

# Benefits of the Agilent 8900 ICP-QQQ with MS/MS operation for routine food analysis

Application note

Food safety

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## Introduction

Growing awareness of and concern about the issue of food safety is reflected in the tightening of regulations governing toxic elements and compounds in food. Many toxic elements such as As, Hg, Cd, Pb etc. are routinely monitored to ensure food safety, while minerals that are beneficial/essential to human health such as Se, Na, Mg, K, Ca, etc., are also measured.

As a fast, high throughput, multi-element technique, with a wide dynamic range and high sensitivity, ICP-MS is increasingly used for routine food analysis. Recent improvements in matrix tolerance with Agilent's High



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Matrix Introduction (HMI/UHMI) technology is a further benefit for the application as food matrices are varied and can be complex. UHMI uses aerosol dilution to reduce the sample matrix load on the plasma, allowing matrix levels up to several percent total dissolved solids (TDS) to be analyzed routinely. This is much higher than the limit of 0.2% (2000 ppm) which has traditionally applied to samples intended for ICP-MS analysis.

Control of polyatomic ion interferences in quadrupole ICP-MS has also improved significantly with the development of collision/reaction cells (CRCs), which use kinetic energy discrimination (KED) to attenuate polyatomic ions in helium (He) collision mode. Agilent's octopole-based CRC, the ORS<sup>4</sup>, is routinely used to suppress a wide range of matrix-based polyatomic ion interferences under one set of cell conditions [1]. Hence, reliable and accurate quantification of all required elements at regulated levels in a variety of sample matrices is now possible using conventional quadrupole ICP-MS (ICP-QMS).

However, some food-analysis applications require greater sensitivity for specific elements, while some complex sample matrices may cause spectral interferences that remain a challenge for ICP-QMS. For example, doubly charged ions of some rare earth elements (REEs) appear at the same mass as key analytes, hindering accurate low-level measurement of arsenic (As) and selenium (Se) in some sample types [2, 3].

#### **Improved interference removal with ICP-QQQ**

The Agilent 8900 Triple Quadrupole ICP-MS (ICP-QQQ) has a unique tandem MS configuration, comprising two scanning quadrupole mass analyzers either side of an octopole-based ORS<sup>4</sup> collision reaction cell. As a result, the 8900 ICP-QQQ is able to utilize reactive cell gases and ion/molecule reaction chemistry in combination with MS/MS mode to resolve difficult spectral interferences [4]. The superior interference removal offered by reaction chemistry with MS/MS led to the previous generation Agilent 8800 ICP-QQQ being widely accepted in industry and research labs in fields such as semiconductor device and high purity chemical/material manufacturing, life-science, geoscience, radionuclides and many others [5-8]. MS/MS mode is also beneficial for the analysis of certain elements which are subject to problematic interferences in routine applications, such

as the analysis of food samples, soils, waste water and groundwater. Since the matrix tolerance and robustness of the Agilent 8900 ICP-QQQ is comparable to Agilent's market-leading single quadrupole ICP-MS systems, the 8900 ICP-QQQ can be used to analyze these high-matrix samples routinely.

#### **Solving problems associated with As and Se analysis**

Arsenic is a well-known toxic element, while Se is an essential element that can be toxic in excess. Consequently, many countries regulate the permitted concentrations of As and Se in food, animal feed, drinking water, surface water and soils. However, As and Se can suffer spectral interferences from polyatomic ions including ArCl<sup>+</sup>, CaCl<sup>+</sup>, ArAr<sup>+</sup>, S<sub>2</sub>O<sup>+</sup>, SO<sub>3</sub><sup>+</sup>, GeH<sup>+</sup>, and BrH<sup>+</sup>. These interferences can be reduced using ICP-QMS operating in helium (He) cell mode, allowing the accurate and precise measurement of As and Se at the concentration levels required to meet typical regulatory demands.

However, He mode is not effective against doubly-charged ion overlaps. The lanthanides or rare earth elements (REE) can form doubly charged ions (REE<sup>++</sup>) which overlap As and Se. These doubly-charged overlaps can be avoided using mass-shift mode with O<sub>2</sub> as the reaction cell gas. In this mode, the analytes are measured as reaction product ions <sup>75</sup>As<sup>16</sup>O<sup>+</sup> and <sup>78</sup>Se<sup>16</sup>O<sup>+</sup>, mass-shifted to *m/z* 91 and 94 respectively, where they are free from the original REE<sup>++</sup> overlaps. This reaction chemistry can be used in the CRC of an ICP-QMS, but existing ions from the plasma may overlap the newly-formed product ions; e.g. <sup>91</sup>Zr<sup>+</sup> on <sup>75</sup>As<sup>16</sup>O<sup>+</sup>, and <sup>94</sup>Mo<sup>+</sup> on <sup>78</sup>Se<sup>16</sup>O<sup>+</sup>. To ensure controlled and consistent reaction chemistry, MS/MS mode on an ICP-QQQ is required, where the first quadrupole (Q1) operates as a mass filter set to the appropriate As<sup>+</sup> or Se<sup>+</sup> precursor ion mass. Q1 rejects all other masses, thereby removing the existing Zr<sup>+</sup> and Mo<sup>+</sup> ions and preventing them from overlapping the new analyte product ions.

Typically, the REE content of food and other natural samples is low, but crops grown in REE-enriched soils can take up high concentrations of these elements. The use of MS/MS mode with O<sub>2</sub> reaction cell gas avoids the potential risk of reporting incorrect results for As and Se in the case of an unexpectedly high level of REEs.

In this study, the Agilent 8900 ICP-QQQ was evaluated as a routine tool for the analysis of 30 elements, including As and Se, in food sample digests.

## Experimental

### Certified Reference Materials (CRMs)

Five food CRMs purchased from National Institute of Standards and Technology (NIST) and High-Purity Standards Inc. (Charleston, SC, USA) were analyzed in this study. The CRMs used were NIST 1567b Wheat Flour, NIST 1568b Rice Flour, NIST 1515 Apple Leaves, NIST 1573a Tomato Leaves and High Purity Standards Mixed Food Diet Solution.

### Sample preparation

Due to the requirement to measure several volatile elements, including Hg, closed vessel microwave digestion using a Milestone ETHOS 1 Advanced Microwave Digestion System was used to digest the food CRMs. Sample weights of approximately 1.0 g for each of the flour CRMs (NIST 1567b, NIST 1568b) and 0.5 g for each of the other sample types (NIST 1515, NIST 1573a) were accurately weighed into closed microwave vessels. 6 mL of HNO<sub>3</sub> and 1 mL of HCl (electronics (EL) grade acids, Kanto Chemicals) were added to the microwave vessels. After 15 minutes held at room temperature, microwave heating was applied, using the heating program shown in Table 1. All CRMs were completely dissolved, resulting in clear solutions which were diluted to a final volume of 100 mL with ultrapure water (Merck, Darmstadt, Germany).

**Table 1.** Microwave digestion heating programs for four CRM food samples.

Power (W)	Temp (°C)	Ramp (min)	Hold (min)
500	70	2	3
1000	140	5	5
1000	200	5	15
Ventilation			30

It is well known that carbon present in the sample solution enhances the ICP-MS signal of some elements, notably As, Se and P, although the precise mechanism of the enhancement is not clearly understood [9, 10]. With the high digestion temperature used in this work (200 °C), the carbon matrix was effectively decomposed during digestion. The effect of any residual carbon in the samples was mitigated by ensuring an excess of carbon was present in all samples and standards, by adding 2% butan-1-ol online with the internal standard solution.

### Instrumentation

An Agilent 8900 ICP-QQQ (Standard configuration) with the standard sample introduction system consisting of a glass concentric nebulizer, quartz spray chamber, and Ni interface cones was used. UHMI technology is included on the 8900 ICP-QQQ Standard configuration, allowing matrices as high as 25% NaCl solution to be analyzed [11]. The plasma conditions were selected according to the sample type and expected matrix level using the “Preset plasma” function of the MassHunter software.

### Acquisition conditions

For the multi-element analysis of the food samples, a multi-tune method was used so all elements could be acquired in the optimum cell gas mode. Multi-tune permits samples to be automatically analyzed using the optimum tune and cell conditions for each analyte element. He mode was used for all elements except P, S, As and Se which were determined in mass-shift mode using O<sub>2</sub> cell gas. The method was based on an appropriate preset method for food samples, which was modified to include O<sub>2</sub> cell gas mode. Preset plasma condition “UHMI-4” was selected, where the number 4 represents the approximate aerosol dilution factor. The UHMI setting automatically applies the predefined and calibrated parameters for RF power, sampling depth, carrier gas flow rate and dilution gas flow rate, giving precise and reproducible plasma conditions for the target sample types. The lens voltages were auto-tuned for maximum sensitivity. Table 2 summarizes the instrument operating parameters.

**Table 2.** Agilent 8900 ICP-QQQ operating conditions.

Parameter	Setting	
Cell mode	He mode	O <sub>2</sub> mode
Scan type	Single Quad	MS/MS
Plasma conditions	UHMI-4	
RF power (W)	1600	
Sampling depth (mm)	10	
Carrier gas flow rate (L/min)	0.77	
Dilution gas flow rate (L/min)	0.15	
Extract 1 (V)	0	
Extract 2 (V)	-250	
Omega bias (V)	-140	
Omega lens (V)	8.8	
Cell gas flow (mL/min)	5.5	0.3 (20% of full scale)
KED (V)	5	-7

Shaded parameters are predefined by selecting preset plasma condition UHMI-4.

### Calibration standards and internal standards

Calibration standards were prepared from an Agilent multi-element environmental calibration standard (p/n 5183-4688) which contains 1000 ppm each of Fe, K, Ca, Na, Mg and 10 ppm each of Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Th, Ti, U, V, and Zn. Standards for B, Rb, Sr, Sn and Hg were prepared from 1000 ppm Atomic-Absorption grade single element standards from Kanto Chemicals (Tokyo, Japan). S and P were prepared from 10,000 ppm Spex single element standards (SPEX CertiPrep, NJ, USA). The internal standard (ISTD) solution was prepared from an Agilent internal standard stock solution for ICP-MS systems (p/n 5188-6525) containing 6-Li, Sc, Ge, Rh, In, Tb, Lu, and Bi. Ir was added from an Atomic-Absorption grade single element standard purchased from Kanto Chemicals. The ISTD was added to the sample using the standard online ISTD kit.

Calibration standards were prepared in 6% HNO<sub>3</sub> and 1% HCl to match the acid content of the sample solutions. The ISTDs were prepared in 1% HNO<sub>3</sub> and 0.5% HCl. The calibration ranges were as follows: major elements: 0-100 ppm, trace elements: 0-500 ppb, B: 0-200 ppb, Hg: 0-1 ppb and Sn: 0-2 ppb.

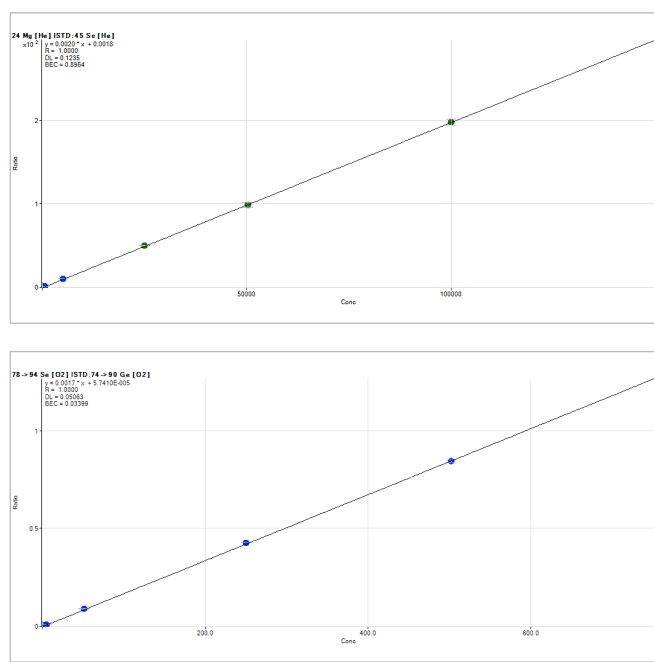


Figure 1. Representative calibration curves for a major element (Mg) and a trace element (Se).

### Sequence of calibrants, samples, and QC solutions

The sequence consisted of an initial multi-level calibration, covering the typical range for the target analytes, followed by a QC block containing an Initial Calibration Blank (ICB) check and Initial Calibration Verification (ICV) solution. After calibration and initial QC check, twelve sample blocks were analyzed per the flow chart shown in Figure 2; each block consisted of 2 preparation blanks and 10 samples (2 each of Wheat Flour, Rice Flour, Apple Leaves, Tomato Leaves and Mixed Food Diet). A Periodic Block consisting of Continuing Calibration Blank (CCB) and Continuing Calibration Verification (CCV) samples was automatically inserted into the sequence after each sample block.

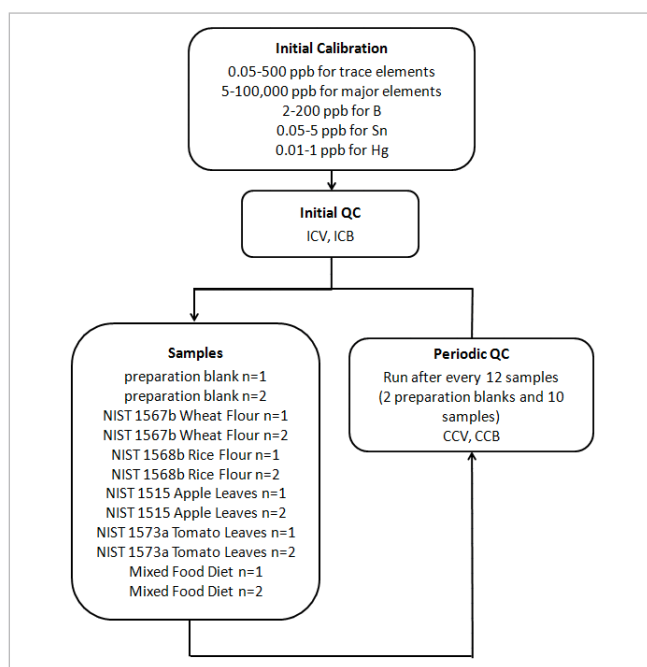
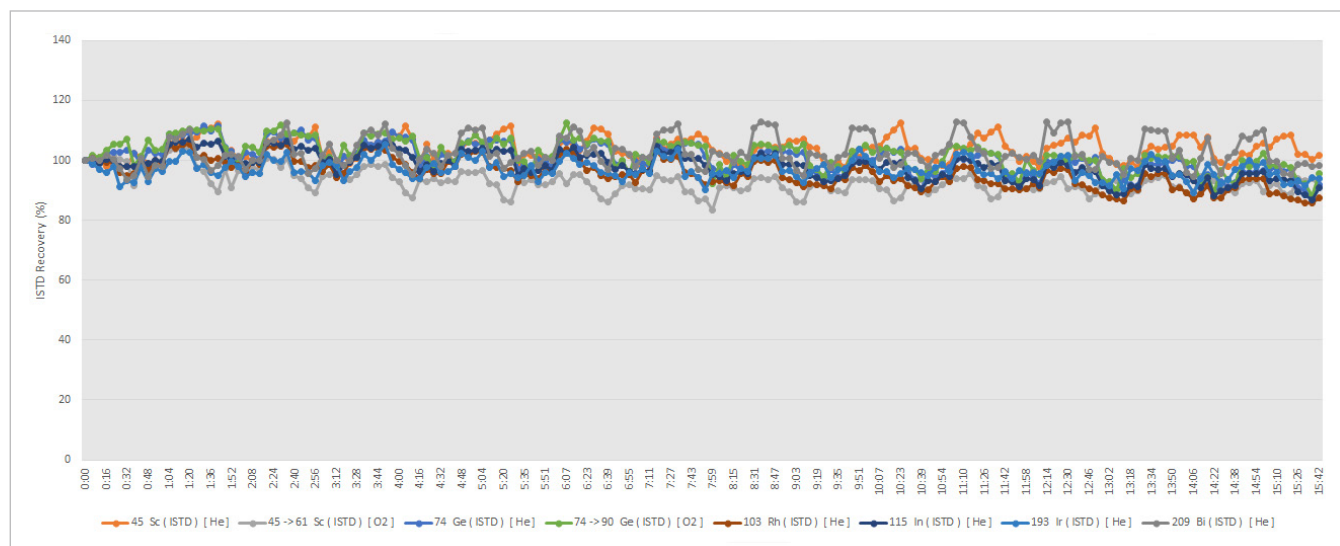


Figure 2. Sequence of calibrants, samples, and QC solutions analyzed in a single 15-hour sequence. Sample Block was repeated continuously with automatic insertion of Periodic QC Block after each Sample Block.

The total number of analyses of calibration standards, QC samples and food digest samples was 183 over ~15 hours. The sample-to-sample run time was about 5 minutes, which included a 10 s probe rinse and 60 s sample introduction system rinse at 0.3 rps peristaltic pump rate. Table 3 shows the detection limits (DL) obtained using this method.

**Table 3.** Method detection limits.

Element	Scan Mode	Q1	Q2	DL (ppb)	Element	Scan Mode	Q1	Q2	DL (ppb)
B	Single Quad		11	0.3653	Se	Single Quad		78	0.3158
Na	Single Quad		23	0.1945	Se	MS/MS	78	94	0.0506
Mg	Single Quad		24	0.1235	Rb	Single Quad		85	0.0115
Al	Single Quad		27	0.1847	Sr	Single Quad		88	0.0006
P	MS/MS	31	47	0.0919	Mo	Single Quad		95	0.0090
S	MS/MS	32	48	0.4367	Ag	Single Quad		107	0.0063
K	Single Quad		39	7.0656	Cd	Single Quad		111	0.0018
Ca	Single Quad		44	8.7579	Sn	Single Quad		118	0.0074
V	Single Quad		51	0.0079	Sb	Single Quad		121	0.0026
Cr	Single Quad		52	0.0880	Ba	Single Quad		138	0.0008
Mn	Single Quad		55	0.0099	Hg	Single Quad		202	0.0005
Fe	Single Quad		56	0.1595	Tl	Single Quad		205	0.0104
Co	Single Quad		59	0.0009	Pb	Single Quad		208	0.0016
Ni	Single Quad		60	0.0484	Th	Single Quad		232	0.0018
Cu	Single Quad		63	0.0102	U	Single Quad		238	0.0009
Zn	Single Quad		66	0.0308					
As	Single Quad		75	0.0044					
As	MS/MS	75	91	0.0040					



**Figure 3.** ISTD signal stability for the sequence of 183 samples analyzed over 15 hours

## Results and discussion

### ISTD and CCV stability

Figure 3 shows the ISTD signal stability for the sequence of 183 samples analyzed over 15 hours. The ISTD recoveries for all samples were well within  $\pm 20\%$  of the value in the initial calibration standard. These ISTD recoveries are comparable to the results obtained routinely using ICP-QMS, demonstrating the equivalent robustness of the 8900 ICP-QQQ.

The midpoint of the calibration standards was used as the CCV solution. CCV recovery over the 15-hour analysis was stable and within  $\pm 10\%$  for all elements, as shown in Figure 4, again demonstrating that the 8900 ICP-QQQ has the high matrix tolerance required for routine food digest analysis.

### CRM recovery results

The accuracy of the method was evaluated by analyzing the five food CRMs as unknown samples. Each CRM was measured 24 times in the batch. The mean concentration and relative standard deviation (%RSD) were calculated for each element and compared to the certified value, as shown in Tables 4 to 8. Using the preferred measurement mode, the results for all elements were in good agreement with the certified and reference values. Results are shown for both He mode and O<sub>2</sub> mass-shift mode for As and Se, to compare the results for samples where a sample might contain an unexpected high level of REEs. NIST 1515 Apple Leaves CRM contains low  $\mu\text{g}/\text{kg}$  concentrations of As and Se (Table 6) and high concentrations of REEs. Reference (non-certified) values for Nd, Sm, and Gd are 17, 3, and 3 mg/kg, respectively. In the case of Apple Leaves and, to a lesser extent, Tomato Leaves, more accurate recovery was obtained for As and Se using O<sub>2</sub> mass shift mode, illustrating the potential error that can be caused by the relatively high level of REE in these two reference materials.

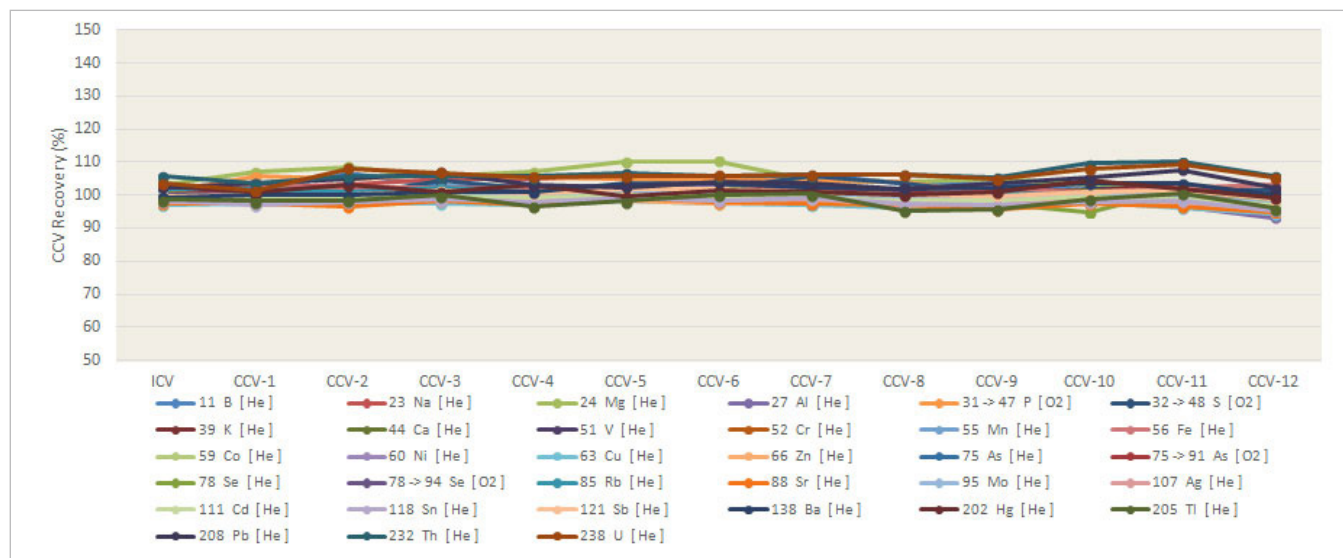


Figure 4. CCV recovery for all elements over the 15-hour analysis

**Table 4.** Results for NIST 1567b Wheat Flour, n = 24

Element	Measured Solution Concentration (µg/L)	RSD (%)	Calculated Sample Concentration (mg/kg)			Certified Concentration (mg/kg)			Recovery (%)
				±			±		
23 Na	65.2	2.3	6.50	±	0.15	6.71	±	0.21	97
24 Mg	3842	1.6	383	±	6	398	±	12	96
27 Al	39	2.8	3.9	±	0.1	4.4	±	1.2	88
31 -> 47 P	12936	2.0	1291	±	26	1333	±	36	97
32 -> 48 S	15496	2.2	1546	±	34	1645	±	25	94
39 K	12700	2.3	1267	±	29	1325	±	20	96
44 Ca	1871	1.8	186.7	±	3.4	191.4	±	3.3	98
51 V	0.10	8.1	0.010	±	0.001	0.01*			100
55 Mn	86	1.7	8.54	±	0.14	9.00	±	0.78	95
56 Fe	142	1.6	14.20	±	0.22	14.11	±	0.33	101
63 Cu	19	1.6	1.94	±	0.03	2.03	±	0.14	96
66 Zn	112	1.9	11.17	±	0.21	11.61	±	0.26	96
75 As	0.047	16.5	0.0046	±	0.001	0.0048	±	0.0003	97
75 -> 91 As	0.049	19.4	0.0049	±	0.001	0.0048	±	0.0003	101
78 Se	11.5	4.2	1.15	±	0.05	1.14	±	0.10	101
78 -> 94 Se	11.8	1.9	1.17	±	0.02	1.14	±	0.10	103
85 Rb	6.54	1.8	0.652	±	0.012	0.671	±	0.012	97
95 Mo	4.60	2.1	0.459	±	0.009	0.464	±	0.034	99
111 Cd	0.239	5.7	0.0238	±	0.0014	0.0254	±	0.0009	94
118 Sn	0.0355	12.8	0.0035	±	0.0005	0.003*			118
202 Hg	0.0066	11.3	0.0007	±	0.0001	0.0005*			131
208 Pb	0.0937	4.4	0.0094	±	0.0004	0.0104	±	0.0024	90

\* Reference value

**Table 5.** Results for NIST 1568b Rice Flour, n = 24

Element	Measured Solution Concentration (µg/L)	RSD (%)	Calculated Sample Concentration (mg/kg)			Certified Concentration (mg/kg)			Recovery (%)
				±			±		
23 Na	65.6	3.2	6.54	±	0.28	6.74	±	0.19	97
24 Mg	5454	1.5	543	±	8	559	±	10	97
27 Al	40.3	3.3	4.01	±	0.13	4.21	±	0.34	95
31 -> 47 P	15162	2.8	1510	±	43	1530	±	40	99
32 -> 48 S	11369	2.5	1133	±	28	1200	±	10	94
39 K	12371	2.0	1233	±	24	1282	±	11	96
44 Ca	1158	2.1	115.3	±	2.5	118.4	±	3.1	97
51 V	182.3	1.0	18.2	±	0.2	19.2	±	1.8	95
55 Mn	75.4	1.0	7.51	±	0.08	7.42	±	0.44	101
56 Fe	0.173	1.7	0.0173	±	0.0003	0.0177	±	0.0005*	98
63 Cu	22.7	1.0	2.26	±	0.02	2.35	±	0.16	96
66 Zn	191.7	1.4	19.10	±	0.26	19.42	±	0.26	98
75 As	2.97	1.4	0.296	±	0.004	0.285	±	0.014	104
75 -> 91 As	3.01	1.7	0.300	±	0.005	0.285	±	0.014	105

Table 5 continued overleaf

78 Se	3.4	8.9	0.341	±	0.030	0.365	±	0.029	93
78 -> 94 Se	3.5	3.8	0.352	±	0.013	0.365	±	0.029	96
85 Rb	61.1	1.1	6.088	±	0.069	6.198	±	0.026	98
95 Mo	13.96	1.2	1.391	±	0.017	1.451	±	0.048	96
111 Cd	0.201	4.9	0.0201	±	0.0010	0.0224	±	0.0013	90
118 Sn	0.060	7.4	0.0060	±	0.0004	0.005	±	0.001*	121
202 Hg	0.0529	2.1	0.0053	±	0.0001	0.0059	±	0.0004	89
208 Pb	0.068	3.0	0.0068	±	0.0002	0.008	±	0.003*	85

\*Reference value

**Table 6.** Results for NIST 1515 Apple Leaves, n = 24

Element	Measured Solution Concentration (µg/L)	RSD (%)	Calculated Sample Concentration (mg/kg)			Certified Concentration (mg/kg)			Recovery (%)
				±			±		
11 B	141	2.9	28	±	0.8	27	±	2	104
23 Na	196	1.6	39.1	±	0.6	24.4	±	1.2	160* <sup>1</sup>
24 Mg	14083	1.3	2812	±	36	2710	±	80	104
27 Al	1458	1.6	291	±	5	286	±	9	102
31 -> 47 P	8088	2.2	1615	±	35	1590*			102
32 -> 48 S	9211	1.4	1839	±	26	1800*			102
39 K	80429	2.2	16057	±	361	16100	±	200	100
44 Ca	74060	1.2	14786	±	172	15260	±	1500	97
51 V	1.20	2.8	0.24	±	0.01	0.26	±	0.03	92
52 Cr	1.3	1.4	0.25	±	0.00	0.3*			85
55 Mn	265	1.0	53	±	1	54	±	3	98
56 Fe	379	0.8	76	±	1	80*			95
59 Co	0.44	1.5	0.088	±	0.001	0.09*			98
60 Ni	4.4	1.7	0.88	±	0.02	0.91	±	0.12	97
63 Cu	28.2	1.0	5.62	±	0.06	5.64	±	0.24	100
66 Zn	60.3	0.9	12.0	±	0.1	12.5	±	0.3	96
75 As	2.0	1.2	0.395	±	0.005	0.038	±	0.007	1040
75 -> 91 As	0.2	3.7	0.036	±	0.001	0.038	±	0.007	94
78 Se	13.43	5.8	2.7	±	0.2	0.050	±	0.009	5364
78 -> 94 Se	0.271	13.8	0.054	±	0.008	0.050	±	0.009	108
85 Rb	46.3	0.9	9.2	±	0.1	9*			103
88 Sr	123.0	1.0	25	±	0	25	±	2	98
95 Mo	0.44	5.3	0.088	±	0.005	0.094	±	0.013	94
111 Cd	0.06	7.0	0.013	±	0.001	0.014*			91
121 Sb	0.06	4.6	0.011	±	0.001	0.013*			85
138 Ba	245	1.9	49	±	1	49	±	2	100
202 Hg	0.21	2.0	0.041	±	0.001	0.044	±	0.004	93
208 Pb	2.3	1.3	0.452	±	0.006	0.470	±	0.024	96
232 Th	0.14	2.2	0.028	±	0.001	0.03*			93
238 U	0.034	3.7	0.0068	±	0.0003	0.006*			113

\*Reference value.

Shaded values for As and Se were obtained in single quad mode with He cell gas. The accurate results obtained using MS/MS mode with O<sub>2</sub> mass-shift are shown in the lines below.

<sup>1</sup>The measured Na result was high compared to the reference value; the same result was obtained from a repeated analysis of the same solution, so a spike recovery test was performed for confirmation. The spike recovery result was good (recovery: 99%), suggesting that the original sample had suffered Na contamination.



**Table 7.** Results for NIST 1573a Tomato Leaves, n = 24

Element	Measured Solution Concentration (µg/L)	RSD (%)	Calculated Sample Concentration (mg/kg)			Certified Concentration (mg/kg)			Recovery (%)
				±			±		
11 B	167	1.9	33.3	±	0.6	33.3	±	0.7	100
23 Na	613	2.5	122	±	3	136	±	4	90
24 Mg	57311	2.0	11412	±	225	12000*			95
27 Al	2573	2.4	512	±	12	598	±	12	86
31 -> 47 P	10928	2.7	2176	±	59	2160	±	40	101
32 -> 48 S	48387	1.4	9635	±	131	9600*			100
39 K	134250	2.2	26732	±	591	27000	±	500	99
44 Ca	243939	1.4	48574	±	671	50500	±	900	96
51 V	4.0	2.2	0.792	±	0.017	0.835	±	0.010	95
52 Cr	9.3	1.6	1.85	±	0.03	1.99	±	0.06	93
55 Mn	1236.5	1.5	246	±	4	246	±	8	100
56 Fe	1843.3	1.7	367	±	6	368	±	7	100
59 Co	2.8	1.4	0.55	±	0.01	0.57	±	0.02	96
60 Ni	7.9	1.9	1.56	±	0.03	1.59	±	0.07	98
63 Cu	23.7	1.5	4.71	±	0.07	4.70	±	0.14	100
66 Zn	149.4	1.5	29.8	±	0.5	30.9	±	0.7	96
75 As	0.7	2.3	0.141	±	0.003	0.112	±	0.004	126
75 -> 91 As	0.6	1.7	0.112	±	0.002	0.112	±	0.004	100
78 Se	1.03	15.6	0.205	±	0.032	0.054	±	0.003	380
78 -> 94 Se	0.31	11.2	0.061	±	0.007	0.054	±	0.003	113
85 Rb	69.7	1.2	13.88	±	0.16	14.89	±	0.27	93
88 Sr	421.0	1.3	84	±	1	85*			99
95 Mo	2.1	2.8	0.42	±	0.01	0.46*			91
107 Ag	0.09	9.1	0.018	±	0.002	0.017*			104
111 Cd	7.4	1.4	1.47	±	0.02	1.52	±	0.04	97
121 Sb	0.28	3.4	0.055	±	0.002	0.063	±	0.006	88
138 Ba	302.8	2.1	60.3	±	1.3	63*			96
202 Hg	0.15	2.4	0.030	±	0.001	0.034	±	0.004	88
232 Th	0.52	2.1	0.104	±	0.002	0.12*			87
238 U	0.14	2.3	0.029	±	0.001	0.035*			81

\*Reference value.

Shaded values for As and Se were obtained in single quad mode with He cell gas. The accurate results obtained using MS/MS mode with O<sub>2</sub> mass-shift are shown in the lines below.

**Table 8.** Results for the High Purity Standard Mixed Food Diet Solution, n = 24

Element	Measured Solution Concentration (µg/L)	RSD (%)	Calculated Sample Concentration (mg/kg)			Certified Concentration (mg/kg)			Recovery (%)
				±			±		
23 Na	15808	2.9	61.8	±	1.8	60.0	±	0.6	105
24 Mg	3300	2.3	12.9	±	0.3	12.0	±	0.1	108
27 Al	26	4.5	0.100	±	0.005	0.100	±	0.002	100
31 -> 47 P	15543	3.3	60.8	±	2.0	60.0	±	0.6	101
39 K	41898	2.2	164	±	4	160	±	2	102
44 Ca	9800	2.7	38.3	±	1.0	40.0	±	0.4	96
52 Cr	0.55	10.4	0.0021	±	0.0002	0.002*			107
55 Mn	49.2	1.7	0.192	±	0.003	0.200	±	0.004	96
56 Fe	204.5	1.8	0.80	±	0.01	0.80	±	0.01	100
59 Co	0.2	2.4	0.0008	±	0.0000	0.0008*			98
60 Ni	5.1	2.5	0.020	±	0.001	0.020	±	0.001	99
63 Cu	15.3	1.7	0.060	±	0.001	0.060	±	0.006	100
66 Zn	74.5	2.0	0.29	±	0.01	0.30	±	0.01	97
75 As	5.1	2.0	0.020	±	0.000	0.020	±	0.001	99
75 -> 91 As	5.2	2.6	0.020	±	0.001	0.020	±	0.001	102
78 Se	1.26	14.8	0.0049	±	0.0007	0.005*			99
78 -> 94 Se	1.31	6.6	0.0051	±	0.0003	0.005*			102
95 Mo	1.5	3.1	0.0059	±	0.0002	0.006*			98
111 Cd	2.0	2.1	0.0078	±	0.0002	0.0080	±	0.0008	98

\*Reference value.

## Conclusions

The Agilent 8900 Standard configuration ICP-QQQ with UHMI offers the robustness and matrix tolerance required for the routine analysis of the widest range of trace and major elements in high matrix samples, such as food digest samples. Doubly-charged REE interferences that can affect the accurate measurement of arsenic and selenium at trace levels were avoided using O<sub>2</sub> cell gas with MS/MS mass-shift mode. Most other elements were measured in He mode; a field-proven method that is widely used to remove common matrix-based polyatomic interferences in complex and variable matrices.

While not all food products, soils and sediments contain significant concentrations of REEs, the use of ICP-QQQ with MS/MS improves the accuracy and confidence in the results for As and Se measured in food and environmental samples that often contain complex, variable, high TDS matrices.

Method development was greatly simplified with the use of Pre-set Methods and auto tuning, which ensures reproducible performance irrespective of operator experience.

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Published June 1 2016

Publication number: 5991-6943EN



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