

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

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Total Diet Studies are one of the most efficient tools for national assessment of chemical contamination of food prepared as consumed. They are endorsed by the World Health Organisation (WHO) and Food and Agriculture Organization of the United Nations (FAO). For assessing potential risks of dietary exposure, a simple, fast and a robust method for routine speciation analysis of arsenic species was optimized and validated, this 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).

In this method, multiple arsenic species including, arsenobetaine (AsB), dimethylarsinic acid (DMA), monomethylarsonic acid (MMA), arsenite (AsIII) and arsenate (AsV) were extracted using two extraction approaches, employing a closed microwave system and a heating block, respectively (only N-ethylmaleimide in Ultrapure water was used, as extraction solution, to prevent the interconversion of AsIII and other AsSugars). The separation was achieved in less than 10 min by an anion exchange high-performance liquid chromatography (HPLC) with a mobile phase consisting of a mixture of methanol and ammonium carbonate ((NH₄)₂CO₃). The separated species were detected online by inductively coupled plasma-mass spectrometry (ICP-MS).

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, six measurement series were repeated in duplicate on (6) different days, over a time span of 6 weeks for constructing the accuracy profile.

The method was applied to the speciation analysis of AsB, DMA, MMA, AsIII and AsV in a variety of foodstuffs of the 3rd (French) Total Diet Study.

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

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