SLOVENSKI STANDARD
SIST EN 16453:2014

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Vlaknine, papir, karton in lepenka - Ugotavljanje ftalatov v ekstraktih papirja, kartona in lepenke

Pulp, paper and paperboard - Determination of phthalates in extracts from paper and paperboard

Zellstoff, Papier und Karton - Bestimmung von Phthalaten in Papier- und Kartonextrakten

Pâtes, papier et carton - Dosage des phtalates dans des extraits de papier et carton

Ta slovenski standard je istoveten z: EN 16453:2014

ICS:

67.250	Materiali in predmeti v stiku z živili	Materials and articles in contact with foodstuffs
85.060	Papir, karton in lepenka	Paper and board

SIST EN 16453:2014 **en,fr,de**

EUROPEAN STANDARD

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English Version

Pulp, paper and paperboard - Determination of phthalates in extracts from paper and paperboard

Pâtes, papier et carton - Dosage des phtalates dans des extraits de papier et carton

Zellstoff, Papier und Karton - Bestimmung von Phthalaten in Papier- und Kartonextrakten

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 16453:2014) has been prepared by Technical Committee CEN/TC 172 “Pulp, paper and board”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2014, and conflicting national standards shall be withdrawn at the latest by July 2014.

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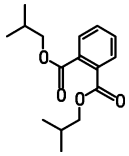
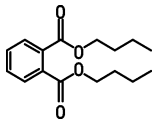
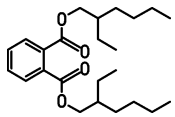
1 Scope

This European Standard specifies an analytical test method for the determination of phthalates in water, solvent and modified polyphenylene oxide (MPPO) extracts of paper and board materials and articles intended for food contact using gas chromatography coupled to mass spectrometry (GC-MS).

This method is applicable to the determination of phthalates in concentration ranging from 0,025 mg/l to 0,5 mg/l for water and solvent extracts and 0,002 mg/dm^2 to 0,040 mg/dm^2 for MPPO migration depending on the individual substance, the specified volume used for analysis and the value of the blank.

Table 1 — Phthlates determined by this method

Name	Abbreviation	Formula	CAS N°	Structure
Diisobutyl-phthalate	DIBP	$C_{16}H_{22}O_4$	84–69–5	
Dibutyl-phthalate	DBP	$C_{16}H_{22}O_4$	84–74–2	
Di-(-ethylhexyl)-phthalate	DEHP	$C_{24}H_{38}O_4$	117–81–7	

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 645, *Paper and board intended to come into contact with foodstuffs - Preparation of a cold water extract*

EN 647, *Paper and board intended to come into contact with foodstuffs - Preparation of a hot water extract*

EN 14338, *Paper and board intended to come into contact with foodstuffs - Conditions for determination of migration from paper and board using modified polyphenylene oxide (MPPO) as a simulant*

EN 15519, *Paper and board intended to come into contact with foodstuffs - Preparation of an organic solvent extract*

EN 27213, *Pulps - Sampling for testing (ISO 7213)*

EN ISO 186, *Paper and board - Sampling to determine average quality (ISO 186)*

EN ISO 536, *Paper and board - Determination of grammage (ISO 536)*

EN ISO 638, *Paper, board and pulps - Determination of dry matter content - Oven-drying method (ISO 638)*

EN ISO 18856, *Water quality - Determination of selected phthalates using gas chromatography/mass spectrometry (ISO 18856)*

3 Principle

3.1 General

Phthalates in the test extract from paper and board materials are determined by gas chromatography / mass spectrometry. The test extract is prepared from water, organic solvent and/or MPPO simulant according to respectively: EN 645, EN 647, EN 15519, EN 14338.

Other phthalates, as listed in Table 1, may also be analysed by this procedure but it is necessary to determine its applicability in each case.

3.2 Interferences

Due to their use as plasticizer agents, phthalates are ubiquitous. Therefore, pay special attention to avoid any contamination. In order to avoid interferences and cross contamination, do not use plastics materials (tubes etc.).

Cross contamination is likely to occur with laboratory air. Therefore, remove as far as possible, plastic materials from the laboratory. Cleaning agents often contain phthalates and may severely contaminate the laboratory air if in use regularly. Therefore, refrain from using these agents during application of this procedure.

Phthalates may bleed from the septa of the injector port into the gas chromatograph, therefore use septa that are not likely to contaminate the system.

Fittings of syringes or equipment and septa of the sampling bottles may as well contain phthalates.

4 Materials

4.1 General

Common laboratory glassware, rinsed with ethyl acetate before use. After rinsing the glassware with solvent, let residual solvent evaporate under a fume hood. In case of contamination, special attention should be paid to the volumetric flasks cleaning. For example the non-volumetric glassware could be cleaned in a furnace at 500°C for at least 6 h.

Glassware for volumetric purpose can change its properties due to the heating process and so should not be treated thus.

Phthalates are ubiquitous laboratory contaminants. Each lot of reagent used for this method should be checked for phthalates contamination.

4.2 Pasteur pipettes

4.3 Double mark glass pipette of 0,05 ml, 0,2 ml, 0,5 ml, 1 ml, 5 ml and 10 ml

4.4 Volumetric flasks of 5 ml, 10 ml, 50 ml and 100 ml

4.5 Screw-cap glass bottle PYREX[1)] of 250 ml with a Polytetrafluorethylene-septa cap

4.6 Graduated cylinder of 200 ml

4.7 Round flask of 50 ml and 250 ml

4.8 Glass funnel

4.9 Polytetrafluorethylene-screw-cap glass vial single use (capacity adapted to the volume of concentrate)

5 Apparatus

5.1 Balance: capable of accurately weighing 0,000 1 g

5.2 Round flask heater

5.3 Rotary vacuum evaporator

5.4 Gas chromatograph coupled to a mass spectrometer (GC-MS)

6 Reagents

6.1 General

Use as far as available reagents of analytical quality or better. Use only reagents with negligibly low concentrations of phthalates and verify by blank determinations.

6.2 Operating gases for gas chromatography/mass spectrometry, of high purity and in accordance with manufacturer's specifications

6.3 Ethyl acetate, $C_4H_8O_2$, CAS N° 141-78-6

6.4 Isooctane, C_8H_{18}, CAS N° 540-84-1

Store the following solutions in glass bottles at −18 °C, protected from light.

6.5 Internal standard stock solution: For example d4-ring-deuterated-dibutyl phthalate (D4-ring-DBP) at 10 g/l

6.5.1 General

Weigh for example, approximately 0,1 g with an accuracy of 0,001 g of d4-DBP in a 10 ml volumetric flask (4.4) and bring to volume with ethyl acetate (6.3).

1) PYREX is an example of a suitable product available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this product.

6.5.2 Solution A of internal standard at 100 mg/l in ethyl acetate

Transfer 1 ml of the solution (6.5.1) into a 100 ml volumetric flask (4.4) and dilute to the mark with the ethyl acetate (6.3).

6.5.3 Solution B of internal standard at 10 mg/l in ethyl acetate

Transfer 0,5 ml of the solution A (6.5.2) into a 5 ml volumetric flask (4.4) and dilute to the mark with the ethyl acetate (6.3).

6.5.4 Solution C of internal standard at 0,5 mg/l in ethyl acetate

Transfer 0,25 ml of the stock solution A (6.5.2) into a 50 ml volumetric flask (4.4) and dilute to the mark with the ethyl acetate (6.3).

6.6 Stock solutions of individual phthalate at 1 000 mg/l in ethyl acetate

6.6.1 General

In a 100 ml volumetric flask (4.4), weigh approximately 0,1 g with an accuracy of 0,001 g of each of the reference substances and bring to volume with ethyl acetate (6.3).

6.6.2 Intermediate solution of phthalates at 50 mg/l in ethyl acetate

Transfer 5 ml of each stock solution (6.6.1) into a 100 ml volumetric flask (4.4) and dilute to the mark with ethyl acetate (6.3).

6.6.3 Calibration standard solutions of phthalates in isooctane

Five standard solutions are prepared by transferring respectively: 0,05 ml, 0,1 ml, 0,2 ml, 0,5 ml and 1 ml of the Intermediate solution (6.6.2) in 10 ml volumetric flasks (4.4) and 0,5 ml of the solution B of internal standard (6.5.3) by adjusting the volume with the solvent used for the extract of paper and board, for the preparation of solution having respectively concentrations of about: 0,25 mg/l, 0,5 mg/l, 1 mg/l, 2,5 mg/l and 5 mg/l of phthalate, and 0,5 mg/l of Internal Standard stock solution in each calibration solution.

6.6.4 Individual solutions of phthalates in ethyl acetate à 1 mg/l for determination of GC retention times

Transfer 0,1 ml of stock solution (6.6.1) into a 100 ml volumetric flask (4.4) and dilute to the mark with ethyl acetate (6.3).

7 Sampling

If the analysis is being made to evaluate a lot of paper, board or pulp, the sample shall be selected in accordance with EN ISO 186 or EN 27213, as relevant. If the analysis is made on another type of sample, report the source of the sample, and, if possible, the sampling procedure. Select the specimens so that they are representative of the sample received.

If required, take a separate sample for the determination of the grammage in accordance with EN ISO 536 and/or for the determination of dry matter content with EN ISO 638.

For any storage or transport between sampling and analysis, protect the sample from contamination by using for example aluminium foil to over-wrap