Analysis of Trace Elements in Rice using an Autodilution System Coupled with the Thermo Scientific iCAP Q ICP-MS

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Key Words
Food, prep*FAST*, KED, multi-element, rice

Goal

To demonstrate a simple and highly sensitive method for the trace elemental analysis of rice by quadrupole ICP-MS with minimal sample preparation.

Analytical Challenge

Rice is the worlds second most produced grain, with a global annual production of about 600 million tons. In most Asian countries, rice is a staple food, with a consumption of between 200-400 g per person per day. For both nutritional research and food safety, the analysis of trace elements in rice and derived products[1] is very important, and there has been a growing emphasis on quality.

Depending on geographical location and production processes, rice may also contain trace elements that are toxic or potentially carcinogenic (Pb, Cd, As, Se, Hg). Consequently, a simple, reliable method for the accurate determination of trace elements would facilitate the assessment of the nutritional and toxicological status of the rice.

Sample and Calibration Solution Preparation

Three locally sourced rice (natural, risotto and short grain white rice) and a certified reference material (Rice Flour IRMM-804), were prepared in triplicate using a microwave digestion method. Approximately 0.5 g of sample was acid digested using a mixture of HNO_3 and H_2O_2 in a closed vessel microwave digestion system. After digestion, the samples were made up to volume (50 mL) using ultra pure water. Calibration stock solutions were prepared in 2% HNO_3.

The two standard stocks and all samples were then loaded directly onto the autosampler. From here the prescriptive dilution capabilities of the Elemental Scientific Inc prep*FAST* autodilution system driven through the Thermo Scientific™ Qtegra™ ISDS were used to perform further dilutions, greatly reducing the overall sample prearation time. Ge, Rh and Ir internal standards (at 20 $\mu g \cdot kg^{-1}$) were added on-line at a constant flow via the prep*FAST* system.

Method

A Thermo Scientific™ iCAP™ Qc ICP-MS was used to perform all analyses. All target analytes were measured using a single pure He, kinetic energy discrimination (KED) collision cell mode.

Full mass range analysis in KED mode is made possible through the use of proprietary Thermo Scientific™ QCell flatapole technology. With the QCell the iCAP Qc ICP-MS maintains ion transmission in He KED mode for reliable and accurate multi-elemental analysis of all analytes, even low mass elements such as Li and B. The automatic low mass cut-off in the QCell provides added reliability in KED mode by stopping unwanted by-products generated in the cell from being subsequently detected.

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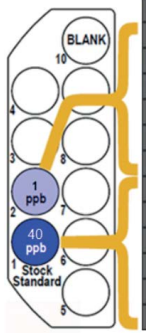
Software

The complete prep*FAST* and iCAP Q ICP-MS system was controlled by the Qtegra ISDS software which allows plug-in based control of a series of accessories as well as providing easy to use data processing for generation of analytical reports.

In combination with Qtegra software, the ESI prep*FAST* is a powerful autodilution system that automatically performs precise and accurate online dilutions. Figure 1 shows a sequence for a complete twelve point external calibration (10 µg kg^{-1} to 40 µg·kg^{-1}) produced from two stock solutions as defined by dilution factors entered into the Qtegra software. The rice samples were analyzed after a prescriptive 4-fold dilution.



	Label	Standard	prepFAST DF
1	Blank		1
2	Blank		1
3	Blank		1
4	10 ppt	Stock 1ppb	100
5	20 ppt	Stock 1ppb	50
6	50 ppt	Stock 1ppb	20
7	100 ppt	Stock 1ppb	10
8	200 ppt	Stock 1ppb	5
9	500 ppt	Stock 1ppb	2
10	1 ppb	Stock 40 ppb	40
11	2 ppb	Stock 40 ppb	20
12	5 ppb	Stock 40 ppb	8
13	10 ppb	Stock 40 ppb	4
14	20 ppb	Stock 40 ppb	2
15	40 ppb	Stock 40 ppb	1

Figure 1. prep*FAST* generation of calibration solutions

Table 1. Results for Rice Flour IRMM-804, Risotto, Natural and Short Grain White (Milk) Rice. All concentrations are reported in µg·kg^{-1} unless otherwise stated. LoD – Instrumental Limit of Detection are based on the analysis of ten replicate measurements. MDL – Method Detection Limit based on a sample weight of 0.5 g in a final volume of 50 mL plus a prescriptive 4-fold dilution (total 400 -fold dilution).

* Indicates this value is not certified.

Isotope	LoD (µg·L^{-1})	MDL	Rice Flour IRMM-804		Risotto Rice	Natural Rice	Milk Rice
			Certified	Measured	Measured	Measured	Measured
^{7}Li	0.04	14		24.7 ± 2.9	23.1 ± 0.9	30.3 ± 1.6	59.0 ± 4.3
^{11}B	0.2	78		980 ± 59	809 ± 41	1146 ± 51	1226 ± 17
^{52}Cr	0.003	1.2		95.5 ± 3.1	22.7 ± 0.6	68.7 ± 1.1	24.2 ± 1.2
^{55}Mn	0.001	0.4	34200 ± 2300	35010 ± 1070	8054 ± 199	26296 ± 1947	5178 ± 121
^{56}Fe	0.01	4.8		9581 ± 248	2203 ± 171	14007 ± 1532	1731 ± 102
^{59}Co	0.0003	0.1		17.1 ± 1.6	16.7 ± 0.6	24.1 ± 2.2	16.9 ± 1.8
^{60}Ni	0.004	1.6		248 ± 4.2	75.6 ± 0.8	355 ± 42	72.1 ± 6.1
^{63}Cu	0.0007	0.29	2740 ± 240	2577 ± 100	1636 ± 3	1241 ± 47	1612 ± 2
^{66}Zn	0.02	6.4	23100 ± 1900	21639 ± 655	9472 ± 227	18950 ± 1965	9205 ± 207
^{75}As	0.004	1.6	49 ± 4	47 ± 1	355 ± 14	158 ± 5	171 ± 5
^{78}Se	0.04	16	38*	36 ± 8	50 ± 2	48 ± 3	41 ± 6
^{88}Sr	0.0009	0.35		195 ± 5.1	154 ± 4.5	535 ± 18	229 ± 5.3
^{111}Cd	0.0008	0.33	1610 ± 70	1550 ± 36	23 ± 0.5	16 ± 1.1	12 ± 1.3
^{121}Sb	0.002	0.8		23.6 ± 1.7	20.7 ± 0.09	24.2 ± 2.0	19.7 ± 2.7
^{202}Hg	0.003	1.2		2.24 ± 0.058	6.79 ± 0.006	3.53 ± 0.11	3.63 ± 0.301
^{208}Pb	0.001	0.4	420 ± 70	383 ± 13	26 ± 0.3	35 ± 0.5	28 ± 3.7

Results

Concentration results obtained for the rice flour reference material and three additional rice samples are presented in Table 1. All measured concentrations are within the certified ranges for the IRMM-804 CRM, demonstrating the accuracy of analysis, even for the low concentration and ICP-MS challenging element, As.

Conclusion

In combination with Qtegra ISDS, the prepFAST system provides fully automated sample handling for high throughput analysis using the iCAP Qc ICP-MS.

In addition to prescriptive dilution capabilities described here, Qtegra ISDS can automatically correct for over calibration range samples through intelligent autodilution. The described system is therefore ideally suited for accurate, routine, multi-elemental analysis of complex food samples.

References

1. AN43126: 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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