
**Textiles — Determination of the
phthalate content — Tetrahydrofuran
method**

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

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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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 248, *Textiles and textile products*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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

The main changes are as follows:

- addition of the [Clause 2](#) “Normative references” and renumbering of subsequent clauses;
- replacement of the original internal standard (DCHP) with Benzyl 2-ethyl-hexyl phthalate;
- addition of five detected phthalates (including DCHP);
- replacement of the term “plasticized or soften material” with “plasticized material” in [3.1](#) (former 2.1);
- revision of the terms and definitions in [3.2](#) (former 2.2) and [3.3](#) (former 2.3);
- deletion of the frequency of thermostatic ultrasonic bath in [6.3](#) (former [5.3](#));
- deletion of “in duplicate” in [7.3.1](#) (former 6.2.1);
- revision of extract temperature from “(60 ± 5) °C” to “about 60 °C” in [7.3.3](#) (former 6.2.3);
- revision of [Formula \(1\)](#) and [Formula \(2\)](#) in [Clause 8](#) (former Clause 7);
- addition of the example of determining the mass of the plastic component (coating) in [Annex A](#).

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 (for example, in the European Union).

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Textiles — Determination of the phthalate content — Tetrahydrofuran method

WARNING — This document calls for the use of substances and/or procedures that can 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).

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 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

with a continuous finish, coating or print

3.3

locally treated

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 bath with tetrahydrofuran. As the plastic polymer is partially or completely dissolved, the phthalate extraction is followed by the

precipitation of the dissolved polymer using the appropriate solvent (acetonitrile, *n*-hexane, etc.). Centrifugate the extract and then take a suitable volume of the organic solution for analysis. 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 Registry Number^{®1)} (CAS RN[®]): 109-99-9.

5.2 Solvents used for the precipitation. Examples are:

a) **Acetonitrile**, CAS Registry Number[®] (CAS RN[®]): 75-05-8; or

b) ***n*-Hexane**, CAS Registry Number[®] (CAS RN[®]): 110-54-3.

5.3 The list of **phthalates** determined by this method is given in [Table 1](#). Other phthalates can be analysed by this method if the laboratory performs a validation.

Table 1 — List of phthalates

No.	Substance ^a	Abbreviation	CAS RN [®]
1	Di-cyclo-hexyl phthalate	DCHP	84-61-7
2	Di-iso-nonyl phthalate	DINP	28553-12-0 or 68515-48-0
3	Bis-(2-ethylhexyl) phthalate	DEHP	117-81-7
4	Di- <i>n</i> -octyl phthalate	DNOP	117-84-0
5	Di-iso-decyl phthalate	DIDP	26761-40-0 or 68515-49-1
6	Butyl benzyl phthalate	BBP	85-68-7
7	Di-butyl phthalate	DBP	84-74-2
8	Di-iso-butyl phthalate	DIBP	84-69-5
9	Di- <i>n</i> -pentyl phthalate	DPP	131-18-0
10	1,2-benzenedicarboxylic acid; Di-C ₆₋₈ -branched alkyl esters, C ₇ -rich	DIHP	71888-89-6
11	Bis-(2-methoxyethyl) phthalate	DMEP	117-82-8
12	Di-iso-pentyl phthalate	DIPP	605-50-5
13	Di- <i>n</i> -hexyl phthalate	DNHP	84-75-3
14	<i>N</i> -pentyl-iso-pentyl phthalate	PIPP	776297-69-9
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 Registry Number[®] (CAS RN[®]): 27215-22-1, internal standard (IS). Other internal standards (for example, deuterated phthalates) can be used if the laboratory performs a validation.

6 Apparatus

6.1 Gas chromatography, coupled with mass spectrometer (GC–MS).

1) CAS Registry Number[®] 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.

- 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 in water bath.
- 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 Analytical balance**, with a resolution of 0,1 mg.
- 6.8 Centrifuge**.

7 Procedure

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

7.1 Cross-contamination provisions

Glassware and/or equipment used shall be free from phthalates in order to minimize cross-contamination. For pre-used glassware, after washing, should be given an extra rinse with 0,1 mol/l nitric acid, water and finally with acetone. Glassware should be completely dried before use. To avoid contamination, do not use any plastic container (for example, for water).

7.2 Preparation of standard solutions

7.2.1 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 7.3).

7.2.2 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 (for example, 5 times) concentrations than for the other phthalates because of their multi-peak nature.

7.2.3 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 (1/3 of tetrahydrofuran to 2/3 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 by 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

Operation items	Blank	L1	L2	L3	L4	L5
Concentration of phthalate (mg/l)	0	1	3	15	30	90
Volumetric flask (ml)	50	100	100	50	50	50
Volume of phthalate stock solution (ml)	/	0,1	0,3	0,75	1,5	4,5
Volume of internal solution (ml)	0,25	0,5	0,5	0,25	0,25	0,25
Fill up to the mark	with a mixture of 1/3 of tetrahydrofuran to 2/3 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 1/3 of tetrahydrofuran to 2/3 of the solvent used for the precipitation containing 5 mg/l of the internal standard, so that the phthalates in 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.

7.3 Ultrasonic extraction and determination of phthalates

7.3.1 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.

7.3.2 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 device ([6.6](#)), add to vial 10 ml of tetrahydrofuran ([5.1](#)) containing 5 mg/l of internal standard ([5.4](#)) and seal it tightly.

7.3.3 Extraction procedure

Place the vial in the ultrasonic bath ([6.3](#)) at about 60 °C for 1 h ± 5 min to allow the extraction of phthalates (by complete or partial dissolution of the polymer). Then, remove the vial from the bath and let it stand still until the extraction solution cools down to room temperature.

Ensure that the vial remain airtight in the ultrasonic bath ([6.3](#)) throughout the entire extraction process.

Using an appropriate volumetric device (6.6), add dropwise to the vial 20 ml of the solvent used for the precipitation (5.2) containing 5 mg/l of internal standard.

Shake the vial vigorously (preferably on a vortex type shaker) for at least 30 s and allow them to stand still for (30 ± 2) min in order to allow the precipitation of polymer.

Centrifugate the vial at least 700 *g* (for example, 2 500 min⁻¹ for 10 cm radius) by the centrifuge (6.8) for at least 10 min to drive and settle any remaining suspended precipitates of polymer in organic phase to the bottom to obtain a transparent organic solution. Then, withdraw and transfer a specimen aliquot of the organic solution into a suitable GC sampling vial for GC-MS analysis.

CAUTION — Direct injection into GC-MS of suspended matter solution or cloudy solution might contaminate the equipment.

If necessary, prepare further diluted solutions using an appropriate volume of a mixture of tetrahydrofuran (5.1) and the solvent used for the precipitation (5.2) mixed by volume at a ratio of 1:2 (1/3 of tetrahydrofuran to 2/3 of the other solvent) containing 5 mg/l of the internal standard (5.4) and then repeat the analysis.

7.3.4 Phthalates determination

Determine the phthalates extracted in 7.3.3 by GC-MS (6.1).

An example of the parameters for GC-MS analysis of target phthalates is given in Annex B.

The statistical data of interlaboratory trials in the first edition is shown in Annex D.

In some cases when the phthalates level is very low, it may be necessary to increase the mass of the test specimens.

8 Calculation of the results

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8.1 Calculation based on the corrected mass (by default)

From the calibration graph, determine the response of each phthalate, corrected for the internal standard peak area, and interpolate the concentration of the phthalate, in milligrams per litre (mg/l), correcting for any dilutions. Subtract the blank concentration from the specimen concentration. Calculate the result using Formula (1):

$$w_c = \frac{V \times (b - a) \times F}{m_c \times 10\,000} \quad (1)$$

where

w_c is the mass fraction of the individual phthalate, based on the corrected mass of the test specimen (%);

V is 30 ml, that is the total volume of the phthalate solution before any dilution (10 ml of tetrahydrofuran used for the extraction +20 ml of the solvent used in the precipitation step);

m_c is the corrected mass of specimen (g);

a is the concentration of the individual phthalate of the blank solution (mg/l);

b is the concentration of the individual phthalate of the specimen solution (mg/l);

F is the dilution factor.