IC-ICP-MS speciation analysis of As in Organic Brown Rice Syrup (OBRS) using the Thermo Scientific iCAP Q ICP-MS

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Key Words
iCAP Q, ICP-MS, ICS-5000, ion chromatography, As, speciation, food

Goal

The analysis of organic brown rice samples (OBRS) for their total arsenic content by ICP-MS and their species concentrations by IC-ICP-MS.

Introduction

Media reports and scientific publications on the determination of arsenic (As) in foodstuffs have sparked renewed interest from consumer groups and politicians leading to responses from national regulatory bodies. Following the publication[1] of a report on high As levels in organic brown rise syrup (OBRS, an ingredient in a variety of organic foods) the United State Food and Drug Administration (FDA) stated that it was carrying out a study on As in rice and rice products that is due to report later in 2012[2].

Based on previous work[3], OBRS samples were analyzed for their total arsenic content by ICP-MS and then subsequently by IC-ICP-MS to determine the concentration of six arsenic species: the two toxic inorganic species (As(III) and As(V)), and four organic species that are considered to be harmless.

Instrument configuration

All measurements – both total As and As speciation – were carried out using the Thermo Scientific iCAP Qc ICP-MS. All As analyses were performed in He KED (Kinetic Energy Discrimination) mode to efficiently reduce polyatomic interferences for the analysis of (monoisotopic) arsenic at m/z 75. Chromatographic separations were carried out using the Thermo Scientific Dionex ICS-5000 ion chromatography system[3] coupled with the iCAP™ Q ICP-MS that was used as a highly sensitive and selective arsenic detector.

Sample Preparation

Three different OBRS samples were sourced and prepared for analysis. For the total As analysis, the closed microwave digestion method described by Jackson et al[1] was used. Preparation of the OBRS samples for As speciation analysis was achieved by taking 1.5 g of OBRS, adding 15 mL of 0.28 M HNO_3 and refluxing for 90 minutes[4].

Total Arsenic Results

A calibration curve for the He KED analysis of total As is shown in Figure 1.

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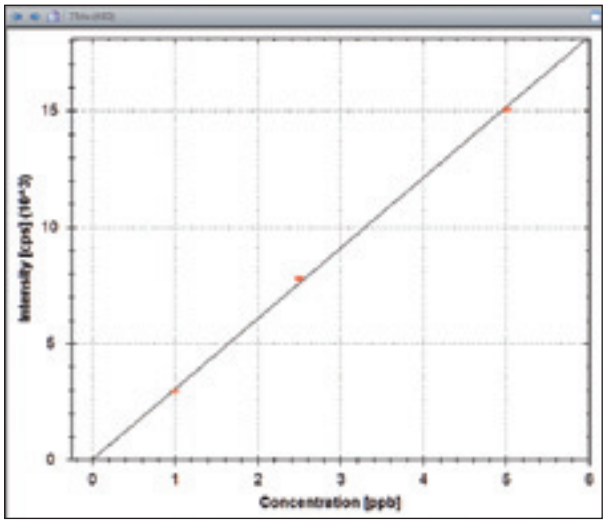

Figure 1. Calibration curve for ^{75}As in He KED mode with standards of 1, 2.5 and 5 ng/g. An ^{75}As sensitivity of 3 kcps per ng/g was achieved.

Table 1. Results from the total As analysis of three OBRS samples. An instrumental detection limit (LoD) of 0.9 pg/g with a background equivalent concentration (BEC) of 0.8 pg/g was achieved using a He KED analysis on the iCAP Qc ICP-MS. Taking into account the 160 fold dilution used, a method detection limit of 0.13 ng/g was achieved.

	^{75}As (ng/g)
OBRS Sample #1	118 ± 7
OBRS Sample #2	136 ± 7
OBRS Sample #3	107 ± 11

As can be seen in Table 1, total As concentrations of > 100 ng/g were found in the three samples analyzed.

Arsenic Speciation Results

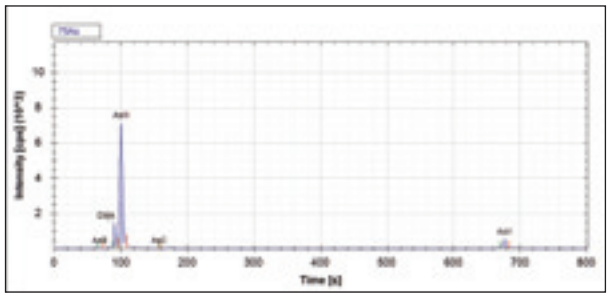

Figure 2. IC-ICP-MS chromatogram of Arsenic species found in a OBRS sample. As (III) was the most abundant species detected.

Conclusion

An ICP-MS method for the quantification of total arsenic and six arsenic species (by IC-ICP-MS) in organic brown rice syrup (OBRS) has been demonstrated. Total arsenic concentrations of between 107 – 136 ng/g were found in the three samples analyzed. Subsequent IC-ICP-MS speciation analysis showed that the predominant As species in the OBRS samples tested was the toxic inorganic As(III) with over 80% of the total arsenic concentration (equivalent to 86 – 109 ng /g As (III)).

References

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