

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

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 16453:2014 sisaldab Euroopa standardi EN 16453:2014 inglisekeelset teksti.	This Estonian standard EVS-EN 16453:2014 consists of the English text of the European standard EN 16453:2014.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 29.01.2014.	Date of Availability of the European standard is 29.01.2014.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile standardiosakond@evs.ee.

ICS 67.250, 85.060

Standardite reprodutseerimise ja levitamise õigus kuulub Eesti Standardikeskusele

Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonsesse süsteemi või edastamine ükskõik millises vormis või millisel teel ilma Eesti Standardikeskuse kirjaliku loata on keelatud.

Kui Teil on küsimusi standardite autorikaitse kohta, võtke palun ühendust Eesti Standardikeskusega:
Aru 10, 10317 Tallinn, Eesti; www.evs.ee; telefon 605 5050; e-post info@evs.ee

The right to reproduce and distribute standards belongs to the Estonian Centre for Standardisation

No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without a written permission from the Estonian Centre for Standardisation.

If you have any questions about copyright, please contact Estonian Centre for Standardisation:
Aru 10, 10317 Tallinn, Estonia; www.evs.ee; phone 605 5050; e-mail info@evs.ee

ICS 67.250; 85.060

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

This European Standard was approved by CEN on 9 November 2013.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents	Page
Foreword	3
1 Scope	4
2 Normative references	4
3 Principle	5
3.1 General.....	5
3.2 Interferences	5
4 Materials	5
4.1 General.....	5
4.2 Pasteur pipettes	6
4.3 Double mark glass pipette of 0,05 ml, 0,2 ml, 0,5 ml, 1 ml, 5 ml and 10 ml	6
4.4 Volumetric flasks of 5 ml, 10 ml, 50 ml and 100 ml	6
4.5 Screw-cap glass bottle PYREX of 250 ml with a Polytetrafluorethylene-septa cap	6
4.6 Graduated cylinder of 200 ml	6
4.7 Round flask of 50 ml and 250 ml	6
4.8 Glass funnel	6
4.9 Polytetrafluorethylene-screw-cap glass vial single use (capacity adapted to the volume of concentrate)	6
5 Apparatus	6
5.1 Balance: capable of accurately weighing 0,000 1 g	6
5.2 Round flask heater.....	6
5.3 Rotary vacuum evaporator	6
5.4 Gas chromatograph coupled to a mass spectrometer (GC-MS).....	6
6 Reagents	6
6.1 General.....	6
6.2 Operating gases for gas chromatography/mass spectrometry, of high purity and in accordance with manufacturer's specifications.....	6
6.3 Ethyl acetate, C ₄ H ₈ O ₂ , CAS N° 141-78-6	6
6.4 Isooctane, C ₈ H ₁₈ , CAS N° 540-84-1.....	6
6.5 Internal standard stock solution: For example d4-ring-deuterated-dibutyl phthalate (D4-ring-DBP) at 10 g/l.....	6
6.6 Stock solutions of individual phthalate at 1 000 mg/l in ethyl acetate.....	7
7 Sampling	7
8 Preparation of the test sample of water extract	8
9 Preparation of the test sample of the organic solvent extract	8
10 Preparation of the test sample of the MPPO Extract	8
11 Procedure	8
11.1 GC conditions	8
11.2 Calibration	9
11.3 Analysis of test samples	9
12 Expression of results	10
12.1 For organic solvent extract: Expression of results.....	10
12.2 For MPPO extract: Expression of results.....	10
13 Test report	10
14 Precision data	11
Annex A (informative) GC-MS total ion chromatogram of a standard calibration solution of 2 mg/l of phthalates	12

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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

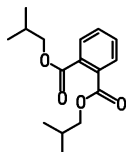
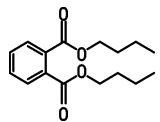
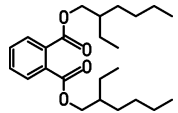
According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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/dm² to 0,040 mg/dm² for MPPO migration depending on the individual substance, the specified volume used for analysis and the value of the blank.

Table 1 — Phthalates 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-(4-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.