Inorganic arsenic - SPE HG-AAS method for RICE tested in-house and collaboratively

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INORGANIC ARSENIC

SPE HG-AAS RICE METHOD TESTED IN-HOUSE AND COLLABORATIVELY

INTRODUCTION
Internationally accepted validated method(s) are needed for establishment of a maximum level (ML) for inorganic arsenic (iAs) in rice as recently emphasised by the European Food Safety Authority (2009), the World Health Organization (2011) and Codex Alimentarius (2012).

Rice contains most often three forms of the trace element arsenic; iAs and the methylated species monomethylarsonic acid (MAV) and dimethylarsinic acid (DMAV). Dietary intake of iAs is of special concern due to its carcinogenicity to humans, whereas DMA and MA are generally considered of less toxicological importance.

CONCLUSION
This SPE HG-AAS method enables selective determination of inorganic arsenic in rice and rice products by use of inexpensive instrumentation (HG-AAS) and is a candidate method for future control.

VALIDATION RESULTS
In-house validation was satisfying (Tab 1.) and was in accordance with previous results for marine samples (Rasmussen et al. 2013). The LOD (0.02 mg·kg⁻¹) was below the proposed maximum levels (0.2-0.3 mg·kg⁻¹).

Collaborative test of the SPE separation method on a wholemeal rice meal sample gave a satisfactory HorRat value of 1.6 among 10 laboratories.

Table 1. In-house validation

<table>
<thead>
<tr>
<th>Spiked rice samples</th>
<th>Rice reference materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>Target level (mg·kg⁻¹)</td>
<td>0.30</td>
</tr>
<tr>
<td>Observations (N)</td>
<td>9</td>
</tr>
<tr>
<td>Mean recovery (%)</td>
<td>104</td>
</tr>
<tr>
<td>Reproducibility RSD (%)</td>
<td>3</td>
</tr>
<tr>
<td>Reproducibility RSD (%)</td>
<td>7</td>
</tr>
</tbody>
</table>

*Concentrations were determined by HG-AAS.

SPE HG-AAS vs HPLC-ICP-MS

A comparison of the results from the two methods (Fig. 3) showed good agreement.

RICE SAMPLES

The iAs concentration determined by SPE HG-AAS in 36 rice samples purchased on the Danish retail market varied (0.03-0.60 mg·kg⁻¹), with the highest concentration found in a red rice sample.

Figure 1. Overlaid HPLC-ICP-MS chromatogram of 3 SPE fractions (load, wash and eluate) of a rice sample (NIST1568a) containing both inorganic and organoarsenic species.

Figure 3. Determination of inorganic arsenic by two different methods, HPLC-ICP-MS and SPE HG-AAS. In total results for 84 spiked and natural incurred rice samples analysed on four different days. The correlation is y=x (99% confidence interval - regression analysis by Excel 2010).