Simple, Rapid Analysis of Trace Metals in Foods Using the Agilent 7700x ICP-MS

Application Note

Foods

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Introduction

The task of efficiently monitoring chemical and biological contaminants in imported and exported food can be overwhelming. Traditionally, analysis of metals in foods has required multiple techniques in order to cover the range of elements, concentrations and food types. This approach is slow and expensive, so a more rapid, sensitive, and cost-effective screening test is necessary. The Agilent 7700x ICP-MS is capable of accurately analyzing a variety of foods for metals at trace and major levels using a single collision cell method. This method is simple to set up and operate routinely, and permits large numbers of samples to be quickly screened for total toxic metals. Samples which are found to contain metals where the potential toxicity is dependent on the chemical form can then be further analyzed for species composition as needed, using Agilent-supported hyphenated ICP-MS techniques such as LC-ICP-MS or GC-ICP-MS.

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Experimental

In order to test the ability of the Agilent 7700x to analyze a variety of foods for a wide range of metals at highly variable concentrations, several certified reference food samples were analyzed. The 7700x was tuned using One-click Plasma setting for robust plasma conditions and autotuned for optimum sensitivity, mass response and minimal interferences. Operating conditions are shown in Table 1. In order to keep the method as quick and simple as possible, the Octopole Reaction System (ORS^3) was operated in a single mode, using helium (He) cell gas, which provides a reliable and effective cell method to remove all polyatomic interferences, regardless of the analyte or matrix composition. The following acquisition masses and integration times (Table 2) provided more than sufficient sensitivity to meet all certified values. Total run time per sample was less than 3 minutes.

Table 1. 7700 Autotuning Conditions

	Parameter	Value
Set by One-click Plasma Setting	RF power (W)	1550
	Carrier gas flow (L/min)	0.99
	Spray chamber temp (°C)	2
	Sample depth (mm)	8
	Extract 1 lens (V)	0
Set by Autotune	CeO^+/Ce^+ (%)	1.114
	Ce^{++}/Ce^+ (%)	1.867
	Sensitivity cps/ppb	Li (62700), Y (92920), Tl (87080)

Traditionally, covering this range of concentrations for these elements would have required ICP-OES for the major elements (Na and Ca), graphite furnace AA for Pb and Cd, either a dedicated Hg analyzer or cold vapor AA for Hg and possibly hydride AA for As and Se. The Agilent 7700x ICP-MS running in He mode was able to measure all elements in a single run easily. Even elements such as Be and Hg, which would typically be acquired under no gas conditions when using ICP-MS, demonstrated excellent sensitivity in He mode (Be DL = 28 ppt, Hg DL = 1.6 ppt).

Table 2. List of Analytes and Acquisition Times. All Elements Were Acquired in He Mode.

Mass	Element	Integration time per mass (sec)	Replicates	
6	Li	0.3	3	Internal standard
9	Be	0.99	3	
23	Na	0.3	3	
40	Ca	0.3	3	
43	Ca	0.3	3	
45	Sc	0.3	3	Internal standard
51	V	0.3	3	
52- 53	Cr	0.3	3	
55	Mn	0.3	3	
56	Fe	0.3	3	
60	Ni	0.99	3	
63	Cu	0.3	3	
66	Zn	0.3	3	
72-74	Ge	0.3	3	Internal standard
75	As	0.99	3	
77- 78, 82	Se	0.99	3	
95	Mo	0.99	3	
111	Cd	0.3	3	
115	In	0.3	3	Internal standard
121	Sb	0.99	3	
137	Ba	0.3	3	
159	Tb	0.3	3	Internal standard
202	Hg	0.99	3	
205	Tl	0.99	3	
208	Pb	0.3	3	
209	Bi	0.3	3	Internal standard
238	U	0.99	3	

Example calibration curves for several critical and difficult elements are shown in Figure 1.

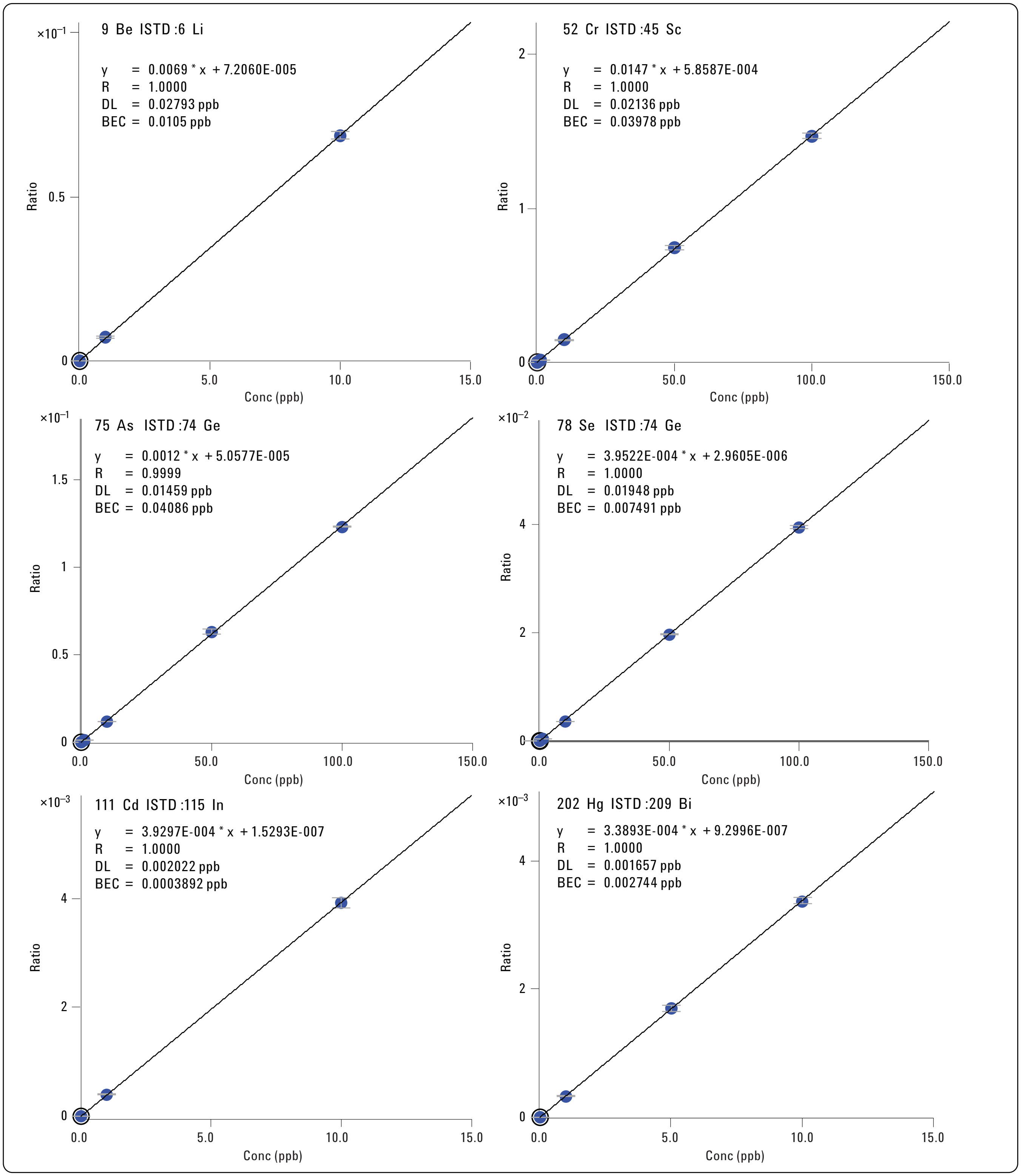

Figure 1. Calibrations for Be, Cr, As, Se Cd and Hg.

The food certified reference materials were analyzed directly after microwave digestion. Between 0.5 g and 1 g of each sample was weighed (after determination of percent moisture) and digested using 6 mL of HNO_3 + 2 mL of H_2O_2 using microwave assisted digestion. All samples were brought to final volume of 100 mL using ultrapure water. Results are shown in Table 3. The trace elements, Ni, Mn, Cu, As, Se, Cd, Hg and Pb exhibited excellent agreement with the certified values for all three samples. Slight deviations from certified values for Fe, Ca and Zn were attributed to the digestion procedure rather than the analytical measurement.

Conclusions

Using a simple procedure based on microwave digestion and single He mode ICP-MS analysis, typical food samples can be quickly and accurately analyzed for trace and major element concentrations without the need for multiple sample preparations and analytical techniques. The Agilent 7700x using He mode alone can provide sensitive, accurate, interference-free analysis of a variety of metals in common foods. Because He mode is both sensitive and universal, it is applicable to trace analysis of all metals in any food sample digest. No prior information about the sample matrix or analyte elements present is required, as He mode removes all polyatomic interferences, regardless of the sample matrix.

Table 3. Measured and Certified Values for Three Certified Reference Food Materials. Recoveries are Dependent on Digestion Efficiency as Well as Analytical Accuracy. All Measured Values are Based on Dry Sample Weight Corrected for Percent Moisture. All Certified Elements are Reported for Each Sample, not all Samples are Certified for all Elements.

	NRC-CNRC DORM3 Fish Protein		**NIST SRM 2976 Mussel Tissue**		**NIST RM 8415 Whole Egg Powder**	
Mass/element	**Certified value (mg/kg)**	**Measured (mg/kg)**	**Certified value (mg/kg)**	**Measured (mg/kg)**	**Certified value (mg/kg)**	**Measured (mg/kg)**
23 Na	–	–	–	–	3770 ± 340	3807
43 Ca	–	–	–	–	2480 ± 190	2703
52 Cr	–	–	–	–	0.37 ± 0.18	0.344
55 Mn	–	–	–	–	1.78 ± 0.38	1.64
56 Fe	347 ± 20	324.0	171 ± 4.9	158.5	–	
60 Ni	1.28 ± 0.24	1.29	–		–	
63 Cu	15.5 ± 0.63	14.4	4.02 ± 0.33	3.32	2.7 ± 0.35	2.61
66 Zn	51.3 ± 3.1	45.86	137 ± 13	121.2	–	
75 As	6.88 ± 0.3	6.15	13.3 ± 1.8	12.57	–	
78 Se	–		1.8 ± 0.15	1.87	1.39 ± 0.17	1.25
95 Mo	–		–		0.247 ± 0.023	0.215
111 Cd	0.29 ± 0.02	0.28	0.82 ± 0.16	0.794	–	
202 Hg	0.355 ± 0.056	0.359	0.061 ± 0.0036	0.068	–	
208 Pb	0.395 ± 0.050	0.398	1.19 ± 0.18	1.163	0.061 ± 0.012	0.055

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