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Food analysis - Determination of benzo[a]pyrene, benz[a]anthracene, chrysene and benzo[b]fluoranthene in foodstuffs by high performance liquid chromatography with fluorescence detection (HPLC-FD)

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 liquide à haute performance avec détection de fluorescence (HPLC-FD)

Lebensmittelanalytik - Bestimmung von Benzo[a]pyren, Benz[a]anthracen, Chrysen und Benzo[b]fluoranthen in Lebensmitteln mittels Hochleistungs-Flüssigkeitschromatographie mit Fluoreszenzdetektion (HPLC-FD)

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Foreword

This document (CEN/TS 16621:2014) has been prepared by Technical Committee CEN/TC 275 "Food analysis - Horizontal methods", the secretariat of which is held by DIN.

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1 Scope

This Technical Specification specifies a method for the determination of benzo[*a*]pyrene (BaP) plus benz[*a*]anthracene (BaA), benzo[*b*]fluoranthene (BbF) and chrysene (CHR) in several food matrices. The method is based on size exclusion chromatography (SEC) cleanup, followed by quantification with high performance liquid chromatography (HPLC) with programmable fluorescence detection. This method has been in-house validated via the analysis of spiked samples of edible olive oil, fresh mussels, smoked fish, smoked meat products, processed cereal-based foods for young children, infant formulae, chocolate and food supplements (isoflavones) at levels ranging from 0,25 μ g/kg to 1,00 μ g/kg and from 4,95 μ g/kg to 23,53 μ g/kg, depending on the Polycyclic Aromatic Hydrocarbon (PAH) or the matrix. This method complies with the performance characteristics specified for BaP, BaA, BbF and CHR in current legislation [3].

The method has been shown to be applicable to a variety of additional matrices as meat products, fresh fish, paprika, roasted coffee, bread, herbs, breakfast cereals, beer, sunflower oil, olives and fried tomato, with a limit of quantification below 0,5 µg/kg.

In addition, the method was tested in-house and shown to be applicable also for the quantification of the other 12 PAHs of the 15+1 EU priority PAHs set (benzo[c]fluorene (BcL), benzo[/]fluoranthene (BjF), benzo[k]fluoranthene (BkF), cyclopenta[cd]pyrene (CPP), dibenz[a,h]anthracene (DhA), dibenzo[a,e]pyrene (DeP), benzo[ghi]perylene (BgP), dibenzo[a,h]pyrene (DhP), dibenzo[a,i]pyrene (DiP), dibenzo[a,/]pyrene (DIP), indeno[1,2,3-cd]pyrene (IcP), 5-methylchrysene (5MC)) in all matrices listed above and at similar level ranges, except for CPP, where a UV detection had to be used with limits of quantification above 8 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 (see [1] and [2]).

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

3 Principle

The PAHs are extracted from solid matrices with dichloromethane. In case of edible oils, the samples are simply dispersed in dichloromethane. Aliquots of crude extracts in dichloromethane are purified by SEC. The final extracts are analysed by HPLC under gradient conditions with programmable fluorescence detection.

4 Reagents

Use only reagents of recognized analytical grade and water complying with grade 1 of EN ISO 3696:1995, unless otherwise specified. Solvents shall be of quality for HPLC analysis. For storing and expiring dates of use of substances and commercially available solutions, supplier indications or certificates shall be followed. Refrigerated standard solutions shall reach room temperature before being used.

WARNING 1 — Some PAHs are considered carcinogenic. Persons using this document should be familiar with normal laboratory practices. It is the responsibility of the user of this document to apply practices which are in agreement with applicable occupational safety and health practices.

WARNING 2 — Dispose chemical waste according to applicable environmental rules and regulations.