Application Note 43101

Multi-element determination in food samples using the Thermo Scientific iCAP Q ICP-MS

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Key Words
iCAP Q, Food, ICP-MS, KED, Multi-Element

Goal

To demonstrate how simultaneous determination of all elements of interest in a wide range of food samples can be efficiently and rapidly performed using the iCAP Q ICP-MS.

Introduction

During the last decade the measurement of toxic, essential and nutritional elements in food has become a major topic of public interest. Intergovernmental bodies sponsored by the Food and Agricultural Organisation and the World Health Organisation are responsible for developing standard test methods for the analysis of food samples. Alongside this regulatory compliance it is necessary to monitor potentially toxic contaminants that could potentially enter the food chain via a series of pathways including, but not limited to, industrial pollution or environmental contamination. Once toxic elements are in the food chain, they can pose significant health risks. For these reasons, it is essential to have a simple, robust, multi-elemental analysis method for major and minor concentrations of elements in food. The elemental and dynamic range of ICP-MS makes it particularly suited to the analysis of food, simultaneously determining trace level contaminants and macro level nutrients.

Sample and calibration solution preparation

A set of 80 food samples including cereal, seafood and dairy products, alongside certified reference materials (Rice Flour IRMM-804 and Chicken NCS ZC73016), were prepared to evaluate the proposed ICP-MS method. Approximately 0.5g of sample was acid digested using a mixture of HNO3 and HCl in a closed vessel microwave digestion system. Calibration solutions at appropriate concentrations for the analytes measured were prepared in 1 % (v/v) HNO3. Internal standardisation was applied, with Ga, Rh and Ir internal standards at 20, 10 and 10 ppb respectively, added on-line via a T-piece before the nebulizer.

Instrument configuration

A Thermo Scientific iCAP Qc ICP-MS was used for all measurements. The sample introduction system used consisted of a Peltier cooled (3 °C), baffled cyclonic spray chamber, PFA nebulizer and quartz torch with a 2.5 mm i.d. removable quartz injector. The instrument was operated in a single collision cell mode, with kinetic energy discrimination (KED), using pure He as the collision gas. All samples were presented for analysis using a SC4 DX autosampler from Elemental Scientific (Omaha, NE, USA).

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General analytical conditions

The iCAP™ Qc ICP-MS was operated in a single KED mode using the following parameters:

Parameter	Value
Forward power	1500 W
Nebuliser gas	0.9 L/min
Auxiliary gas	0.8 L/min
Cool gas flow	14.0 L/min
Collision cell gas	He at 4.5 mL/min
Sample uptake/wash time	45 s each
Dwell times	Optimized per analyte
Number of points per peak	1
Number of repeats per sample	3
Total acquisition time	3 min

Table 1: Instrument operating parameters.

Results and Discussion

The use of a single KED mode is made possible through the use of unique Thermo Scientific QCell flatapole technology. Sample throughput is significantly improved by using a single analysis mode – a significant advantage for the analysis of food since large numbers of samples may have to be rapidly screened. With previous ICP-MS instrumentation, the use of a single He KED mode for full mass range multi-elemental analyses gave lower sensitivity and consequently poorer detection limits for low mass analytes such as Li. However, the iCAP Qc ICP-MS in He KED mode provides sufficient low mass sensitivity for accurate multi-elemental analyses in food samples. Table 2 shows the typical method detection limits achievable for a range of analytes measured using this method.

Taking into account the 1:100 dilution factor required for this analysis, Table 2 shows that ppb range method detection limits are easily achieved for all analytes, and that detection limits for the major constituent elements are well below the target levels required for food analysis.

Figures 1 and 2 show typical external calibration curves for the high (Na, 0-100 ppm) and low level (Li, 0-100 ppb) analytes determined simultaneously with the iCAP Qc ICP-MS single KED mode.

The results of the rice flour and chicken reference material measurements are presented in Tables 3 and 4.

Isotope	Interference removed/attenuated	Method detection limit (ppb) unless stated
^{7}Li	---	3
^{11}B	---	10
^{23}Na	---	0.3 (ppm)
^{25}Mg	---	0.01 (ppm)
^{31}P	$^{14}N^{16}O^{1}H^{+}$	0.6 (ppm)
^{34}S	$^{16}O^{18}O^{+}$	9 (ppm)
^{39}K	---	0.5 (ppm)
^{44}Ca	---	0.2 (ppm)
^{52}Cr	$^{40}Ar^{12}C^{+}$, $^{35}Cl^{16}O^{1}H^{+}$, $^{36}Ar^{16}O^{+}$	0.2
^{55}Mn	$^{40}Ar^{14}N^{1}H^{+}$, $^{40}Ar^{15}N^{+}$	1
^{56}Fe	$^{40}Ar^{16}O^{+}$, $^{40}Ca^{16}O^{+}$	4
^{60}Ni	$^{44}Ca^{16}O^{+}$	2
^{65}Cu	---	0.8
^{66}Zn	---	2
^{75}As	$^{40}Ar^{35}Cl^{+}$	0.2
^{78}Se	$^{38}Ar^{40}Ar^{+}$	1
^{88}Sr	---	0.1
^{98}Mo	---	1
^{111}Cd	---	0.3
^{138}Ba	---	0.3
^{141}Pr	---	0.02
^{208}Pb	---	0.1

Table 2: Typical method detection limits achievable in food using He KED mode on the iCAP Qc.

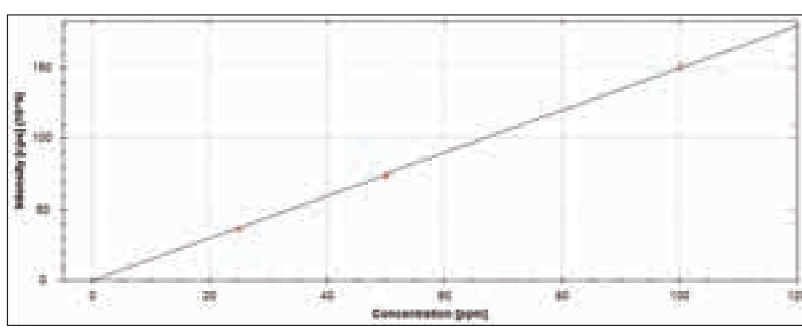

Figure 1: Screenshot from the Thermo Scientific Qtegra software showing the Na (0-100 ppm) calibration curve.

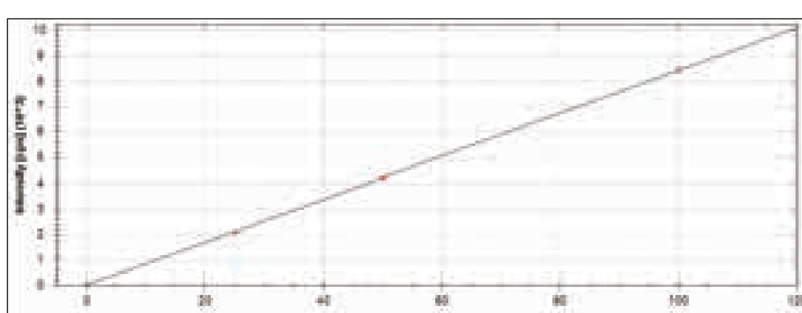

Figure 2: Screenshot from the Qtegra™ software showing the Li (0-100 ppb) calibration curve.

Isotope	Measured	Certified
^{55}Mn	35800 ± 470	34200 ± 2300
^{65}Cu	2650 ± 30	2740 ± 240
^{66}Zn	23100 ± 270	23100 ± 1900
^{75}As	52.3 ± 0.8	49 ± 4
^{78}Se	35.1 ± 1.0	38 (Reference value)
^{111}Cd	1620 ± 9	1610 ± 70
^{208}Pb	460 ± 8	420 ± 70

Table 3: Results for the IRMM-804 rice flour reference material (dilution corrected, all concentrations reported in ppb).

Isotope	Measured	Certified
^{7}Li	28 ± 1	34 ± 7
^{11}B	730 ± 23	760 ± 130
^{23}Na (ppm)	1310 ± 25	1440 ± 90
^{25}Mg (ppm)	1200 ± 22	1280 ± 100
^{31}P (ppm)	8950 ± 220	9600 ± 800
^{34}S (ppm)	8310 ± 220	8600 ± 500
^{39}K (ppm)	14000 ± 480	14600 ± 700
^{44}Ca (ppm)	200 ± 4	220 ± 20
^{52}Cr	450 ± 10	590 ± 110
^{55}Mn	1640 ± 20	1650 ± 70
^{56}Fe	32700 ± 260	31300 ± 3000
^{60}Ni	153 ± 2	150 ± 30
^{65}Cu	1350 ± 11	1460 ± 120
^{66}Zn	25300 ± 220	26000 ± 1000
^{75}As	115 ± 1	109 ± 13
^{78}Se	549 ± 11	490 ± 60
^{88}Sr	611 ± 11	640 ± 80
^{98}Mo	112 ± 1	110 ± 10
^{138}Ba	1610 ± 16	1500 ± 400
^{141}Pr	2.6 ± 0.1	2.8 ± 0.6
^{208}Pb	90.7 ± 2.0	110 ± 20

Table 4: Results for the NCS ZC73016 chicken reference material (dilution corrected, all concentrations reported in ppb except where stated).

Excellent agreement was observed between the measured and reference values for all target analytes in the two reference materials analyzed.

Continuing calibration checks (CCVs) and the reference materials were periodically analyzed throughout the analytical run with good agreement to expected levels illustrating the robustness of the method. Six CCV checks were analyzed at intervals during the 8 hour analysis. Figure 3 shows the average concentration of the CCV standard and the in-run relative error for a range of high and low level analytes.

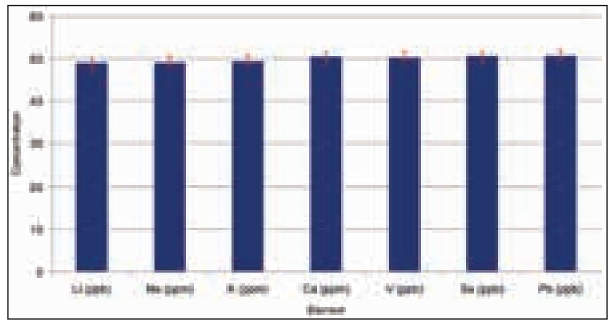

Figure 3: Calibration check verification standards measured during the analysis.

The results from the CCV checks throughout the analysis show that there was minimal drift between the batches of food samples, eliminating the need for any sensitivity re-calibration within the 8 hour analysis period. To further demonstrate the stability and robustness of the iCAP Qc ICP-MS for prolonged measurement of high matrix samples, the absolute suppression and relative drift of the internal standards throughout the analysis was evaluated. The variation in the internal standard signals during the run is shown in Figure 4.

While there is minimal per sample variation in the internal standard intensities, by the end of the 8 hour analysis the internal standard response is essentially the same as it was initially. This behaviour highlights the excellent robustness of the iCAP Qc ICP-MS in terms of both matrix resistance and interference removal in the analysis of food samples. This latter aspect is particularly significant since food samples are well recognized to contain a range of elements at widely varying concentrations.

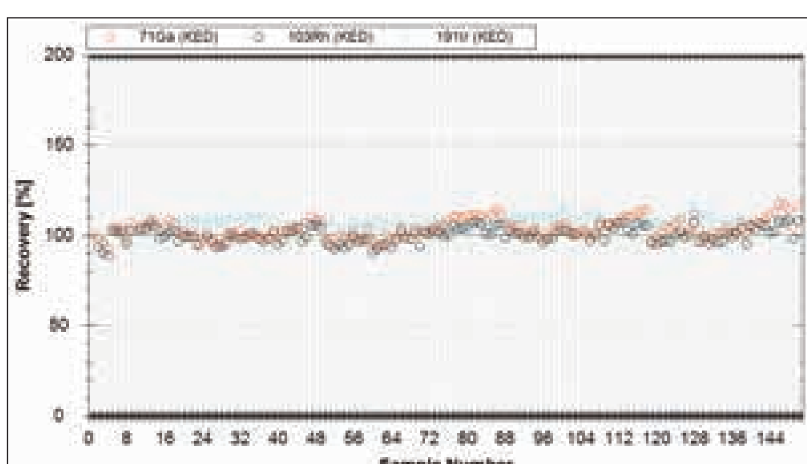

Figure 4: Variation of the internal standard intensities throughout the 8 hour analysis.

As part of this study, the reference materials were repeatedly analyzed during the analysis. Five independent measurements were made of separate aliquots of each reference material to assess the reproducibility of the method. The results from these analyses are shown in Tables 5 and 6.

Isotope	Mean	Standard deviation	% RSD
^{55}Mn	35500	170	0.5
^{65}Cu	2670	11	0.4
^{66}Zn	23400	170	0.7
^{75}As	52.7	0.7	1.4
^{78}Se	34.7	0.5	1.3
^{111}Cd	1630	11	0.7
^{208}Pb	490	3.9	0.8

Table 5: Reproducibility of the IRMM-804 rice flour reference material (n=5). All concentrations are reported in units of ppb.

Isotope	Mean	Standard deviation	% RSD
^{7}Li	29	0.6	1.9
^{11}B	720	14	1.9
^{23}Na (ppm)	1380	18	1.3
^{25}Mg (ppm)	1170	13	1.1
^{31}P (ppm)	8700	150	1.7
^{34}S (ppm)	8230	150	1.9
^{39}K (ppm)	13800	240	1.8
^{44}Ca (ppm)	196	3.3	1.7
^{52}Cr	440	4.2	0.9
^{55}Mn	1630	13	0.8
^{56}Fe	32600	225	0.7
^{60}Ni	149	1.2	0.8
^{65}Cu	1330	9	0.7
^{66}Zn	25200	150	0.6
^{75}As	114	1.0	0.9
^{78}Se	550	8.9	1.6
^{88}Sr	616	9.7	1.6
^{98}Mo	113	2.1	1.9
^{138}Ba	1620	23	1.4
^{141}Pr	2.5	0.04	1.6
^{208}Pb	89.8	0.9	1.0

Table 6: Reproducibility of the NCS ZC73016 chicken reference material (n=5). All concentrations are reported in units of ppb except where stated.

The results in Table 5 and 6 demonstrate that excellent reproducibility was achieved for the five repeat analyses of Rice flour and Chicken reference materials over 8 hours, with RSD's of <2 % obtained for all of the elements determined.

Conclusions

The iCAP Qc ICP-MS has been shown to be an excellent tool for multi-elemental determination in complex food samples. With its high sensitivity and analytical robustness, the iCAP Qc is routinely capable of measuring major and minor analyte concentrations in food in a single KED analysis mode, following a simple sample digestion step. Using the iCAP Q ICP-MS collision cell technology, interference-free, simultaneous determination of all elements of interest in a wide range of food sample types can be efficiently and rapidly performed.

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