_7-rich	DIHP	71888-89-6
5.3.11	Bis-(2methoxyethyl) phthalate	DMEP	117-82-8
5.3.12	Di-iso-pentyl phthalate	DIPP	605-50-5
5.3.13	Di-n-hexyl phthalate	DNHP	84-75-3
5.3.14	N-pentyl-iso-pentyl phthalate	PIPP	776297-69-9
5.3.15	Di-iso-hexyl phthalate	DIHxP	71850-09-4
[a] Not all commercially available phthalate standards are of analytical grade.			

5.4 Benzyl 2-ethyl-hexyl phthalate, CAS number: 27215-22-1, internal standard (IS).

6 Apparatus

6.1 Gas chromatograph-mass spectrometry (GC-MS).

6.2 Vial, airtight glassware to be sealed with a PTFE septum. 40 ml vials have been found suitable.

6.3 Thermostatic ultrasonic bath, with controllable heating capable of maintaining a temperature of about 60 °C.

6.4 Glass flasks with glass stoppers. 100 ml glass flasks have been found suitable.

6.5 Calibrated volumetric flasks, of capacities 50 ml and 100 ml.

6.6 Volumetric device, of capacities 10 ml and 20 ml.

6.7 Balance, with a resolution of 0,1 mg.

6.8 Water bath.

6.9 Rotary evaporator.

7 Procedure

WARNING — The vapour of the organic solvents is highly flammable, especially at high temperature. Allow glassware to cool down before use.

Avoid direct contact between the samples and glassware and/or equipment used in order to minimize cross-contamination. Glassware, after washing, should be given an extra rinse with 0,1 N nitric acid, water and finally with acetone. Glassware should be completely dried before use. To avoid contamination, do not use any plastic container (e.g. for water).

7.1 Preparation of standard solutions

7.1.1 Internal standard solution

Prepare a 1 000 mg/l stock standard solution of the internal standard in the solvent used for the precipitation (5.2) after the ultrasonic extraction (see 7.2).

7.1.2 Preparation of stock standard solutions

Separately prepare a series of 1 000 mg/l individual stock standard solutions of the individual phthalate (5.3) in the solvent used for the precipitation.

For example, weigh 50,0 mg of a phthalate in a 50 ml volumetric flask and fill the volumetric flask up to the mark with the solvent used for the precipitation and mix thoroughly to dissolve completely the substance.

Most of the stock standards may be made in a mixed stock. This saves time and effort when preparing calibration solutions. DINP, DIDP and DIHP have overlapping peaks. It is recommended to make up these stock standard solutions separately, as their calibration solutions have to be made in higher (5x) concentrations than for the other phthalates because of their multi-peak nature.

7.1.3 Preparation of the calibration solutions

From the stock standard solutions, prepare at least five appropriate phthalate calibration solutions (example of concentrations at 1 mg/l, 3 mg/l, 15 mg/l, 30 mg/l and 90 mg/l as described in the Table 2), each containing an equal amount of the target phthalates (5.3) and an amount of internal standard (5.4) in a mixture of tetrahydrofuran and the solvent used for the precipitation, mixed by volume at a ratio of 1:2 (33 parts tetrahydrofuran to 66 parts of the other solvent), as shown in Table 2. Each calibration solution should have a final internal standard concentration of 5 mg/l. Prepare one calibration blank. Analyse the calibration solutions and calibration blank with the GC-MS. Qualitatively analyse the result to ensure proper retention times and the absence of contamination and built up the calibration curve.

Table 2 — Examples of calibration solutions

Concentration	Instructions
Blank	In a 50 ml volumetric flask, add 0,25 ml of internal standard (5.4) stock solution; then, fill up to the mark with a mixture of tetrahydrofuran and the solvent used for the precipitation, mixed by volume at a ratio of 1:2 (33 parts tetrahydrofuran to 66 parts of the other solvent)
1 mg/l	Add 0,1 ml of each stock standard solution in 100 ml volumetric flask plus 0,5 ml of the internal standard (5.4) stock solution; then, fill up to the mark with a mixture of 33 parts of tetrahydrofuran to 66 parts of the solvent used for the precipitation.
3 mg/l	Add 0,3 ml of each stock standard solution in 100 ml volumetric flask plus 0,5 ml of the internal standard (5.4) stock solution; then, fill up to the mark with a mixture of 33 parts of tetrahydrofuran to 66 parts of the solvent used for the precipitation.
15 mg/l	Add 0,75 ml of each stock standard solution in 50 ml volumetric flask plus 0,25 ml of the internal standard (5.4) stock solution; then, fill up to the mark with a mixture of 33 parts of tetrahydrofuran to 66 parts of the solvent used for the precipitation.
30 mg/l	Add 1,5 ml of each stock standard solution in 50 ml volumetric flask plus 0,25 ml of the internal standard (5.4) stock solution; then, fill up to the mark with a mixture of 33 parts of tetrahydrofuran to 66 parts of the solvent used for the precipitation.
90 mg/l	Add 4,5 ml of each stock standard solution in 50 ml volumetric flask plus 0,25 ml of the internal standard (5.4) stock solution; then, fill up to the mark with a mixture of 33 parts of tetrahydrofuran to 66 parts of the solvent used for the precipitation.

If target ions other than 149 are used for quantification of DEHP, DNOP, DINP, DIDP and DIHP, all the listed phthalates may be calibrated and quantified together.

Typical quantification ions for phthalates are shown in Annex B.

If DIDP and DINP overlap in the chromatogram; choose target ions indicated in Annex B.

In case the concentration of some phthalate in the extraction solution of a specimen lies outside the limits of the calibration curve, dilute the solution with a mixture of 33 parts of tetrahydrofuran to 66 parts of the solvent used for the precipitation containing 5 mg/l of the internal standard, so that the sample can be properly quantified.

NOTE The stock standard solutions are stored at 0 °C to 4 °C for up to six months, and the working solutions are stored at 0 °C to 4 °C for up to three months, or sooner if ongoing quality control indicates problems.

7.2 Ultrasonic extraction and determination of phthalates

7.2.1 General

Ultrasonic extraction is performed using tetrahydrofuran on test specimens prepared from each textile sample, followed by precipitation of the (partially or completed) dissolved plastic component using an appropriate solvent, centrifugation, and determination of phthalates. A blank is run in parallel to avoid errors caused by contamination from the laboratory environment.

NOTE For example, polyvinylchloride (PVC) is completely dissolved in tetrahydrofuran.

7.2.2 Preparation of test specimen

A representative test specimen shall be prepared by mixing and cutting pieces from every coated area/part of the textile sample (see Annex A for further information on coating types). Cut the representative specimen into small pieces (less than 5 mm in the greatest dimension), homogenize it, and weigh (0,30 ± 0,01) g of these pieces in the vial (6.2).

Using an appropriate volumetric graduated pipette (6.6), add to vial 10 ml of tetrahydrofuran (5.1) containing 5 mg/l of internal standard (5.4) and seal it tightly.

