Poster

Fine tuning and validation of an analytical method for determinating heavy metals in food samples by ICP-MS technique.

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ABSTRACT

Heavy metals are chemical elements with high density that can be toxic for humans. For that reason consumer and human nutrition agencies have established maximum limits of heavy metals in food. In order to quantify heavy metals, analytical techniques must have low detection limits and high precision and accuracy. In this sense, inductively coupled plasma mass (ICP-MS) is an attractive method as is a multielemental analytical technique wich is able to detect metals and non-metals at concentrations of ppb. The aim of this work is to tune up and validate a method for determining heavy metals (lead, mercury and cadmium) in food by ICP-MS.

ICP-MS used was an ICP Mass Spectrometer ELAN DRC-e Axial Field Technology (Perkin Elmer SCIEX, Waltham). This equipment had a cooler recirculator PolyScience (Nile) coupled and a S10 autosampler (Perkin Elmer, Waltham). Operating conditions were Nebulizer gas flow: 0.87, Plasma flow: 15.00, lens voltage: 6.00, ICP radio frequency generator: 1100. Food samples were homogenized with a mixer and digested with a Digi-Prep 50/24 digestor with HNO3:HCl 6:1.

In order to tune up the method, calibrating method was studied in the matrices to be analyzed. Linearity was also studied, designing calibration curves and adjusting them by instrumental calibration technique ICP-MS requires. In the same way, the response factor, the relation between the signal of the internal standard and each standard of calibration curve, deviations of parameters of the equation and residuals were done. Once the method was tuned up, validation tests were performed; detection and quantification limits were determined, and recovery of interferences correction, tests of precision and accuracy, uncertainty estimation and definition of criteria for acceptance and rejection were carried out.

This study allowed the tuning and establishment of different criteria to give an appropriate measure method, which was validated in order to calculate precision and accuracy of the analytical technique. Which is more, it was possible to verify that the interfering selenium, tin and molybdenum were properly corrected.

REFERENCES

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Reglamento (CE) No 1881/2006 de la Comisión, de 19 de Diciembre, por el que se fija el contenido máximo de determinados contaminantes en los productos alimenticios.