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**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. [www.iso.org/directives](http://www.iso.org/directives)

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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

The committee responsible for this document is ISO/TC 38, *Textiles*.

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## Introduction

This International Standard 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).

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# Textiles — Determination of the phthalate content — Tetrahydrofuran method

**WARNING** — This International Standard 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 International Standard that the execution of its provisions is entrusted to appropriately qualified and experienced operators.

## 1 Scope

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

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

## 2 Terms and definitions

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

### 2.1

#### **plasticized or softened material**

plastic material that is treated with chemicals to make it more flexible

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

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

### 2.2

#### **overall treated textile**

textile with a continuous finish, coating or print

### 2.3

#### **locally treated textile**

textile with a discontinuous finish, coating or print

### 2.4

#### **representative specimen**

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

## 3 Principle

The phthalates are extracted from textile specimen by ultrasonic generator 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.). 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).

## 4 Reagents

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

- 4.1 **Tetrahydrofuran (THF)**, CAS number: 109-99-9.
- 4.2 **Solvents used for the precipitation.** Examples are:
- 4.2.1 **Acetonitrile**, CAS number: 75-05-8.
- 4.2.2 ***n*-Hexane**, CAS number: 110-54-3.
- 4.3 **Di-cyclohexyl phthalate (DCHP)**, CAS number: 84-61-7, internal standard (IS).
- 4.4 **Di-isononyl phthalate (DINP)**, CAS number 28553-12-0 or 68515-48-0.
- 4.5 **Di-(2-ethylhexyl) phthalate (DEHP)**, CAS number 117-81-7.
- 4.6 **Di-*n*-octyl phthalate (DNOP)**, CAS number 117-84-0.
- 4.7 **Di-iso-decyl phthalate (DIDP)**, CAS number 26761-40-0 or 68515-49-1.
- 4.8 **Butyl benzyl phthalate (BBP)**, CAS number 85-68-7.
- 4.9 **Di-butyl phthalate (DBP)**, CAS number 84-74-2.
- 4.10 **Di-iso-butyl phthalate (DIBP)**, CAS number 84-69-5.
- 4.11 **Di-pentyl phthalate (DPP)**, CAS number: 131-18-0.
- 4.12 **Di-iso-heptyl phthalate (DIHP)**, CAS number: 71888-89-6.
- 4.13 **Di-methoxyethyl phthalate (DMEP)**, CAS number: 117-82-8.

## 5 Apparatus

- 5.1 **Gas chromatograph-mass spectrometer (GC-MS)**, with mass selective detector (MSD).
- 5.2 **Vial**, of capacity 40 ml.
- 5.3 **Thermostatic ultrasonic bath**, with a frequency of  $(40 \pm 5)$  kHz.
- 5.4 **Glass flasks with glass stoppers**, of capacity 100 ml.
- 5.5 **Calibrated volumetric flasks**, of capacities 50 ml and 100 ml.
- 5.6 **Volumetric graduated pipette**, of capacities 10 ml and 20 ml.
- 5.7 **Balance**, with a resolution of 0,1 mg.
- 5.8 **Water bath**.
- 5.9 **Rotary evaporator**.



## 6 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).

### 6.1 Preparation of standard solutions

#### 6.1.1 Internal standard solution

Prepare a 1 000 mg/l stock standard solution of the internal standard in the solvent used for the precipitation (4.2) after the ultrasonic extraction (see 6.2).

#### 6.1.2 Preparation of stock standard solutions

Prepare a series of individual stock standard solutions of the individual phthalate ester in the solvent used for the precipitation as shown in Table 1.

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.

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Table 1 — Stock standard solutions

Phthalate ester	DCHP (IS)	DINP	DEHP	DNOP	DIDP	BBP	DBP	DIBP	DPP	DIHP	DMEP
Concentration, mg/l	1 000										

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. Hence, the higher limit of detection in Annex B.

#### 6.1.3 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 (4.4 through 4.13) and an amount of internal standard (4.3) 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 (DCHP) 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 (DCHP) 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 (DCHP) 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 (DCHP) 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 (DCHP) 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 (DCHP) 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 12 months, and the working solutions are stored at 0 °C to 4 °C for up to six months, or sooner if ongoing quality control indicates problems.

## 6.2 Ultrasonic extraction and determination of phthalates

### 6.2.1 General

Ultrasonic extraction is performed using tetrahydrofuran on test specimens prepared in duplicate from each textile sample, followed by precipitation of the (partially or completely) 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.

## 6.2.2 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. Cut the representative specimen into small pieces (less than 5 mm in the greatest dimension), homogenize it, and weigh  $(0,30 \pm 0,01)$  g of these pieces in each of two 40 ml airtight vials (5.2) sealed with a PTFE septum. Ensure that the vials remain airtight in the ultrasonic bath (5.3) throughout the entire extraction process.

Using a volumetric graduated pipette (5.6), add to each vial 10 ml of tetrahydrofuran (4.1) containing 5 mg/l of internal standard (4.3).

## 6.2.3 Extraction procedure

Place the vials in the ultrasonic bath (5.3) at  $(60 \pm 5)$  °C for  $1 \text{ h} \pm 5 \text{ min}$  to allow the extraction of phthalates (by complete or partial dissolution of the polymer). Then, remove the vials from the bath and let them stand still until the extraction solution cools down to room temperature.

Using a volumetric graduated pipette, add dropwise to each vial 20ml of the solvent used for the precipitation (4.2) containing 5mg/l of internal standard.

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

Centrifuge the vials at least 700 *g* (e.g. 2 500 r/min for 10 cm radius) 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 two suitable GC sampling vials for GC-MS analysis.

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

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If necessary, prepare further diluted solutions using an appropriate volume of a mixture of tetrahydrofuran (4.1) and the solvent used for the precipitation (4.2) mixed by volume at a ratio of 1:2 (33 parts tetrahydrofuran to 66 parts of the other solvent) containing 5mg/l of the internal standard (4.3) and then repeat the analysis.

## 6.2.4 Phthalates determination

Determinate the phthalates extracted in 6.2.3 by GC-MS (5.1). An example of a programme and the parameters for GC-MS analysis of target phthalates are given in Annex B.

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

## 7 Calculation of the results

### 7.1 Calculation based on the corrected mass (by default)

From the calibration graph, determine the response of each phthalate, taking into account 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):

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

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.

The determination of the corrected mass of test specimen is given in [Annex A](#), as well as an example of the calculation. Overall treated material is illustrated in [Figure A.1](#) and locally treated material in [Figure A.2](#).

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If the corrected mass of the test specimen,  $m_c$ , cannot be obtained, give a remark in the test report and calculate the result according to [7.2](#).

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## 7.2 Calculation based on the total mass (when relevant)

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If the corrected mass of the test specimen,  $m_c$ , as described in [Annex A](#), cannot be obtained and used for the calculation of the results, then calculate the result using Formula (2).

$$w_T = \frac{V \times (b \times F - a)}{m_T \times 10\,000} \quad (2)$$

where

- $w_T$  is the mass fraction of the individual phthalate, based on the total 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);
- $m_T$  is the total 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.