# Rasvased toiduained. Pestitsiidide ja polüklorobifenüülide määramine. Osa 3: Puhastamismeetodid

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



# EESTI STANDARDI EESSÕNA

## **NATIONAL FOREWORD**

Käesolev Eesti standard EVS-EN 1528- 3:2000 sisaldab Euroopa standardi EN 1528-3:1996 ingliskeelset teksti.	This Estonian standard EVS-EN 1528-3:2000 consists of the English text of the European standard EN 1528-3:1996.
Käesolev dokument on jõustatud 19.07.2000 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes.	This document is endorsed on 19.07.2000 with the notification being published in the official publication of the Estonian national standardisation organisation.
Standard on kättesaadav Eesti standardiorganisatsioonist.	The standard is available from Estonian standardisation organisation.

## Käsitlusala:

See EN 1528 osa määrab kindlaks meetodid (A kuni H) rasvade ja õlide või eraldatud rasvaportsjonite puhastamiseks, kasutades vastavalt kas vedelikvedelikekstraheerimist, adsorbeerimist või geelkolonnkromatograafiat.

## Scope:

ICS 67.050

**Võtmesõnad:** keemiline analüüs, kromatograafia, pestitsiidid, polüklorobifenüül, puhtus, sisalduse määramine, toiduainetooted, toidurasvad

# EUROPEAN STANDARD NORME EUROPÉENNE EUROPÄISCHE NORM

EN 1528-3

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ICS 67.040

Descriptors: Fatty food, pesticides, polychlorinated biphenyls, clean-up methods.

## **English version**

## Fatty food

Determination of pesticides and polychlorinated biphenyls (PCBs)

Part 3: Clean-up methods

Aliments gras – Dosage des pesticides et des polychlorobiphényles (PCB) – Partie 3: Méthodes de purification

Fettreiche Lebensmittel – Bestimmung von Pestiziden und polychlorierten Biphenylen (PCB) – Teil 3: Reinigungsverfahren

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.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom.

# CEN

European Committee for Standardization Comité Européen de Normalisation Europäisches Komitee für Normung

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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 acceptable results.

The residues to be analysed in this European Standard are associated with the fat portion of the samples. In addition to the residues, the extracts obtained in accordance with EN 1528-2: 1996 or in accordance with the following methods contain material including fats and other lipids, which could interfere in the analysis. To purify the crude extracts or the fats and oils to be analysed, several methods may be used.

This European Standard contains the following clean-up methods that have been subjected to interlaboratory studies and are adopted throughout Europe:

- Method A: Liquid-liquid partitioning with acetonitrile and clean-up on a Florisil® column (AOAC) [1]
- Method B: Liquid-liquid partitioning with dimethylformamide and clean-up on a Florisil® column (Specht) [2]
- Method C: Column chromatography on activated Florisil® (AOAC) [3]
- Method D: Column chromatography on partially deactivated Florisil® (Stijve) [4]
- Method E: Column chromatography on partially deactivated aluminium oxide (Greve & Grevenstuk) [5]
- Method F: Gel permeation chromatography (GPC) (AOAC) [6]
- Method G: Gel permeation chromatography (GPC) and column chromatography on partially deactivated silica gel (Specht) [7]
- Method H: High performance gel permeation chromatography (HPGPC) (MAFF) [8]

## 1 Scope

This Part of EN 1528 specifies 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. The applicable usage of the methods A to H is given in detail in each method described.

NOTE: See also EN 1528-4 which 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 1528-4: 1996

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

## 3 Principle

Removal of interfering materials from the sample extract to obtain a solution of the extracted residue in a solvent which is suitable for quantitative examination by the selected method of determination.