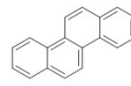


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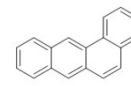
INTRODUCTION

Heterogenous PAH-like compounds interfere PAH analysis in often colourfull food supplements.

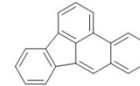
Development of a less tedious method than gelpermeation (CEN /TS 16619:2015) was therefore challenging. We describe results of a collaborative study of a fast two times SPE clean up before PAH4 analysis.



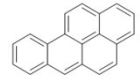
Chrysen



Benz[a]anthracene



Benzo[b]fluoranthene



Benzo[a]pyrene

METHOD



1 g ± 0.05g



Internal D-labelled standards
20 ml Ethylacetate:Cyclohexane (1:1)



1. Supelclean TM EZ-POP NP (2.5g Supelco)
2. SupelMIP SPE-PAH (50mg, Supelco)
Eluent: Cyclohexane



9 Laboratories
GC-MS or GC-MS/MS

4 Laboratories
LC-FLD

COLLABORATIVE STUDY PERFORMANCE RESULTS

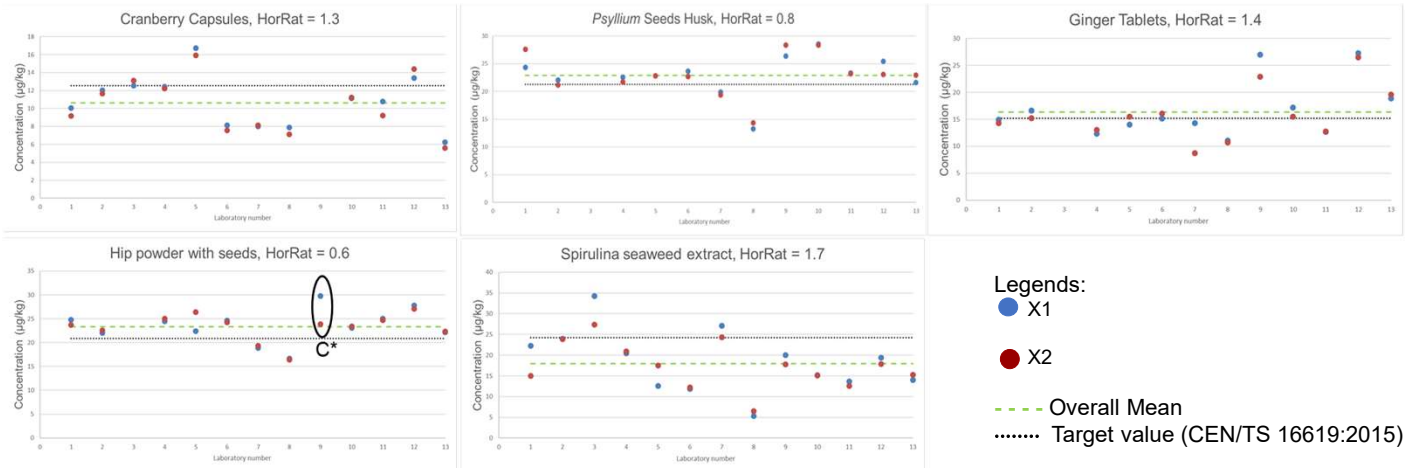
Homogeneity Test (N = 10 duplicate):

Compound	Cranberry capsules	Psyllium Seeds Husk	Ginger Tablets	Hip powder with seeds	Spirulina seaweed extract
Chrysene	Pass	Pass	Pass	Pass	Pass
Benz[a]anthracene	Pass	Pass	Pass	Pass	Pass
Benzo[b]fluoranthene	Pass	Pass	Pass	Pass	Pass
Benzo[a]pyrene	Pass	Pass	Pass	Pass	Pass

Statistical evaluation according to the international harmonized protocol for proficiency testing and ISO 13582:2015. True value based on CEN/TS 16619:2015.

Details in Wilde and Duedahl-Olesen, 2022.

Sum PAH4 compliant results from participating laboratories:



Data from Wilde and Duedahl-Olesen 2022 with Cochran's (C) and Grubbs' (G) stragglers (95% level of confidence) included (C* for hip). Outliers (99% level of confidence) excluded. One-way ANOVA for between-laboratory variability (ISO 5725-2:2019) and Thompson modified Horwitz equation of Horwitz ratio, HorRat = RSD/22% (EU Regulation 333/2007).

CONCLUSIONS

- ✓ Between laboratory variability < 22%
- ✓ Between laboratory HorRat < 2
- ✓ Time saving compared to CEN 16619:2015
- ✓ Detection of PAH4 below ML in food supplements

Parts of the current study was performed within the framework of the European Union Reference Laboratory (EURL) for processing contaminants financed by the European Commission. Authors would like to thank all participating laboratories for their voluntary contributions.

References:

- Wilde, A.S, Duedahl-Olesen, L. (2022) Analysis of 4 polycyclic aromatic hydrocarbons in plant based food supplements – results from a collaborative trial. Food Additives and Contaminants Part A: 39:1380-1389.
- CEN/TS 16619:2015. Determination of benzo[a]pyrene, benz[a]anthracene, chrysene and benzo[b]fluoranthene in foodstuffs by gas chromatography mass spectrometry (GC-MS).
- European Commission 2007. Commission Regulation (EC 333/2007 of 28 March 2007 Laying down the methods of sampling and analysis for the Official Control of the levels of lead, Cadmium, Mercury, Inorganic Tin, 3-MCPD and Benzo[a]pyrene in foodstuffs. Off. J. Eur. Union L88: 29-38.
- ISO 5725-2:2019. Accuracy (Trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.
- ISO 13528:2015 Statistical method for use in proficiency testing by interlaboratory comparison.

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