
**Footwear — Critical substances
potentially present in footwear and
footwear components —**

**Part 1:
Determination of phthalate with
solvent extraction**

*Chaussures — Substances critiques potentiellement présentes dans les
chaussures et les composants des chaussures —*

Partie 1: Détermination des phtalates par extraction au solvant

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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 216, *Footwear*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 309, *Footwear*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This first edition of ISO 16181-1, together with ISO 16181-2, cancels and replaces ISO/TS 16181:2011, which has been technically revised.

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

- addition of number of phthalates from 7 to 26;
- addition of new [Clause 2](#) and [Clause 3](#), and renumbering of subsequent clauses;
- replacement of “n-hexane/acetone” with “toluene” and alternative “tetrahydrofuran” as extraction solution;
- splitting of former [Clause 3](#) into [Clauses 5](#) and [6](#), with technical modification;
- technical revision of [Clauses 4](#) and [5](#);
- deletion of [5.2.4](#);
- addition of [Clause 9](#);
- addition of [Annex D](#).

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.

Footwear — Critical substances potentially present in footwear and footwear components —

Part 1:

Determination of phthalate with solvent extraction

WARNING — The use of this document can involve hazardous materials, operations and equipment. It does not purport to address all of the safety or environmental problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel and the environment prior to application of the document, and to determine the applicability of regulatory limitations for this purpose.

1 Scope

This document specifies a test method to determine the qualitative and quantitative presence of phthalate compounds (see [Annex A](#)) in footwear and footwear components.

NOTE 1 A list of relevant materials potentially containing phthalates can be found in ISO/TR 16178:2012, Annex A or in CEN/TR 16417.

NOTE 2 This test method can also be used to determine phthalates other than those listed in [Annex A](#), subject to validation.

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 4787:2010, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use*

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 test sample is extracted using toluene at 60 °C in an ultrasonic bath for 1 h. An aliquot is then analysed using a gas chromatograph (GC) with a mass selective detector (MS).

All the abbreviations of phthalates used are given in [Annex A](#).

When compared with ISO 16181-2, the two analytical methods should give similar trends but not necessarily the same absolute result. Therefore, in cases of dispute, the method in this document shall be used in preference to ISO 16181-2.

5 Reagents

5.1 Chemicals

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

5.1.1 Toluene, Chemical Abstract Service Registry Number¹⁾ (CAS RN[®]): 108-88-3.

5.1.2 Internal standard

Either of the following chemicals can be used as internal standard:

- Bis (2-Ethylhexyl) phthalates-3,4,5,6-D4 (CAS RN[®]: 93951-87-2);
- Diphenylphthalate (CAS RN[®] 84-62-8)

NOTE 1 Diphenylphthalate gives appropriate results in case of DEHP <1 %. In case of DEHP >1 % the results of all other phthalates can be affected because of poor resolution between DEHP and Diphenylphthalate.

NOTE 2 Other internal standards have been found convenient as Di-n-propylphthalate-3-4-5-6 D4 (CAS RN[®]: 358731-29-0).

5.1.3 Phthalates, see [Annexes A](#) and [B](#).

5.1.4 Acetone, CAS RN[®]: 67-64-1.

5.1.5 Methanol, CAS RN[®]: 67-56-1.

5.1.6 Cyclohexane, CAS RN[®]: 110-82-7.

5.1.7 Tetrahydrofurane (THF), CAS RN[®]: 109-99-9.

5.1.8 n-hexane, CAS RN[®]: 110-54-3.

5.2 Standard solutions

5.2.1 Target phthalates — Stock solution

Based on its tasks a laboratory shall decide which phthalates from [Table A.1](#) need to be determined.

Based on this decision, standard stock solution for each phthalate shall be available either as commercially available certified mixes, individual components in solution or self-prepared individual standard stock solutions of each phthalate in toluene ([5.1.1](#)).

Prepare, for example, standard stock solutions with concentrations of 1 000 µg/ml, weigh 50,0 mg of each phthalate depending on the purity ([Table A.1](#)) into 50 ml volumetric flasks, fill the volumetric flasks up to the mark with toluene ([5.1.1](#)) and mix thoroughly to dissolve completely the substance.

5.2.2 Internal standard — Stock solution

Prepare an internal standard stock solution that is in the same concentration range as the standard stock solutions ([5.2.1](#)) by dissolving the internal standard ([5.1.2](#)) in toluene ([5.1.1](#)) according to [5.2.1](#).

1) CAS Registry Number (CAS RN) is a registered trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named.

5.2.3 Calibration solutions

Prepare at least five appropriate phthalate calibration solutions, each containing an equal amount of the target phthalates (5.1.3) and an amount of internal standard (5.1.2) in toluene (5.1.1) respectively in n-hexane (5.1.8).

An examples of calibration solutions are shown in Table 1 and 2.

Table 1 — Example of calibration solutions extraction (8.1.1)

Standard	L1	L2	L3	L4	L5
Concentration of phthalate (µg/ml)	2,5	25	50	75	100
Volume of the phthalate stock solution (µl) (5.2.1)	25	250	500	750	1 000
Volume of the internal standard stock solution (µl) (5.2.2)	500				
Concentration of the internal standard (µg/ml)	50				
Volume of toluene (µl) (5.1.1) (completed to 10ml)	9 475	9 250	9 000	8 750	8 500

Table 2 — Example of calibration solutions with THF extraction (8.1.2)

Standard	L1	L2	L3	L4	L5
Concentration of phthalate (µg/ml)	1,0	3,0	15	30	90
Volume of the phthalate stock solution (µl) (5.2.1)	10	30	150	300	900
Volume of the internal standard stock solution (µl) (5.2.2)	500				
Concentration of the internal standard (µg/ml)	50				
Volume of n-hexane (µl) (5.1.8) (completed to 10 ml)	9 490	9 470	9 350	9 200	8 600

5.2.4 Extraction solution with internal standard (optional)

Prepare the extraction solution with the same concentration of an internal standard as in the calibration solutions (e.g. 50 µg/ml) by diluting internal standard working solution (5.2.2) with toluene (5.1.1).

6 Apparatus

The usual laboratory apparatus and laboratory glassware, according to ISO 4787:2010, shall be used, in addition to the following.

Glassware, after washing, should be given an extra rinse with 0,1 N nitric acid and finally with acetone, acetone/methanol and/or cyclohexane.

- 6.1 **Analytical balance**, with a precision of at least 0,1 mg.
- 6.2 **Flask with Teflon stopper**, for example 20 ml.
- 6.3 **Ultrasonic bath with adjustable temperature**, suitable for operation at 60 °C.
- 6.4 **PTFE-membrane filter**, pore width 0,45 µm.
- 6.5 **Volumetric flasks of suitable volume**.
- 6.6 **GC vials**, for example 2 ml.
- 6.7 **Gas chromatograph with mass-selective detector (GC-MS)**.

7 Sampling

The test piece consists of a single material taken from the footwear, such as coated leather, textile, polymer, coated material or others. The preparation of the sample should involve removal of the individual materials from the footwear.

Each material type is cut into pieces of about 3 mm to 5 mm edge length.

In order to avoid cross-contamination, the cutting device shall be cleaned with acetone after cutting of any individual material.

Up to three test specimens of equal mass and the same material type can be tested together, taking into consideration the limit of quantification and the limit given by the applicable regulations.

8 Test procedure

8.1 Extraction

8.1.1 Ultrasonic extraction

Weigh accurately ($1,0 \pm 0,1$) g of the pieces of a representative specimen (Clause 7) into a glass flask (6.2), record the mass (m) to the nearest 10 mg. Add 10 ml of extraction solution (5.2.4) to wet the entire specimen, and seal the flask with a Teflon stopper. If the test specimen is not sufficiently immersed in the extraction solution, add more solvent and report the final volume (V) for calculation of the phthalate amount.

Alternatively, toluene (5.1.1) can be used for extraction. In this case, an appropriate amount of internal standard solution (5.2.2) shall be added in order to get the same concentration of internal standard as in the calibration solutions.

Extract the phthalate in the ultrasonic bath (6.3) for $1 \text{ h} \pm 5 \text{ min}$ at $(60 \pm 5) \text{ }^\circ\text{C}$.

After cooling to room temperature, filter this solution through a PTFE membrane filter (6.4).

8.1.2 Alternative extraction procedure for PVC materials

For a PVC sample, THF can also be used as extraction solvent, and the following alternative extraction procedure may be applied.

- a) Weigh accurately ($0,5 \pm 0,01$) g of the pieces of a representative specimen (Clause 7) into a 50-ml glass flask fitted with a Teflon stopper. Add 10 ml of THF to wet the entire specimen.
- b) Extract the phthalates in the ultrasonic bath for $1 \text{ h} \pm 5 \text{ min}$ at $(50 \pm 5) \text{ }^\circ\text{C}$. For samples that have not completely dissolved at this point, add an additional $2 \text{ h} \pm 10 \text{ min}$ of extraction time and then proceed.
- c) Add 20 ml n-hexane to precipitate the sample matrix, then filter or centrifuge to obtain a transparent extraction solution.
- d) Transfer the extract to a 50-ml volumetric flask; fill to the mark with a mixture of THF and n-hexane at a volume ratio of 1:2.
- e) Transfer a known volume of organic phase into a suitable GC sampling vial, add an appropriate volume of internal standard solution in n-hexane and perform GC-MS analysis.

NOTE Annex F includes the results of a comparison of phthalates extraction phthalates by using THF/toluene.

8.1.3 Preparation of a method blank

For each series of tests, a method blank shall be prepared. For the preparation of the blank, the complete procedure (extraction [8.1.1](#) or [8.1.2](#) and GC-MS analysis [8.2](#)) shall be done in a 20-ml glass flask ([6.2](#)) without a sample.

8.2 Determination with GC-MS

Transfer an aliquot of the extract with internal standard into a suitable GC sampling vial ([6.6](#)) and seal with a cap.

Determine the phthalates extracted in [8.1](#) by GC-MS ([6.7](#)). An example of chromatographic conditions for GC-MS analysis of target phthalates are given in [Annex C](#). An example of chromatogram is given in [Annex E](#).

In cases when the phthalates level is very high, prepare further diluted solutions using the original solution and repeat the analysis. In the case of toluene ([5.1.1](#)), add an appropriate volume of internal standard solution.

9 Expression of results

9.1 Calibration curve

Set up the linear regression function by using the ratios (A_s/A_{is}) and (C_s/C_{is}) according to [Formula \(1\)](#).

$$\frac{A_s}{A_{is}} = a \times \left(\frac{C_s}{C_{is}} \right) + b \quad (1)$$

where

A_s is the area of the target phthalate based on the target ions;

A_{is} is the area of the peak of internal standard based on the target ions;

C_s is the concentration of the target phthalate in the calibration standard, in $\mu\text{g/ml}$;

C_{is} is the concentration of internal standard in the calibration standard, in $\mu\text{g/ml}$;

a is the slope of the linear function;

b is the ordinate intercept of the calibration curve; the units depend on the evaluation.

Calculate the concentration of each phthalate in the sample (C_{ss} , in $\mu\text{g/ml}$) using [Formula \(2\)](#).

$$C_{ss} = \frac{C_{is}}{a} \times \left[\left(\frac{A_s}{A_{si}} \right) b \right] \quad (2)$$

9.2 Determination of the phthalates content

9.2.1 For each phthalate

From the calibration graph, determine the content of each phthalate, corrected for the internal standard peak area, and interpolate the concentration of the phthalate in $\mu\text{g/ml}$, correcting for any dilutions. Subtract the blank concentration (see [8.1.3](#)) from the specimen concentration.

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The content of phthalate is calculated according to [Formula \(3\)](#) as a percentage, P .

$$P = \frac{V \times (C_{ss} - C_b)}{m \times 10\,000} \quad (3)$$

Alternatively, it can be expressed as a mass fraction, W , in mg/kg using [Formula \(4\)](#).

$$W = \frac{V}{m} \times (C_{ss} - C_b) \quad (4)$$

where

V is the final volume as given in [8.1.1](#) or [8.1.2 d\)](#), in ml;

m is the specimen mass as given in [8.1.1](#) or [8.1.2 a\)](#), in g;

C_b is the concentration of the individual phthalate of blank solution, in $\mu\text{g/ml}$;

C_{ss} is the concentration of the individual phthalate of the specimen solution, corrected for any dilutions, in $\mu\text{g/ml}$, determined by [Formula \(2\)](#).

9.2.2 When a sum of phthalates is requested

In certain cases, a final requested result can be expressed as a sum of different phthalates.

All the phthalates included in the sum shall be clearly identified.

The results of the identified phthalates (as obtained in [9.2.1](#)) are added to give the result of the sum. If the result for a single phthalate is lower than the limit of quantification of the test method (see [9.3](#)), this result is considered as zero and is not included in the sum.

9.3 Performance of the test method

The results of the interlaboratory test is given in [Annex D](#).

This method is able to quantify Phthalates listed in [Table A.1](#) with a limit of quantification (LOQ) of

- 50 mg/kg with toluene extraction ([8.1.1](#))
- 100 mg/kg with THF extraction ([8.1.2](#))

NOTE For complex matrix (for example, leather, rubber, materials with high amount of paraffins), these limits of quantification might be difficult to achieve. That is possible for phthalates that yield a single peak. If a phthalate yields several peaks, it will be difficult to achieve this LOQ.

10 Test report

The test report shall include at least the following:

- a) all the details necessary for complete identification of the tested sample;
- b) a reference to this document, i.e. ISO 16181-1:2021;
- c) the method used (solvent of extraction);
- d) the amount determined for each phthalate that was requested to be tested in mg/kg or in percentage by mass of each listed phthalate in the tested material;
- e) any deviation by agreement or otherwise from the procedure specified;
- f) any unusual features observed;

g) the date of the test.

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Annex A (informative)

Information on some identified phthalates

Table A.1 — List of phthalates that can be determined by this document

No.	Substance ^a	Abbreviation	CAS RN®
1	Dibutyl phthalate	DBP	84-74-2
2	Benzyl butyl phthalate	BBP	85-68-7
3	Bis (2-ethyl(hexyl)phthalate)	DEHP	117-81-7
4	Di-n-octyl phthalate	DNOP	117-84-0
5	Diisononyl phthalate	DINP	28553-12-0 68515-48-0
6	Diisodecyl phthalate	DIDP	26761-40-0 89-16-7 68515-49-1
7	Diisobutyl phthalate	DIBP	84-69-5
8	Bis(2-methoxyethyl) phthalate	DMEP	117-82-8
9	Diisopentyl phthalate	DIPP	605-50-5
10	N-pentyl-isopentyl phthalate	PIPP	776297-69-9
11	Di-n-pentyl phthalate	DNPP	131-18-0
12	Diisohexyl phthalate	DIHxP	71850-09-4
13	Di-n-hexyl phthalate	DNHP	84-75-3
14	Butyl octyl phthalate (*)	BOP	84-78-6
15	1,2-Benzenedicarboxylic acid, di-C6-8-branched alkyl esters, C7-rich	DIHP	71888-89-6
16	Diisooctyl phthalate (*)	DIOP	27554-26-3
17	Diundecyl phthalate (*)	DUP	3648-20-2
18	1,2-Benzenedicarboxylic acid, dipentylester, branched and linear	DPP	84777-06-0
19	1,2-Benzenedicarboxylic acid, dihexyl ester, branched and linear	DHP	68515-50-4
20	1,2-Benzenedicarboxylic acid, di-C7-11-branched and linear alkyl esters	DHNUP	68515-42-4
21	1,2-benzenedicarboxylic acid, di-C6-10-alkyl esters; 1,2-benzenedicarboxylic acid, mixed decyl and hexyl and octyl diesters with \geq 0.3% of dihexyl phthalate	—	68515-51-5 68648-93-1
22	Diethylphthalate (*)	DEP	84-66-2
23	Dimethylphthalate (*)	DMP	131-11-3
24	Dicyclohexylphthalate	DCHP	84-61-7
25	Di-n-propyl phthalate (*)	DPRP	131-16-8
26	Dinonyl phthalate (*)	DNP	84-76-4

^a See ISO/TR 16178 and CEN/TR 16417 for detailed information.

Annex B (informative)

Information on some identified phthalates

B.1 DPP

DPP is a group of branched and linear dipentyl phthalates. It includes DIPP, PIPP and DNPP. DPP should only be reported as the sum of these phthalates when three peaks of DIPP, PIPP and DNPP co-exist in the same sample. Otherwise, report DIPP, PIPP and DNPP individually.

B.2 DHP

DHP is a mixture of phthalate containing DNHP and DIHxP giving multiple peaks in GC-MS chromatogram. If a single peak is found, identify the analyte as DNHP or DIHxP and quantify the sample using routine methodology for DNHP or DIHxP. If multiple peaks are found, identify the analyte as DHP. DHP is reported by semi-quantification with DNHP. Semi-quantify the result of DHP using a calibration curve prepared by DNHP.

B.3 DHNUP

DHNUP is a group of phthalates containing three main individual phthalates (BOP, DNOP and DUP). DNOP and DUP are found to be the major peaks of DHNUP, therefore these are used as markers for easy identification. If a sample contains both DNOP and DUP, then it defines as DHNUP. If the sample also contains BOP, this should be included for reporting (see interpretation in [Table B.1](#)).

Table B.1 — Interpretation for DHNUP

Example	Concentration of phthalates µg			Interpretation for DHNUP
	BOP	DNOP	DUP	
Sample 1	2	2	2	6 µg of possible DHNUP (if both DNOP and DUP are present, then add BOP)
Sample 2	2	1	2	5 µg of possible DHNUP (if both DNOP and DUP are present, then add BOP)
Sample 3	1	0	5	No DHNUP since no DNOP found so report BOP and DUP individually
Sample 4	0	1	5	6 µg of possible DHNUP (both DNOP and DUP are present)

B.4 1,2-benzenedicarboxylic acid, di-C6-10-alkyl esters; 1,2-benzenedicarboxylic acid, mixed decyl and hexyl and octyl diesters with ≥0,3 % of dihexyl phthalate

1,2-benzenedicarboxylic acid, di-C6-10-alkyl esters; 1,2-benzenedicarboxylic acid, mixed decyl and hexyl and octyl diesters with ≥0,3 % of dihexyl phthalate (DNHP) is a group of phthalates composed of C6-C10 alkyl esters. Dihexyl phthalate(C6), 1,2-Benzenedicarboxylic acid, di-C6-8-branched alkyl esters, C7-rich(C7), diisooctyl phthalate(C8), diisononyl phthalate(C9) and diisodecyl phthalate(C10) are used for identification. If all identifiers are found in the sample and the amount of dihexyl phthalate contributes ≥0,3 % to the total content of all identifiers, report the result of 1,2-benzenedicarboxylic

acid, di-C6-10-alkyl esters; 1,2-benzenedicarboxylic acid, mixed decyl and hexyl and octyl diesters with $\geq 0,3$ % of dihexyl phthalate (DNHP) as the sum of concentration of all identifiers. If any one of the identifiers is not detected or the amount of dihexyl phthalate contributes $< 0,3$ % to the total content of all identifiers, report individually (see interpretation in [Table B.2](#)).

Table B.2 — Interpretation for [Clause B.4](#)

Example	Concentration of phthalates μg					Interpretation for 1,2-Benzenedicarboxylic acid, di-C6-10-alkyl esters; 1,2-benzenedicarboxylic acid, mixed decyl and hexyl and octyl diesters with $\geq 0,3$ % of dihexyl phthalate
	DHP (C6)	DIHP (C7)	DIOP (C8)	DINP (C9)	DIDP (C10)	
Sample 1	4	8	17	22	3	Report 54 μg for this phthalate Contribution of DHP = 7,4 %, i.e. $\geq 0,3$ %
Sample 2	2	16	21	34	19	Report 92 μg for this phthalate Contribution of DHP = 2,2 %, i.e. $\geq 0,3$ %
Sample 3	1	312	188	256	144	Report DHP, DIHP, DIOP, DINP & DIDP individually Contribution of DHP = 0,1 %, i.e. $< 0,3$ %
Sample 4	1	20	55	27	0	Report DHP, DIHP, DIOP and DINP individually DIDP is not found in the sample

Annex C (informative)

Suitable gas chromatography-mass spectrometry (GC-MS) apparatus and detection limits for determination of phthalates

The following equipment, column and operating conditions have been found suitable:

Equipment: gas chromatograph (GC) with mass-selective device (MS), quadrupole.

Column: 5 % phenyl methyl siloxane for MS; length 30 m; 0,25 mm interior diameter and 0,25 µm film thickness.

Carrier gas: helium.

Flow rate: 1,5 ml/min.

Injector temperature: 320 °C, mode splitless or split.

Injection volume: 1,0 µl.

Temperature programme: 150 °C for 1 min.
Up to 320 °C at 20 °C/min.
Isothermal 5 min.

Transfer line: 320 °C.

MS mode: Electron impact.

Typical quantification ions for phthalates are shown in [Table C.1](#).

Table C.1 — Typical quantification ions for phthalates

Phthalates	Target ion	Q1	Q2
Bis (2-Ethylhexyl) phthalates-3,4,5,6-D4	283	153	
Dibutyl phthalate (DBP)	149	223	205
Benzyl butyl phthalate (BBP)	149	206	238
Bis (2-ethyl(hexyl)phthalate) (DEHP)	149	167	279
Di-n-octyl phthalate (DNOP)	149	279	261
Diisononyl phthalate (DINP)	293	149	127
Diisodecyl phthalate (DIDP)	307	149	141
Diisobutyl phthalate (DIBP)	149	223	205
Bis(2-methoxyethyl) phthalate (DMEP)	59	149	104
Diisopentyl phthalate (DIPP)	71	43	219
N-pentyl-isopentyl phthalate (PIPP)	71	237	219
Di-n-pentyl phthalate (DNPP)	150	237	219
Diisohexyl phthalate (DIHxP)	85	251	43
Di-n-hexyl phthalate (DNHP)	43	251	41
Butyl octyl phthalate (BOP)	223	41	43

Table C.1 (continued)

Phthalates	Target ion	Q1	Q2
1,2-Benzenedicarboxylic acid, di-C6-8-branched alkyl esters, C7-rich (DIHP)	99	265	—
Diisooctyl phthalate (DIOP)	279	149	167
Diundecyl phthalate (DUP)	321	43	57
1,2-Benzenedicarboxylic acid, dipentylester, branched and linear (DPP)	—	—	—
1,2-Benzenedicarboxylic acid, di-C7-11-branched and linear alkyl esters (DHNUP)	—	—	—
1,2-Benzenedicarboxylic acid, dihexyl ester, branched and linear (DHP)	—	—	—
1,2-benzenedicarboxylic acid, di-C6-10-alkyl esters; 1,2-benzenedicarboxylic acid, mixed decyl and hexyl and octyl diesters with $\geq 0,3$ % of dihexyl phthalate (EC No. 201-559-5)	—	—	—
Diethyl phthalate (*)	177	149	105
Dimethyl phthalate (*)	163	164	
Dicyclohexyl phthalate	249	149	
Di-n-propyl phthalate (*)	209	149	191
Dinonyl phthalate (*)	293	149	167

Depending on the type of equipment used, the appropriate operating conditions might need to be established.

Annex D (informative)

Results of the interlaboratories tests – Method using toluene extraction (8.1.1)

D.1 Sample 1 — PVC

	lab1	lab2	lab3	lab4	lab5	av	sd	100 sd ---- av	z-score				
								lab1	lab2	lab3	lab4	lab5	
DnBP	1 160	1 176	963	1 101	1 090	1 098	84	8 %	0,74	0,93	-1,61	0,04	-0,09
BBP	1 045	949	866	888	971	944	71	8 %	1,43	0,07	-1,10	-0,79	0,38
DEHP	944		819	911	867	885	54	6 %	1,08		-1,23	0,47	-0,33
DNOP	953,5	1 025	788	1 011	1 120	979	122	13 %	-0,21	0,37	-1,56	0,26	1,15
DINP	778		836	1 039	1 030	921	133	14 %	-1,07		-0,64	0,88	0,82
DIDP	936		854	1 050	754	899	126	14 %	0,30		-0,35	1,21	-1,15
DIBP	983		881	894	902	915	46	5 %	1,47		-0,74	-0,46	-0,28
DNPP	846,5		934		1 101	960	129	13 %	-0,88		-0,21		1,09
DNHP	1 095		802	831		909	162	18 %	1,15		-0,67	-0,48	-5,63
DCHP	929	991	846			922	73	8 %	0,10	0,95	-1,05		
								11 %					

D.2 Sample 2 — Textile

	lab1	lab2	lab3	lab4	lab5	av	SD	100 sd --- av	z-score				
								lab1	lab2	lab3	lab4	lab5	
DnBP	515	501	508	496	531	510	14	3 %	0,32	-0,69	-0,18	-1,00	0,32
BBP	448	501	474	506	523	491	30	6 %	-1,44	0,35	-0,55	0,54	-1,44
DEHP	552	678	615	611	588	609	46	8 %	-1,24	1,50	0,13	0,05	-1,24
DNOP	462	439	451	518	613	497	72	14 %	-0,48	-0,80	-0,64	0,30	-0,48
DINP	429	514	472	576	640	526	84	16 %	-1,16	-0,14	-0,65	0,60	-1,16
DIDP	618	523	570	764	683	632	95	15 %	-0,15	-1,14	-0,65	1,40	-0,15
								10 %					

D.3 Sample 3 — PP high

	lab1	lab2	lab3	lab4	lab5	av	SD	100 sd av	z-score				
									lab1	lab2	lab3	lab4	lab5
DnBP	938	896	794	911	1 004	908	76	8 %	0,39	-0,17	-1,50	0,03	0,39
BBP	903		775	926	1 030	909	105	12 %	-0,05		-1,27	0,16	-0,05
DEHP	930	718		916	999	891	120	14 %	0,33	-1,43		0,21	0,33
DNOP	966	876	731	961	1 226	952	180	19 %	0,08	-0,42	-1,23	0,05	0,08
DINP	1 048	1 142	856	983	1 274	1 061	158	15 %	-0,08	0,51	-1,29	-0,49	-0,08
DIDP	911	862	824	885	836	864	36	4 %	1,32	-0,03	-1,12	0,62	1,32
DIBP	930		865	899	1 015	927	64	7 %	0,05		-0,97	-0,44	0,05
DMEP	901		800	935		879	71	8 %	0,32		-1,12	0,80	
DNPP	960		863	973	939	934	49	5 %	0,54		-1,44	0,80	0,54
DNHP	929			927		928	1	0 %	0,71			-0,71	
DIHP	1 034			977		1006	41	4 %					
DEP	891		840	944		891	52	6 %	-0,01		-0,99	1,01	
DCHP													
								8 %					

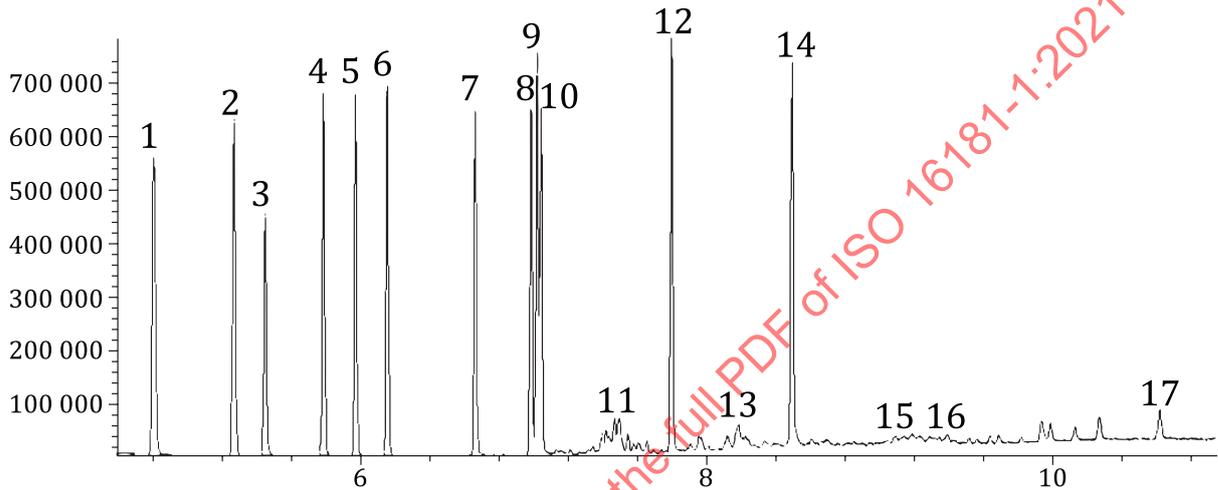
D.4 Sample 4 — PP high

	lab1	lab2	lab3	lab4	lab5	av	SD	100 sd av	z-score				
									lab1	lab2	lab3	lab4	lab5
DnBP	103	73	89	101		91	13	15 %	0,84	-1,34	-0,19	0,69	
BBP	95		86	95	88	91	4	5 %	0,81		-1,11	0,88	0,81
DEHP	96	97		95	80	92	8	9 %	0,43	0,66		0,40	0,43
DNOP	99		80	91	125	99	19	19 %	0,03		-0,98	-0,41	0,03
DINP	111	81	92	102	126	102	17	17 %	0,48	-1,23	-0,61	0,00	0,48
DIDP	93		87	91	100	93	5	6 %	0,01		-1,02	-0,34	0,01
DIBP	101		95	93		96	4	4 %	1,13		-0,35	-0,78	
DMEP	104		91	104		100	8	8 %	0,61		-1,15	0,55	
DNPP	99		95	98	98	98	2	2 %	0,73		-1,47	0,28	0,73
DNHP	102		81	97		93	11	11 %	0,77		-1,13	0,36	
DIHP	106			101		104	3	3 %					
DEP	96		93	95		95	2	2 %	0,74		-1,14	0,40	
DCHP													
								8 %					

Annex E (informative)

Example of a chromatogram for selected phthalates

The chromatograms for phthalates in [Figure E.1](#) were obtained by GC-MS analysis using the parameters described in [Annex C](#).



Key

- 1 Diisobutyl phthalate
- 2 Dibutyl phthalate
- 3 Bis(2-methoxyethyl) phthalate
- 4 Diisopentyl phthalate
- 5 N-pentyl-isopentyl phthalate
- 6 Di-n-pentyl phthalate
- 7 Diisohexyl phthalate
- 8 Di-n-hexyl phthalate
- 9 Butyl octyl phthalate
- 10 Benzyl butyl phthalate
- 11 1,2-Benzenedicarboxylic acid; di-C6-8-branched alkylesters, C7-rich
- 12 Bis(2-ethylhexyl) phthalate
- 13 Diisooctyl phthalate
- 14 Di-n-octyl phthalate
- 15 Diisononyl phthalate
- 16 Diisodecyl phthalate
- 17 Diundecyl phthalate

Figure E.1 — Chromatogram of phthalates