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

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NATIONAL FOREWORD

See Eesti standard EVS-EN 16619:2015 sisaldab Euroopa standardi EN 16619:2015 ingliskeelset teksti.	This Estonian standard EVS-EN 16619:2015 consists of the English text of the European standard EN 16619:2015.
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 22.04.2015.	Date of Availability of the European standard is 22.04.2015.
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.

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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.

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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of an identical text or by endorsement, at the latest by October 2015 and conflicting national standards shall be withdrawn at the latest by October 2015.

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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.