

**Rasvased toiduained. Pestitsiidide ja  
polüklorobifenüülide määramine. Osa 4:  
Määratlemine, kontrollkatsed,  
mitmesugust**

Fatty food - Determination of pesticides and  
polychlorinated biphenyls (PCBs) - Part 4:  
Determination, confirmatory tests, miscellaneous

## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

<p>Käesolev Eesti standard EVS-EN 1528-4:2000 sisaldab Euroopa standardi EN 1528-4:1996 ingliskeelset teksti.</p> <p>Käesolev dokument on jõustatud 19.07.2000 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes.</p> <p>Standard on kättesaadav Eesti standardiorganisatsioonist.</p>	<p>This Estonian standard EVS-EN 1528-4:2000 consists of the English text of the European standard EN 1528-4:1996.</p> <p>This document is endorsed on 19.07.2000 with the notification being published in the official publication of the Estonian national standardisation organisation.</p> <p>The standard is available from Estonian standardisation organisation.</p>
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<p><b>Käsitlusala:</b> See EN 1528 osa annab juhtnööre mõnede soovitatavate meetodite kohta pestitsiidide ja polüklorobifenüülide määramiseks rasvastes toiduainetes ja kontrollkatsete kohta ning esitab puhastusprotseduurid enamiku lipiidide eemaldamiseks, kui analüüsitakse suurt hulka rasva.</p>	<p><b>Scope:</b></p>
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**ICS** 67.050

**Võtmesõnad:** gaasikromatograafia, katsed, keemiline analüüs, keemilised jäägid, pestitsiidid, polüklorobifenüül, puhtus, sisalduse määramine, toiduainetooted, toidurasvad

ICS 67.040

Descriptors: Fatty food, pesticides, polychlorinated biphenyls, analysis.

**English version**

**Fatty food**

**Determination of pesticides and polychlorinated biphenyls (PCBs)**

**Part 4: Determination, confirmatory tests, miscellaneous**

Aliments gras – Dosage des pesticides et des polychlorobiphényles (PCB) – Partie 4: Détermination, essais de confirmation, divers

Fetteiche Lebensmittel – Bestimmung von Pestiziden und polychlorierten Biphenylen (PCB) – Teil 4: Verfahren zur Bestimmung und Absicherung, Verschiedenes

This European Standard was approved by CEN on 1996-10-27.

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 Central Secretariat 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 Central Secretariat has the same status as the official versions.

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**CEN**

European Committee for Standardization  
Comité Européen de Normalisation  
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### Foreword

This European Standard has been prepared by Technical Committee CEN/TC 275 "Food analysis, horizontal methods", 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 May 1997, and conflicting national standards shall be withdrawn at the latest by May 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

This European Standard consists of the following Parts:

Part 1 "General" presents the scope of the standard and describes general considerations with regard to reagents, apparatus, gas chromatography etc., applying to each of the analytical methods selected.

Part 2 "Extraction of fat, pesticides and PCBs, and determination of fat content" presents a range of analytical procedures for extracting the fat portion containing the pesticide and PCB residues from different groups of fat-containing foodstuffs.

Part 3 "Clean-up methods" presents the details of methods A to H for the clean-up of fats and oils or the isolated fat portion, respectively, using techniques such as liquid/liquid partition, adsorption or gel permeation column chromatography.

Part 4 "Determination, confirmatory tests, miscellaneous" gives guidance on some recommended techniques for the determination of pesticides and PCBs in fatty foodstuffs and on confirmatory tests and lists a clean-up procedure for the removal of the bulk of lipids when analysing large quantities of fat.

## Introduction

This European Standard comprises a range of multi-residue methods of equal status: no single method can be identified as the prime method because, in this field, methods are continuously developing. The methods selected for inclusion in this standard have been validated and are widely used throughout Europe. Any variation in the methods used should be shown to give comparable results.

## 1 Scope

This Part of EN 1528 gives guidance on some recommended techniques for the determination of pesticides and polychlorinated biphenyls (PCBs) in fatty foodstuffs and on confirmatory tests and lists a clean-up procedure for the removal of the bulk of lipids when analysing large quantities of fat.

## 2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 1528-1 : 1996

Fatty food - Determination of pesticides and polychlorinated biphenyls (PCBs) - Part 1: General

EN 1528-2 : 1996

Fatty food - Determination of pesticides and polychlorinated biphenyls (PCBs) - Part 2: Extraction of fat, pesticides and PCBs, and determination of fat content

EN 1528-3 : 1996

Fatty food - Determination of pesticides and polychlorinated biphenyls (PCBs) - Part 3: Clean-up methods

## 3 General

The methods described in this Part of EN 1528 permit the residues present to be provisionally identified and quantified, by gas chromatographic methods using selective detectors.

All positive results require confirmation of identity and quantity.

The procedures listed for confirmation such as alternative GC columns, alternative GC detectors, thin-layer chromatography (TLC), high performance liquid chromatography (HPLC), column fractionation, derivatization, spectral measurements, etc., are all of value. Results obtained using mass spectrometry (MS) present definitive evidence for confirmation/identification purposes.

## 4 Determination

### 4.1 Gas chromatography

#### 4.1.1 General

A suitable GC system, preferably equipped with separate heaters for injector, detector and column ovens, should be used. Although the choice of the different parts of the GC system is a matter for the experience of the analyst, the following general recommendations are made.

The detectors should be properly adjusted, according to the manufacturers' instructions. Variations in detector sensitivity should be checked periodically by verifying the linearity of the calibration curves using standard solutions of pesticides.

The quantification unit of the gas chromatographic system needs to include an integration system which permits the calculation not only of peak heights but also of peak areas.

It has been found in practice that equivalent results can be achieved despite the adoption of different GC conditions, and different makes of instruments. On the other hand, specifying standard GC parameters does not in any way guarantee that the quality of the results generated will be identical.

For typical GC conditions, see annex B.