APPLICATION BRIEF

ICP - Mass Spectrometry

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The Elemental Analysis of Meat and Seafood with the NexION 300/350 ICP-MS

Introduction

The elemental content of food products is very important, both in relation to nutritional and toxic elements. Nutritional elements can either be native to the food substance or can be added to enhance the health benefits. Toxic elements can enter food either through the environment or processing during production. Hopefully, toxic elements will be present at extremely low levels, while nutritional elements will be present at optimal levels: if too high, they may be toxic; if too low, the food will not provide the necessary nutrition. Therefore, the elemental analysis of food requires the ability to measure both trace and high levels.

The elemental capabilities and dynamic range of inductively coupled plasma mass spectrometry (ICP-MS) make it ideally suited for the analysis of food materials. The ultratrace detection limits of ICP-MS permit the determination of low-level contaminants, such as Pb, As, Se, and Hg, while the macro-level nutritional elements, such as Ca, Mg, K, and Na, can be quantified using the extended dynamic range capability of ICP-MS which provides the ability to measure concentrations over nine orders of magnitude. However, there are still a number of challenges to overcome, including complex sample matrices, high levels of dissolved solids, and interferences. With the proper ICP-MS instrumental conditions and design, all of these issues can be overcome, allowing for the successful analysis of food samples, as described elsewhere[1]. This work will focus on the analysis of meat and seafood; foods such as these are high in protein content which is important for body growth and repair.

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Experimental

Sample Preparation

NIST® 8414 Bovine Muscle and NIST® 2976 Mussel Tissue were used in this work. Approximately 0.5-0.6 g were digested in duplicate with 5 mL of nitric acid (Fisher Scientific™, Optima grade) and 2 mL of hydrogen peroxide (Fisher Scientific™, Optima grade) in pre-cleaned PTFE microwave sample vessels. The digestion program consisted of 30 min of heating and 15 min of cooling, as shown in Table 1. All samples were completely dissolved, resulting in clear solutions that were diluted to a final volume of 50 mL with deionized water. No further sample dilutions were necessary. Gold was added to all solutions at a final concentration of 200 µg/L to stabilize mercury. Preparation blanks, consisting of the acid mixture, were taken through the same microwave digestion program as the samples.

Table 1. Microwave Digestion Program.

Step	Power (W)	Ramp (min)	Hold (min)
1	500	1	4
2	1000	5	5
3	1400	5	10
4 (cooling)	0	—	15

Instrumental Conditions

All data in this study were generated under normal operating conditions on a PerkinElmer NexION® 300/350X ICP-MS using an autosampler. The instrumental operating conditions are shown in Table 2.

Table 2. ICP-MS Instrumental Operating Conditions for this Application.

Parameter	Value
Nebulizer	Glass concentric
Spray chamber	Glass cyclonic
Cones	Nickel
Plasma gas flow	18.0 L/min
Auxiliary gas flow	1.2 L/min
Nebulizer gas flow	0.98 L/min
Sample uptake rate	300 µL/min
RF power	1600 W
Total integration time	0.5 (1.5 seconds for As, Se, Hg)
Replicates per sample	3
Universal Cell Technology™*	Collision mode

*PerkinElmer, Inc.

Calibration

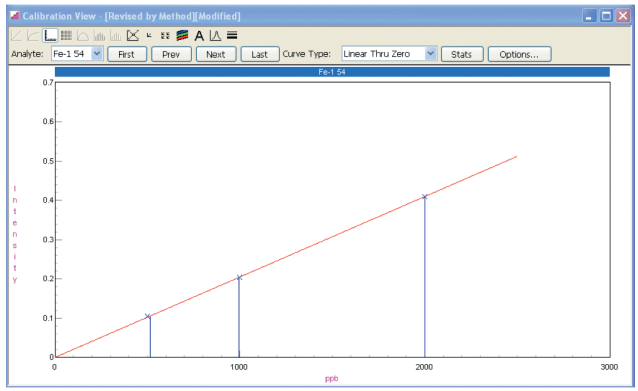
Multielement calibration standards, representing all the analytes in the SRMs, were made up from PerkinElmer Pure single and multielement standards and diluted into 10% HNO_3. Gold was added to all solutions at a final concentration of 200 µg/L to stabilize mercury. Calibration standard ranges were based on whether the analyte was expected to be a high-level nutritional element like potassium (K) or sodium (Na), low/medium-level essential element like manganese (Mn) or iron (Fe), or trace/ultratrace contaminant such as lead (Pb) or mercury (Hg).

Depending on the certified value of the analytes, five different calibration ranges were made up to cover the complete range of elements being determined:

- High-level nutritional analytes: 0-300 ppm
- Medium-level essential analytes: 0-20 ppm
- Low-level essential analytes: 0-2 ppm
- Trace-level contaminants: 0-200 ppb
- Ultratrace-level contaminants: 0-20 ppb

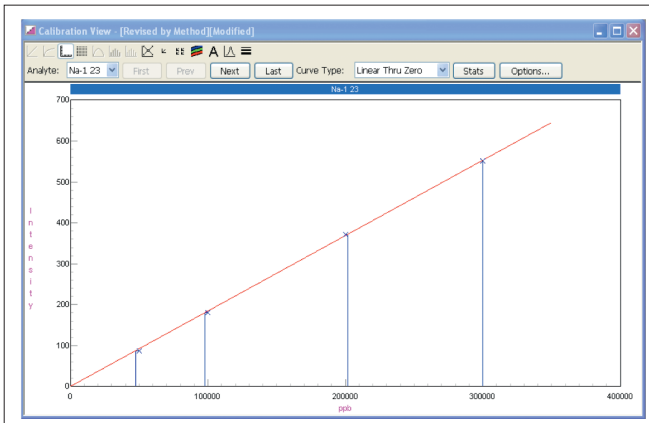
Figures 1 to 5 show representative calibration curves for each range.

In addition to the analyte elements used for the multielement calibration, the standards, blanks, and samples were also spiked on-line using a mixing tee with a solution of ^{6}Li, Sc, Ge, In, and Tb for internal standardization across the full mass range. Acetic acid was added to the internal standard solution to compensate for residual carbon left over from the sample digestion.



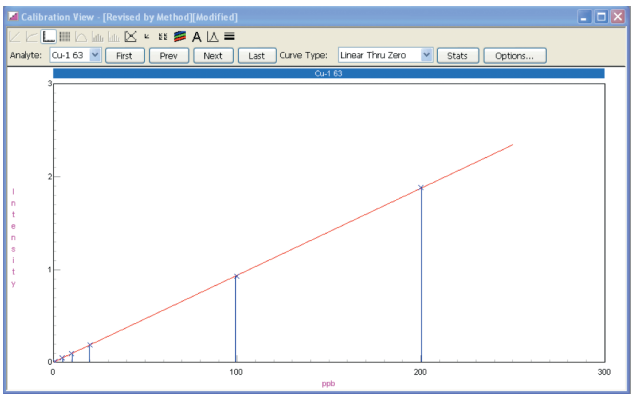
^{54}Fe Correlation Coefficient = 0.99997.

Figure 1. Calibration curves for ^{54}Fe (0-2 ppm).



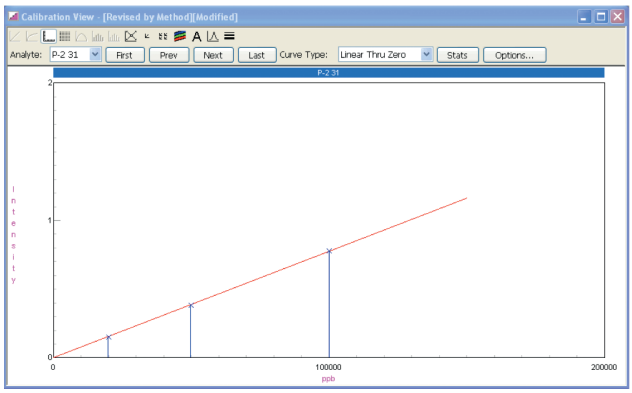
^{23}Na Correlation Coefficient = 0.99996.

Figure 2. Calibration curve for ^{23}Na (0-300 ppm).



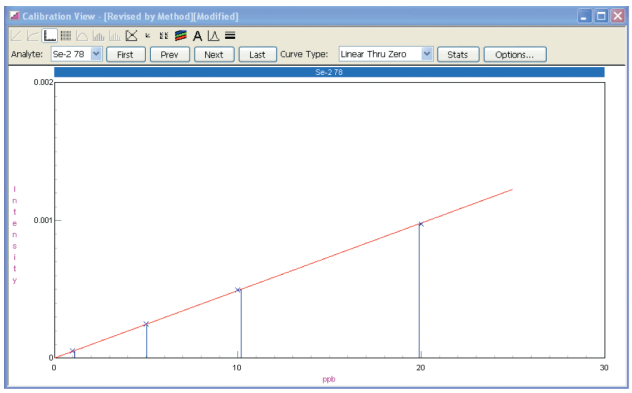
^{63}Cu Correlation Coefficient = 0.99999.

Figure 3. Calibration curve for ^{63}Cu (0-200 ppb).



^{31}P Correlation Coefficient = 0.99999.

Figure 4. Calibration curve for ^{31}P (0-100 ppm).



^{78}Se Correlation Coefficient = 0.99995.

Figure 5. Calibration curve for ^{78}Se (0-20 ppb).

Results

Quantitative results for two sample preparations of the NIST® 8414 Bovine Muscle and NIST® 2976 Mussel Tissue reference materials are shown in Tables 3 and 4. All elements in every sample were determined with Universal Cell operating in Collision mode using helium as the cell gas. Figures in parentheses ( ) in the Reference Value column are not certified values but are included for information purposes only. The data show very good agreement with the certified values, especially for the elements that suffer from known spectral interferences. The elements that are outside the specified limits are mostly the ones that are well recognized as environmental contaminants, which have most likely been impacted by the sample preparation procedure.

Table 3. Analysis of NIST® 8414 Bovine Muscle using the NexION 300/350 ICP-MS.

Element	Mass (amu)	Reference Value (mg/kg)	Experimental Value (mg/kg)
B	11	0.6 ±0.4	0.4
Na	23	2100 ±80	2000
Mg	26	960 ±95	960
Al	27	1.7 ±1.4	1.6
P	31	8360 ±450	7250
S	34	7950 ±410	6820
K	39	15170 ±370	14180
Ca	44	145 ±20	143
V	51	(0.005)	0.006
Cr	52	0.071 ±0.038	0.092
Fe	54	71.2 ±9.2	71.2
Mn	55	0.37 ±0.09	0.44
Co	59	0.007 ±0.003	0.014
Ni	60	0.05 ±0.04	0.05
Cu	63	2.84 ±0.45	2.81
Zn	66	142 ±14	140
As	75	0.009 ±0.003	0.011
Se	78	0.076 ±0.010	0.11
Sr	88	0.052 ±0.015	0.081
Mo	98	0.08 ±0.06	0.08
Cd	111	0.013 ±0.011	0.013
Sn	118	–	0.14
Sb	121	(0.01)	0.01
Ba	137	(0.05)	0.04
Hg	202	0.005 ±0.003	0.003
Pb	208	0.38 ±0.24	0.34
Tl	205	–	0.002
Th	232	–	<0.00008
U	238	–	<0.00002

Table 4. Analysis of NIST® 2976 Mussel Tissue using the NexION 300/350 ICP-MS.

Element	Mass (amu)	Reference Value (mg/kg)	Experimental Value (mg/kg)
B	11	–	27.5
Na	23	(35000 ±1000)	35000
Mg	26	(5300 ±500)	4800
Al	27	(134 ±34)	149
P	31	(8300)	6900
S	34	(19000)	16000
K	39	(9700 ±500)	9700
Ca	44	(7600 ±300)	7400
V	51	–	0.87
Cr	52	(0.50 ±0.16)	0.50
Fe	54	171.0 ±4.9	190
Mn	55	(33 ±2)	40
Co	59	(0.61 ±0.02)	0.67
Ni	60	(0.93 ±0.12)	0.87
Cu	63	4.02 ±0.33	3.91
Zn	66	137 ±13	145
As	75	13.3±1.8	16.4
Se	78	1.80 ±0.15	2.52
Sr	88	(93 ±2)	79
Mo	98	–	0.56
Cd	111	0.82 ±0.16	0.88
Sn	118	(0.096 ±0.039)	0.103
Sb	121	–	0.011
Ba	137	–	0.61
Hg	202	0.061 ±0.0036	0.058
Pb	208	1.19 ±0.18	1.06
Tl	205	(0.0013)	0.003
Th	232	(0.011 ±0.002)	0.012
U	238	–	0.22

Conclusion

This work has demonstrated the ability of PerkinElmer's NexION 300/350X ICP-MS to effectively measure macro-level nutritional elements in the same analysis run as lower-level elements, without having to dilute the samples. The agreement between experimental and certified results for NIST® 8414 Bovine Muscle and NIST® 2976 Mussel Tissue demonstrates the accuracy of the analysis. Instrument design characteristics eliminate deposition on the ion optics, leading to long-term stability in high-matrix samples, while permitting trace levels to be accurately measured.

References

1. "The Determination of Toxic, Essential, and Nutritional Elements in Food Matrices Using the NexION 300/350 ICP-MS", PerkinElmer Application Note.

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