

Development & validation of a novel method for Hg speciation analysis in food by HPLC-ICP-MS and application to the 3rd (French) Total Diet Study

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A simple, fast and a robust method for routine speciation analysis of inorganic (Hg^{2+}) and methylmercury (MeHg) by reverse phase (RP) high-performance liquid chromatography coupled to inductively coupled plasma mass spectrometry (HPLC-ICP-MS) was optimized and validated. This novel method is to be applied for the analysis of a large panel of foodstuffs samples within the framework of the 3rd (French) total Diet Study (3rd TDS). TDSs are endorsed by the World Health Organisation (WHO) and Food and Agriculture Organization of the United Nations (FAO) and they are one of the most efficient tools for national assessment of chemical contamination of food prepared as consumed. This allows estimating human dietary exposure after matching contamination data (occurrence) with the consumption patterns based on samples representativeness. TDSs provide valuable scientific information to national authorities in order to address the risk for the general population in terms of food chemical hazards and permitting to protect the public health on a long-term basis.

An effective separation of Hg^{2+} and MeHg species was achieved in less than 7 min using a peptide mapping RP column. The development was carried out using an experimental design for the HPLC separation and the sample extraction. In the latter case, two extraction approaches, employing a closed microwave system and the use of a heating block, respectively, were compared.

The method was validated based on the accuracy profile approach according to the NF V03-110 French standard, which takes into account the simultaneous assessment of the accuracy and precision of the method. The accuracy profile is an expression of the combination of the systematic (trueness) and the random error (repeatability and/or intermediate precision) for a series of analyst's levels in various matrices in range of concentrations called validity domain. For this purpose, five measurement series were repeated in duplicate on (5) different days, over a timespan of two months for constructing the accuracy profile.

The method was applied to the speciation analysis of MeHg and Hg^{2+} in a variety of foodstuffs of the 3rd (French) Total Diet Study.

Keywords: Hg speciation analysis, food, microwave extraction, HPLC-ICP-MS, Total Diet Study

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