This is a free page sample. Access the full version online.

NSAI Standards

Irish Standard
I.S. EN 16619:2015

# Food analysis - Determination of benzo[a]pyrene, benz[a]anthracene, chrysene and benzo[b]fluoranthene in foodstuffs by gas chromatography mass spectrometry (GC-MS)

© CEN 2015 No copying without NSAI permission except as permitted by copyright law.

This is a free page sample. Access the full version online.

**I.S. EN 16619:2015**

*Incorporating amendments/corrigenda/National Annexes issued since publication:*

The National Standards Authority of Ireland (NSAI) produces the following categories of formal documents:

I.S. xxx: Irish Standard — national specification based on the consensus of an expert panel and subject to public consultation.

S.R. xxx: Standard Recommendation — recommendation based on the consensus of an expert panel and subject to public consultation.

SWiFT xxx: A rapidly developed recommendatory document based on the consensus of the participants of an NSAI workshop.

*This document replaces/revises/consolidates the NSAI adoption of the document(s) indicated on the CEN/CENELEC cover/Foreword and the following National document(s):*

*NOTE: The date of any NSAI previous adoption may not match the date of its original CEN/CENELEC document.*

| *This document is based on:* | *Published:* |
|---|---|
| EN 16619:2015 | 2015-04-22 |

| *This document was published* under the authority of the NSAI and comes into effect on: | ICS number: |
|---|---|
| 2015-05-09 | 67.050 |
| | NOTE: If blank see CEN/CENELEC cover page |


| | | |
|---|---|---|
| NSAI | T +353 1 807 3800 | Sales: |
| 1 Swift Square, | F +353 1 807 3838 | T +353 1 857 6730 |
| Northwood, Santry | E standards@nsai.ie | F +353 1 857 6729 |
| Dublin 9 | W NSAI.ie | W standards.ie |

Údarás um Chaighdeáin Náisiúnta na hÉireann

This is a free page sample. Access the full version online.


EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

# EN 16619

April 2015

ICS 67.050

English Version

# Food analysis - Determination of benzo[a]pyrene, benz[a]anthracene, chrysene and benzo[b]fluoranthene in foodstuffs by gas chromatography mass spectrometry (GC-MS)

Analyse des produits alimentaires - Dosage du benzo(a)pyrène, benzo(a)anthracène, chrysène et benzo(b)fluoranthène dans les denrées alimentaires par chromatographie en phase gazeuse couplée à la spectrométrie de masse (CG-SM)

Lebensmittelanalytik - Bestimmung von Benzo[a]pyren, Benz[a]anthracen, Chrysen und Benzo[b]fluoranthen in Lebensmitteln mit Gaschromatographie und Massenspektrometrie (GC-MS)

This European Standard was approved by CEN on 7 February 2015.

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**

© 2015 CEN All rights of exploitation in any form and by any means reserved worldwide for CEN national Members.

This is a free page sample. Access the full version online.


# Contents

Page

**Foreword** ..... 3
**1 Scope** ..... 4
**2 Normative references** ..... 4
**3 Principle** ..... 4
**4 Reagents** ..... 5
**5 Standard preparation** ..... 9
**6 Apparatus** ..... 12
**7 Procedure** ..... 15
**8 GC-MS analysis** ..... 17
**9 Calculation and reporting** ..... 22
**10 Quality control** ..... 22
**11 Precision data** ..... 24
**12 Test report** ..... 27
**Annex A** (informative) **Typical chromatograms** ..... 28
**Annex B** (informative) **Precision data** ..... 30
**Annex C** (informative) **Precision data from single laboratory validation** ..... 35
**Bibliography** ..... 37

This is a free page sample. Access the full version online.


# Foreword

This document (EN 16619:2015) 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 October 2015 and conflicting national standards shall be withdrawn at the latest by October 2015.

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.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

**WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.**

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.

This is a free page sample. Access the full version online.


# 1 Scope

This European Standard specifies a method for the determination of 4 of the 16 EU priority polycyclic aromatic hydrocarbons (PAHs), identified as target PAHs. They are benz[*a*]anthracene (BaA), benzo[*a*]pyrene (BaP), benzo[*b*]fluoranthene (BbF) and chrysene (CHR). The method allows their quantification in the presence of the other 12 EU priority PAHs (benzo[*j*]fluoranthene (BjF), cyclopenta[*cd*]pyrene (CPP), benzo[*k*]fluoranthene (BkF), dibenz[*a,h*]anthracene (DhA), benzo[*c*]fluorene (BcL), dibenzo[*a,e*]pyrene (DeP), benzo[*ghi*]perylene (BgP), dibenzo[*a,h*]pyrene (DhP), dibenzo[*a,i*]pyrene (DiP), dibenzo[*a,l*]pyrene (DlP), indeno[1,2,3-*cd*]pyrene (IcP), 5-methylchrysene (5MC)) in extruded wheat flour, smoked fish, dry infant formula, sausage meat, freeze-dried mussels, edible oil and wheat flour, by gas-chromatography mass-spectrometry (GC-MS). The extraction of PAHs from solid samples is performed by pressurized liquid extraction (PLE). Soxhlet extraction was applied by some participants in the method validation study by collaborative trial as alternative to PLE. The sample cleanup is performed by applying the following techniques in the reported sequence: size exclusion chromatography (SEC), and solid phase extraction (SPE).

This method complies with the performance characteristics specified in Commission Regulation (EU) No 836/2011 (see [1]). In particular the specifications for the limit of detection (LOD) and of the limit of quantification (LOQ) (0,30 µg/kg and 0,90 µg/kg respectively) were met.

The method has been validated in an interlaboratory study via the analysis of both naturally contaminated and spiked samples, ranging from 0,5 µg/kg to 11,9 µg/kg. However, linearity of the instrument response was proven for the concentration range 0,5 µg/kg to 20 µg/kg.

For the determination of PAHs in edible fats and oils, two other standards are also available, EN ISO 22959 and EN ISO 15753, for more information see [2] and [3].

# 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 ISO 1042:1999, *Laboratory glassware - One-mark volumetric flasks (ISO 1042:1998)*

EN ISO 3696:1995, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

# 3 Principle

The sample is homogenized. A test portion is mixed with desiccant, sand and the stable isotope labelled internal standard solution. It is then extracted with *n*-hexane or cyclohexane by pressurized liquid extraction, or alternatively by Soxhlet extraction. If applicable, co-extracted water is separated from the organic phase of the extract. The organic extract is evaporated to a small volume, filtered and purified by SEC, using a mixture of ethyl acetate and cyclohexane as eluent.

After SEC, 200 µl of toluene are added as a keeper to the collected SEC fraction. The SEC fraction is evaporated to about 200 µl, and cleaned up by SPE on silica, using cyclohexane as eluent. The cleaned up sample extract is evaporated again to about 200 µl. Finally, an injection standard solution is added to the sample prior to measurement by GC-MS.

The injection is performed with a PTV, or split/splitless injection port. The chromatographic separation is obtained on a mid-polar capillary column with high selectivity for PAHs. The analytes are ionised by electron ionization (EI) at 70 eV. The target PAHs are recorded in Single Ion Monitoring (SIM) mode, and quantified by comparison with the stable isotope labelled analogues.

SAI GLOBAL

This is a free preview.  Purchase the entire publication at the link below:

Product Page

Looking for additional Standards? Visit Intertek Inform Infostore

Learn about LexConnect, All Jurisdictions, Standards referenced in Australian legislation