

Certification measurement of the cadmium, copper and lead contents in rice using isotope dilution inductively coupled plasma mass spectrometry

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Abstract

This paper describes the certification of the Cd, Cu and Pb amount contents in a rice material, that were used as reference values for round 19 of the international measurement evaluation programme (IMEP).

The protocol developed in this study was based on isotope dilution associated to inductively coupled plasma mass spectrometry (ID-ICP-MS). A multiple spiking approach was applied to reduce significantly the number of analytical steps. For the decomposition of the sample, three different microwave assisted digestion procedures were tested and compared. The use of hydrofluoric acid was found necessary to ensure full solubilisation and complete isotopic equilibration. Estimation of the combined uncertainty attached to the measurement results was performed according to the ISO guidelines. Contributions from the correction for moisture content, sample homogeneity, procedural blank, spike impurities, spectral interferences, instrumental background and dead-time effects were evaluated.

SI-traceable values with less than 2% combined uncertainty ($k = 2$) were obtained for Cd, Cu and Pb, respectively, $(14.39 \pm 0.21) \times 10^{-6} \text{ mol kg}^{-1}$, $(44.31 \pm 0.42) \times 10^{-6} \text{ mol kg}^{-1}$ and $(2.034 \pm 0.034) \times 10^{-6} \text{ mol kg}^{-1}$.

The rice powder was observed to be highly hygroscopic and a second series of isotope dilution mass spectrometry measurements was carried out on samples in equilibrium with the ambient moisture conditions ('saturated' samples). The Comité Consultatif pour la Quantité de Matière (CCQM) requested this approach for the participation to the key comparison 24 on the same rice test material. The excellent agreement for Cd between the IMEP-19 reference value, the value submitted by the institute for reference materials and measurements (IRMM) to CCQM-K24 and the corresponding reference value obtained as the arithmetic mean from the results of the 11 participating National Measurement Institutes (less than 0.27% difference) further validated this work. Eventually, this series of results on 'saturated' samples was submitted, for the 3 elements, as the IRMM contribution to the certification of the IRMM-804 rice candidate certified reference material (CRM).

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1. Introduction

Rice is the main foodstuff for about half of the world's population. For the last decades rice consumption has been expanding beyond the traditional rice-grown areas, particularly in Europe. In most countries, surveillance measures are taken regarding the presence of contaminants in important

foodstuff such as rice to protect the public health. Cd and Pb maximum levels in foodstuff are set in relevant regulation such as the EC Directive 466/2001 [1].

Cadmium may induce dysfunction and reproductive deficiencies in humans and is suspected to act as a human carcinogen [2]. Lead can cause brain damage, kidney failure, seizures and anaemia [3]. Accurate measurement of trace levels of essential elements like Cu in foodstuffs is also of crucial importance to estimate dietary uptake rates required for normal human growth.

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