International Measurement Confederation (IMEK0)
TC 23 "Metrology in Foods and Nutrition"
7th IMEKOFOODS International Conference. Worldwide food trade and consumption: quality and risk assessment
25-27 October 2023, Maisons-Alfort/Paris, France

Development and validation of a new analytical approach for simultaneous speciation analysis of Hg and Se in fishery products by HPLC-ICP-MS

M. Ribeiro[1,2,3], E. Galli[1], J. A. L. Silva[3], I. Castanheira[2], A. Leufroy[1] and P. Jitaru[1*]

[1]ANSES, Laboratory for Food Safety, Université Paris-Est, 14 Pierre & Marie Curie, F-94701 Maisons-Alfort, France
[2]Departamento de Alimentação e Nutrição, Instituto Nacional de Saúde Doutor Ricardo Jorge, INSA IP, Avenida Padre Cruz, 1649-016, Lisbon, Portugal
[3]Centro de Química Estrutural, Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, 1049-001, Lisbon, Portugal

This study reports the development and validation of a new analytical method for the simultaneous speciation analysis of Se and Hg in fish muscle. For this purpose, four Se species (Se(IV), Se(VI), Se-methionine/SeMet and Se-cysteine/SeCys) and two Hg species (Hg(II) and methylmercury/MeHg) were extracted simultaneously by microwave assisted enzymatic hydrolysis (using protease XIV at pH ≅ 7 and 2-mercaptoethanol) and then separated in less than 15 min by using a mixed mechanism anion-cation exchange HPLC with a mobile phase consisting of a mixture of methanol 5% (v/v), 45 mM HNO_3, 0,015% 2- mercaptoethanol and 1,5 mM sodium 3-mercapto-1-propanesulfonate. The separated species were detected online by inductively coupled plasma-mass spectrometry.

The speciation method was validated by means of the accuracy profile approach by carrying out 3 measurement series in duplicate on (3) different days over a time-span of 3 weeks. The quantification limits (LQ) are in the range of 0.055-0.074 µg/L for all selenium species, except for Se(IV) (0.85 µg/L) while the coefficient of variation in terms of intermediate reproducibility (CV_R) was < 7%. For MeHg, the LQ for was 0.037 µg/L, while the CV_R was 3%.

The method was successfully applied to the analysis of muscle samples from four different fish species: rainbow trout, tuna, swordfish and dogfish.

Keywords: SeMet, Se(IV), Se(IV), SeCys, MeHg, anion-cation exchange, fish, MAEE

* E-mail: petru.jitaru@anses.fr