

WP-CBR001: a new CRM for the determination of PAHs and toxic elements



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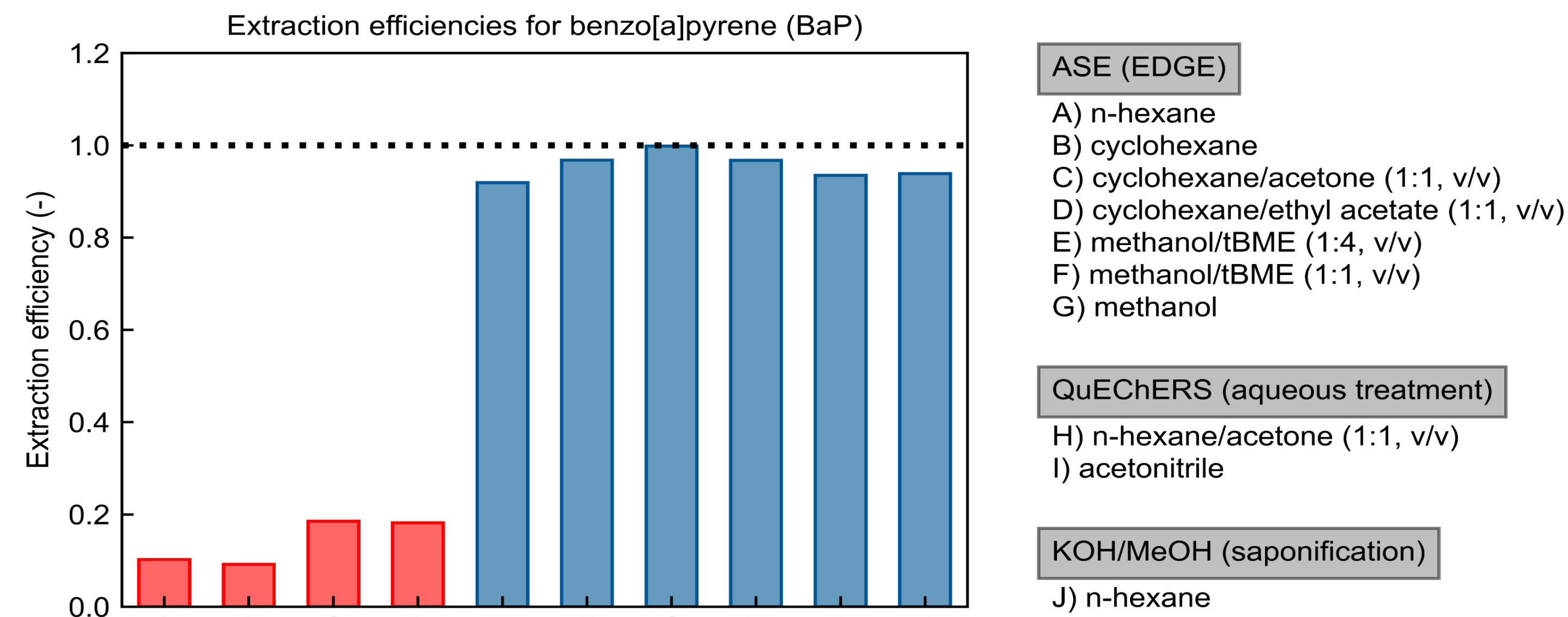
The analysis of food contaminants is often very challenging, due to the complexity of the matrix and the presence of the analytes in trace and ultra-trace amounts. Certified reference materials (CRMs) are required by laboratories to validate analytical methods, improve measurement accuracy and assess measurement uncertainty, among other things. The development and production of CRMs in food matrices, according to ISO 17034 [1], require significant efforts in terms of production processes, number of studies and measurements.

WP-CBR001 is a whey protein CRM developed for the determination of PAHs (BaA, BaP, BbF and Chr) and toxic elements (As, Cd, Hg and Pb) in a protein rich food matrix. For the production of this CRM, a whey with 80% of protein, often used in food industry, was chosen as matrix. Liquid whey was spiked with the above-mentioned contaminants and spray dried in an industrial pilot plant. The contaminants are completely integrated into the whey protein matrix and consequently the final material is very close to a real contaminated food matrix rich in protein. The stability and the homogeneity of PAHs and toxic elements in the CRM were assessed through systematic studies. The assignment of the certified mass fractions for toxic elements and PAHs in WP-CBR001 is based on an in-house study at METAS. The mass fractions of As and Hg were determined by standard addition methods in combination with ICP-MS and direct mercury analysis, respectively. The mass fractions of Cd and Pb were determined by double isotope dilution (ID) ICP-MS and the mass fractions of the PAHs by isotope dilution (ID) GC-MS/MS.

Polycyclic aromatic hydrocarbons (PAHs)

The choice of solvent is of crucial importance to extract the PAHs completely from this matrix. Only polar and protic solvents such as methanol or water provide access for complete extraction of the PAHs. In contrast, nonpolar and polar aprotic solvents, which are often used as extraction solvents for PAHs (e.g. n-hexane or acetone) exhibit only very low extraction efficiencies below 20% [2].

- The maximum levels of PAHs in various foods are regulated in the EU by the Commission Regulation (EU) 2023/915 [3] and in Switzerland by the regulation SR 817.022.15 on the maximum levels for contaminants [4] and are 1 µg/kg to 10 µg/kg for BaP and 1 µg/kg to 50 µg/kg for the sum of the four PAHs BaA, BaP, BbF and Chr.
- Although PAHs in whey protein powder are not specifically regulated, the material still offers an excellent opportunity to mimic protein-rich food matrices and test analytical methods.



PAHs	Certified value (µg/kg)	Uncertainty* (µg/kg)
BaA	3.17	0.34
BaP	4.18	0.46
BbF	4.73	0.51
Chr	2.85	0.30

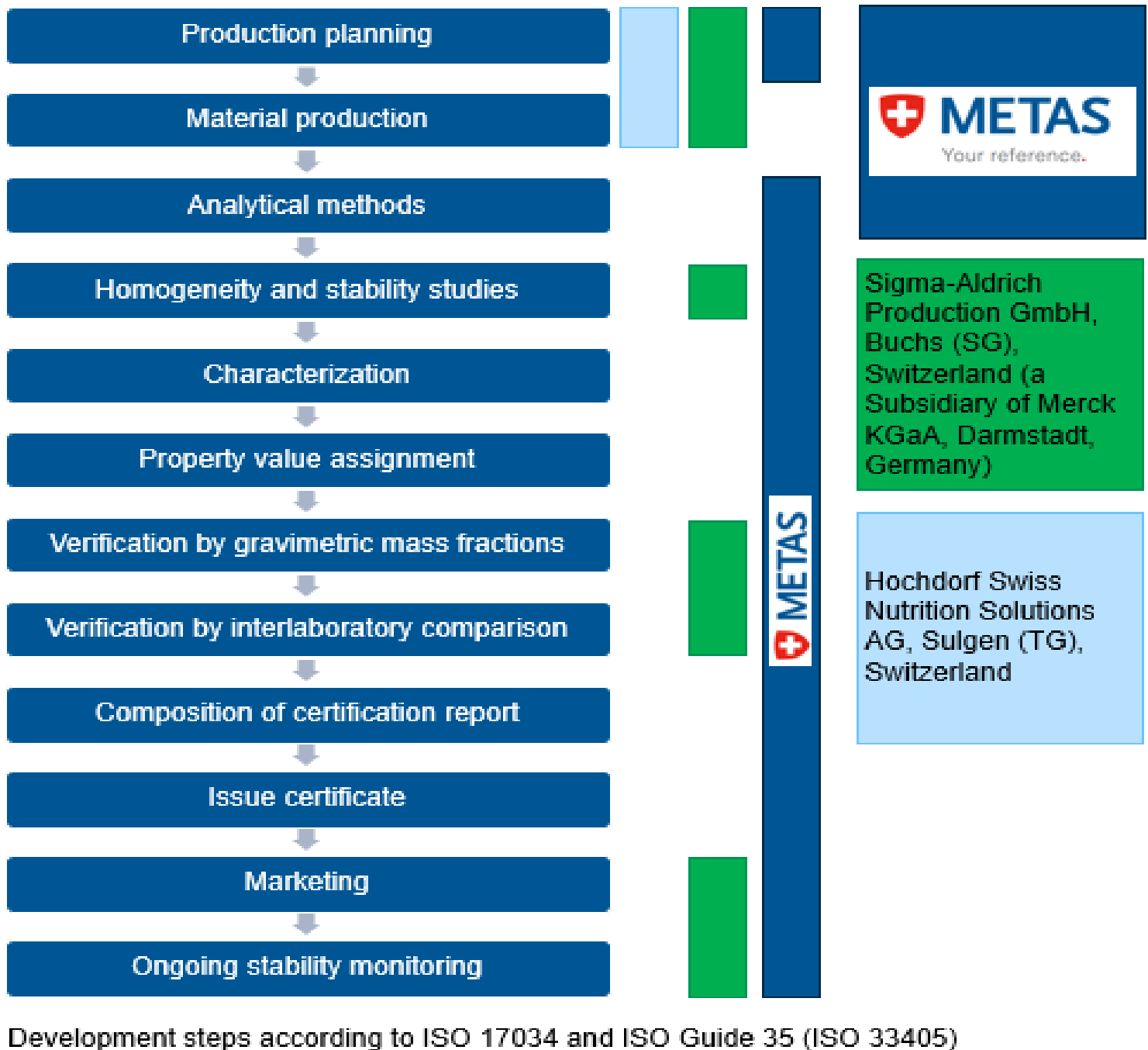
* The uncertainty of the certified value is the expanded uncertainty using a coverage factor $k = 2$, corresponding to a confidence interval of approximately 95%

References

- [1] P. Rohrer, S. Lobsiger, Certified reference materials: metrology in support of food safety, The Reference. (2023), <https://www.metas.ch/metas/en/home/dok/publikationen/diereferenz.html>
- [2] S. Lobsiger, L. Märki, S. Mallia, G. Umbricht, H. Sprecher, K. Breitruck, M. Obkircher, Development of a novel certified reference material for the determination of polycyclic aromatic hydrocarbons (PAHs) in whey protein powder, Anal Bioanal Chem (2023), <https://doi.org/10.1007/s00216-023-04863-9>
- [3] European Commission. Commission Regulation (EU) 2023/915 of 25 April 2023 on maximum levels for certain contaminants in food and repealing Regulation (EC) No 1881/2006 (Text with EEA relevance). Off J Eur Union. 2023;119:103–57.
- [4] Federal Department of Home Affairs (FDHA). Regulation of the FDHA on maximum levels for contaminants of 16 December 2016 (status at 1 February 2024). SR 817.022.15.

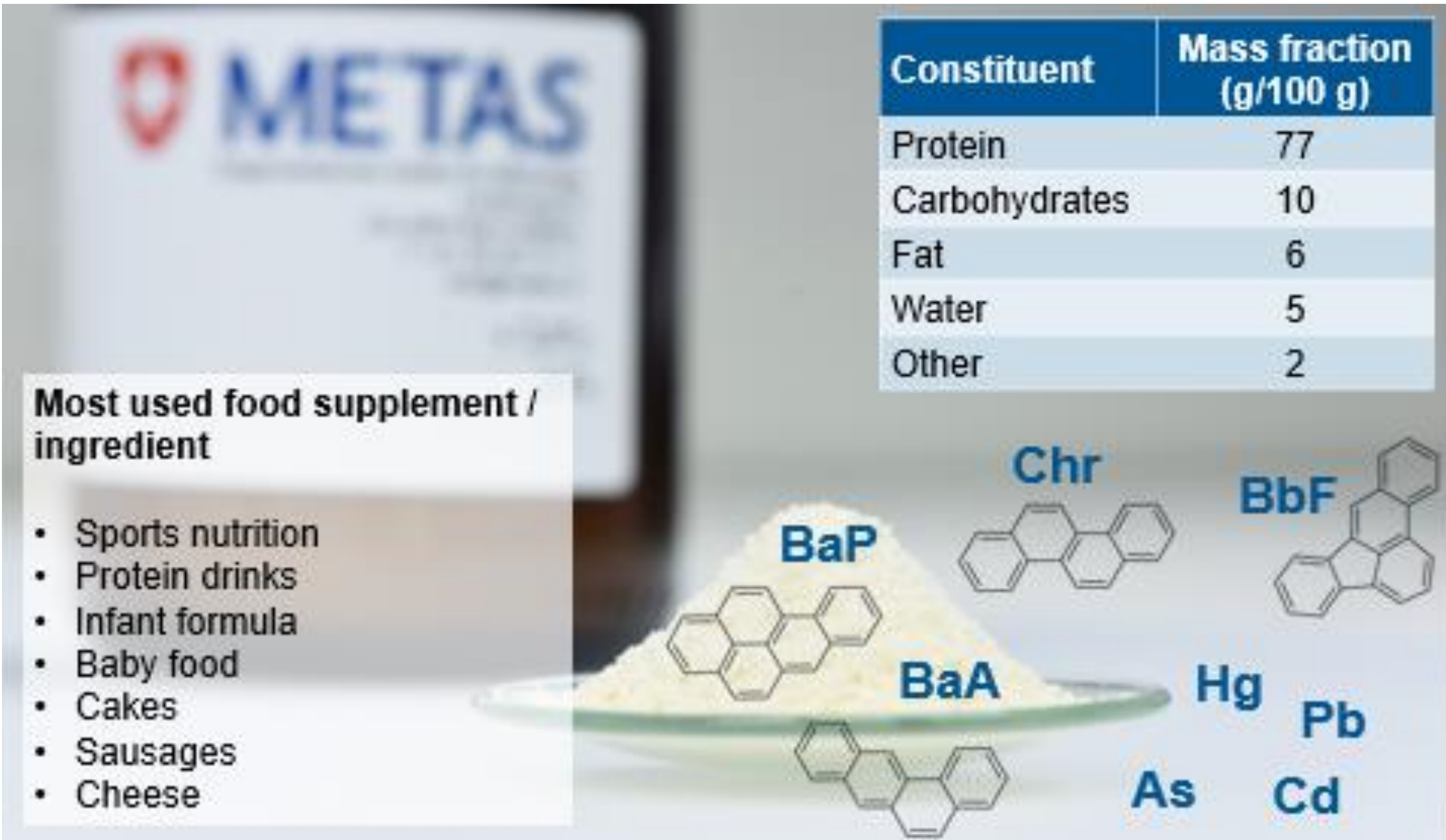


CRM-Development



Development steps according to ISO 17034 and ISO Guide 35 (ISO 33405)

Whey protein



Toxic elements

The maximum levels of toxic elements in food supplements are regulated in the EU by the Commission Regulation (EU) 2023/915 [3] and in Switzerland by the regulation SR 817.022.15 on the maximum levels for contaminants [4] and are 1 mg/kg for Cd, 0.1 mg/kg for Hg and 3 mg/kg for Pb.

The use of WP-CBR001 allows the control of the efficiency of microwave digestion and the comparability of the results using different analytical methods, such as Inductively Coupled Plasma-Mass Spectrometry (ICP-MS), Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) or Direct Mercury Analyzer (DMA).

Element	Certified value (mg/kg)	Uncertainty* (mg/kg)
As	0.21	0.06
Cd	0.200	0.026
Pb	0.494	0.039
Hg	0.055	0.011

* The uncertainty of the certified value is the expanded uncertainty using a coverage factor $k = 2$, corresponding to a confidence interval of approximately 95%

Conclusion

This CRM is helpful for choosing the most appropriate solvent for the extraction of the PAHs in order to obtain correct results. The WP-CBR001 is a versatile CRM for PAHs and toxic elements and can be used not only for whey protein, but also for a variety of protein matrices, including plant-based proteins and meat alternatives.

Distribution of WP-CBR001

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International
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Product No. WP-CBR001

