Determination of Critical Elements in Foods in Accordance with US FDA EAM 4.7 ICP-MS Method

Extending the scope of routine food analysis using IntelliQuant data analysis

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Introduction

Consumers expect that the food they buy will be safe to eat, so manufacturers take steps to ensure the levels of harmful chemicals and pathogens are strictly controlled. In addition, governments and regulatory bodies in many countries have a legal obligation to protect public health in relation to food. The chemicals that are controlled in foodstuffs include organic contaminants such as pesticide residues, and inorganic contaminants such as heavy metals. In the United States (US), the Food and Drug Administration (FDA) regulates a wide range of foods. The US FDA also publishes details of the analytical methods that laboratories should use to help ensure food safety. For example, FDA Elemental Analysis Manual (EAM) 4.7 is a comprehensive method that describes how to determine 12 elements in food by ICP-MS following microwave assisted acid decomposition. EAM 4.7 also outlines a series of quality control (QC) tests to ensure instrument performance and data accuracy (*1*).

It is now easier than ever for food-testing laboratories using the EAM 4.7 method to carry out the analysis with Agilent ICP-MS instruments. Agilent single quadrupole ICP-MS use an Octopole Reaction System (ORS^4) cell operating in helium (He) collision cell mode with Kinetic Energy Discrimination (KED). This combination provides the optimum configuration to control common polyatomic interferences, leading to more accurate results. To improve detection of analytes with intense background overlaps, such as Se-78, P-31, and Si-28, the ORS^4 cell can use an enhanced, high energy He mode. Enhanced He mode provides low detection limits for these interfered analytes, avoiding the need for a reactive cell gas such as H_2, O_2, or NH_3. Avoiding reactive gases ensures that no new molecular interferences are formed in the cell, improving the quality of the data and streamlining the method (*2*). Agilent ICP-MS instruments have a wide (10 or 11 orders) linear dynamic range, so major and trace analytes in food samples can be measured in a single run. The wide dynamic range simplifies method setup by removing the need for custom tuning conditions for major elements, while also ensuring that fewer reruns occur due to over-range results.

Agilent High Matrix Introduction (HMI) technology further improves the already exceptional plasma robustness, enabling the ICP-MS to handle samples with total dissolved solids (TDS) levels up to 3% (and up to 25% with Ultra (U) HMI). HMI (and UHMI) uses aerosol dilution to handle high-matrix samples, reducing sample preparation time and minimizing the risk of introducing sampling errors or contamination from conventional liquid dilution (*3, 4*). A further benefit of HMI/UHMI is that it practically eliminates matrix suppression, so varied, high matrix sample digests can be run against a simple, synthetic calibration, without requiring matrix-matching.

Agilent ICP-MS MassHunter IntelliQuant Quick Scan function simplifies data review by collecting full mass spectrum data for every sample in only a few seconds. The IntelliQuant results provide semiquantitative concentrations for up to 78 elements, together with identification and confirmation of unexpected elements by comparison with isotopic abundance templates. The periodic table “heat map” view of the results provides a quick and simple overview of the concentration of all elements within the sample. IntelliQuant can also calculate and display the total matrix solids (TMS) content of each sample. TMS data provides a useful indication of the total matrix load and any variation in dissolved solids level through the batch (*5*).

This study describes the use of the Agilent 7800 ICP-MS and Agilent SPS 4 autosampler for the analysis of 20 elements in different food samples using a single He cell gas method. The list of elements included the 12 elements that are specified in EAM 4.7: arsenic, cadmium, chromium, copper, lead, manganese, mercury, molybdenum, nickel, selenium, thallium, and zinc. The data quality obtained for these elements was assessed through the measurement of three food standard reference materials (SRMs), a fortified method blank (FMB), and two fortified analytical portions (FAPs). FAP refers to samples that are spiked before sample preparation.

Experimental

Calibration standards

The calibration standards were prepared in 3% nitric acid (HNO_3) and 0.5% hydrochloric acid (HCl). HCl is routinely added to samples for analysis using Agilent ICP-MS systems, as it ensures that chemically unstable elements such as Hg are retained in solution. Any Cl-based polyatomic overlaps formed are easily controlled using the standard He cell mode. Calibration standards were prepared from Agilent standard solutions including environmental calibration standard, p/n 5183-4688, multi-element calibration standard-1, p/n 8500-6944, and 1000 µg/mL single calibration standard for Hg, p/n 5190-8485. Most elements were calibrated from 0.1 to 25 ppb. Cu, Zn, and Mn were calibrated up to 250 ppb. Hg was calibrated from 0.01 to 2.5 ppb. Continuing calibration verification (CCV) standards were prepared at 1 ppb (2 ppb for Hg), and/or 10 ppb.

An Agilent internal standard (ISTD) solution (part number 5188-6525), containing 2 ppm Sc, Ge, Rh, In, Tb, Lu, and Bi, was prepared in 1% HNO_3, 0.5% HCl, and 10% isopropanol (IPA). Per the 4.7 method; IPA was added to the ISTD to help equalize As and Se sensitivities due to residual carbon post microwave digestion. The ISTD solution was added automatically online at a flow rate approximately 16 times lower than the sample flow.

Reference materials and samples

Three varied food matrix SRMs from National Institute of Standards and Technology (NIST, Gaithersburg, US) were used to validate the method. The SRMs used were NIST 1546a Meat Homogenate, NIST 1549 Non-Fat Milk Powder, and NIST 2385 Slurried Spinach.

In addition to the SRMs, a diverse set of food samples with different % composition of fats, proteins, and carbohydrates were analyzed in this study. The following products were bought in a supermarket in North Carolina, USA: beef jerky, fortified nutritional shake, gouda cheese, gummy bears, powdered donuts, and dark chocolate. Other samples consisting of pepperoni, rice noodles, frozen dinner, and frozen pizza were also digested and analyzed in the same batch and the results are reported elsewhere (*6*).

Standard and sample preparation

All the food samples included in this study were digested "as received", except for the donut, which was crushed in a bag before sampling. The samples were then prepared for analysis according to the digestion procedure outlined in the EAM 4.7 method using a MARS 6 closed-vessel microwave digestion system located at CEM Corporation, USA. Approximately 0.5 g of each food sample or SRM was accurately weighed into a 75 mL PFA-lined MARS Xpress vessel. 8 mL of HNO_3 and 1 mL of H_2O_2 was added to each vessel. Duplicates of the samples, SRMs, and spiked samples (FAPs) were prepared and digested in a single batch, using the heating program shown in Table 1. Each digestion batch can accommodate up to 40 vessels containing a variety of food sample matrices, with a single program being used for all sample types. Finally, 0.5 mL concentrated HCl was added to the digests, followed by de-ionized water to a final weight of 100 g.

Table 1. Microwave digestion parameters.

Parameter	Setting
Power (W)	1800
Ramp Time (min)	25
Hold Time (min)	15
Temperature (°C)	200

The analytical sequence of calibration standards, samples, and QC solutions is shown in Figure 1. The sample block was analyzed repeatedly with automatic insertion of the periodic QC block after every 10 samples.

Instrumentation

An Agilent 7800 ICP-MS, which includes the ORS^4 collision cell and HMI aerosol dilution system, was used for the analysis. The 7800 has been superseded by the Agilent 7850 ICP-MS, but the configuration and analytical settings reported here apply to both models. Sampling was performed using an Agilent SPS 4 autosampler. The ICP-MS was configured with the standard sample introduction system, consisting of a MicroMist glass concentric nebulizer, temperature-controlled quartz spray chamber, and quartz torch with 2.5 mm id injector. The interface consisted of a nickel-plated copper sampling cone and a nickel skimmer cone.

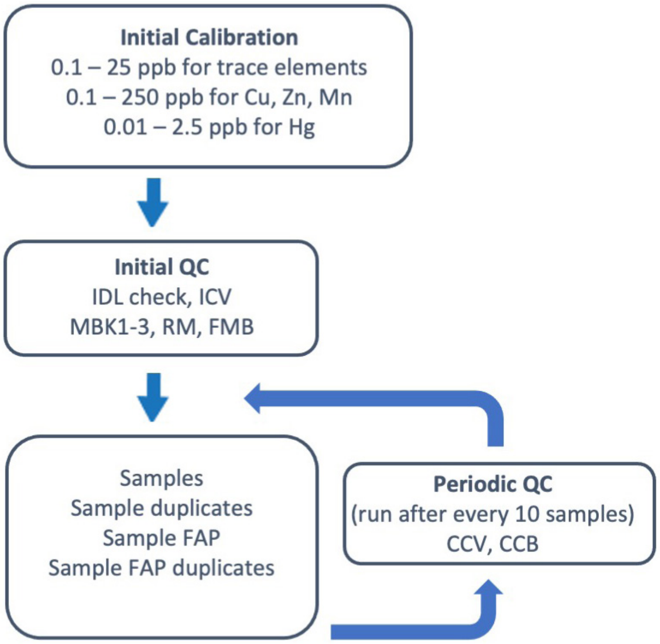

Figure 1. Analytical sequence.
Key: Instrument detection limit (IDL), initial calibration verification (ICV), method blank (MBK), reference material (RM), fortified method blanks (FMB), fortified analytical portion (FAP), continuing calibration verification (CCV), continuing calibration blank (CCB).

The Rare Earth Elements (REEs) have relatively low second ionization potentials, so readily form doubly charged ions (M^{2+}) in the plasma. If REEs such as Nd, Sm, Gd, and Dy are present in a sample at a high enough concentration, M^{2+} interferences can affect the accuracy of the measurement of As and Se. Therefore, the EAM 4.7 method recommends that analysts monitor the following isotopes: ^{146}Nd, ^{147}Sm, ^{155}Gd, ^{163}Dy to assess the potential for M^{2+} overlaps. The Agilent ICP-MS MassHunter software includes an easy-to-use, automated function to correct for the contribution that REE^{2+} ions make to the signals measured for As and Se.

Unknown samples can also be easily screened for REEs (and the entire periodic table) using the IntelliQuant function in the ICP-MS MassHunter software (version 4.6 and later). IntelliQuant works by performing a full mass-spectrum scan with only two seconds measurement time. The IntelliQuant results provide valuable information about the food samples, including:

- The elemental composition of each sample, including REEs. The results can be displayed in a table or as a periodic table heat map.
- Confirmation of an unexpected element using isotope templates.
- An estimation of the Total Matrix Solids (TMS) level for each sample or based on the analysis of a typical sample.

The TMS function uses the IntelliQuant semiquantitative data to calculate the approximate solids levels of a sample. The calculation excludes gas elements, such as Ar, O, and N, together with C, P, S, and the halides, ensuring a more accurate result. To simplify method setup and ensure the best possible accuracy for uncalibrated elements, the semiquant data (and TMS calculation) uses a mass response profile generated from the quantitative calibration standards. The TMS function is a great diagnostic tool to identify possible causes of ISTD suppression. It is especially useful when dealing with unknown and potentially difficult food sample matrices and deciding if a sample needs to be diluted or a higher HMI setting is needed. The measured TMS levels for the samples analyzed in this study are shown in Table 2.

Table 2. Total matrix solids data for six food samples obtained by the TMS function of ICP-MS MassHunter. Each sample was prepared in duplicate and each preparation was measured twice. The data is corrected for dilution. Concentration units: ppm.

	Beef Jerky	Fortified Nutritional Shake	Gouda Cheese	Gummy Bears	Powdered Donuts	Dark Chocolate
TMS	36433	3872	15136	768	6637	17292

Main operating conditions of the Agilent 7800 – such as plasma settings – are typically loaded from a preset method, selected as appropriate for the sample types being measured. In this case, a plasma preset of HMI-4 (aerosol dilution factor of four) was used, based on the typical matrix levels for the food types being measured (Table 2). When HMI is selected, all related settings are autotuned as appropriate for the matrix levels of the target sample types. Other instrument operating settings were optimized automatically using the ICP-MS MassHunter autotune function. All analytes were acquired in He mode (enhanced He mode for Se). Instrument operating conditions are listed in Table 3.

Table 3. ICP-MS operating conditions*.

ICP-MS Parameter	Setting
RF Power (W)	1600
Sampling Depth (mm)	10
Nebulizer Gas Flow (L/min)	0.6
Dilution (HMI) Gas Flow (L/min)	0.35
Lens Tune	Autotune
Helium Cell Gas Flow (mL/min)	4.3 (10**)
Energy Discrimination (V)	5 (7**)

** Shaded parameters are defined in the method and HMI-4 plasma presets; all parameters were automatically optimized during start-up and autotuning. ** Enhanced He mode settings used for Se.*

Results and discussion

Typical 7800 ICP-MS instrument detection limits (DLs) and background equivalent concentrations (BECs) calculated from the ICP-MS MassHunter calibrations are shown in Table 4. The EAM method detection and quantification limits – also shown in Table 4 – were calculated based on method blanks measured at the end of the run, n=10 (7). Data was acquired for 20 elements, including the 12 elements required by EAM 4.7, using only He as a cell gas. The 7800 ICP-MS analytical limits are better than the nominal limits provided in EAM 4.7 for all elements.

Table 4. 7800 ICP-MS detection limits and EAM 4.7 nominal analytical limits.

	ICP-MS MassHunter		Calculated based on EAM 4.7 Analytical Limits		EAM 4.7 Nominal Analytical Limits	
Element	DL (µg/kg)	BEC (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)
27 Al	0.148	0.421	1.167	9.103		
51 V	0.065	0.311	0.996	7.770		
52 Cr	0.032	0.176	0.275	2.144	5.39	48.9
55 Mn	0.016	0.125	1.224	9.546	2.33	21.2
56 Fe	0.012	0.671	0.875	6.827		
59 Co	0.004	0.006	0.029	0.225		
60 Ni	0.011	0.073	0.316	2.463	6.38	58.0
63 Cu	0.008	0.550	0.196	39.32	6.02	54.7
66 Zn	0.049	0.286	1.865	14.555	37.4	340
75 As	0.005	0.059	0.217	1.693	1.27	11.6
78 Se	0.016	0.046	0.404	3.149	7.28	66.1
95 Mo	0.010	0.014	1.196	9.333	5.18	47.1
111 Cd	0.002	0.003	0.037	0.287	0.408	3.71
121 Sb	0.005	0.012	0.075	0.582		
137 Ba	0.018	0.009	0.151	1.176		
201 Hg	0.003	0.010	0.681	5.313	0.861	7.82
205 Tl	0.002	0.001	0.174	1.355	*0.281	*2.10
208 Pb	0.003	0.013	0.034	0.266	1.20	10.9
232 Th	0.006	0.004	0.052	0.409		
238 U	0.001	0.002	0.026	0.202		

*All elements were acquired in He mode (enhanced He for Se). The Nominal Analytical Limits are given in EAM 4.7 and are based on method blanks measured during the single lab validation over one year; n = 143. *Based on a single lab validation (n = 27).*

Verification of instrument calibration and sample digestion process

As part of the method quality control procedure specified in EAM 4.7, and to ensure the ongoing validity of the calibration, a CCV standard was analyzed five times during the analytical sequence. As shown in Figure 2, all elements in the five CCVs and the initial ICV were recovered within the EAM acceptance criteria of ±10% of the actual concentration.

Table 5. Mean measured concentrations in three food SRMs corrected for dilution. Mean calculated from two separate digests, each measured twice in triplicate.

	NIST 1546a Meat Homogenate				NIST 1549 Non-Fat Milk Powder				NIST 2385 Slurried Spinach			
Element	**Certified Conc (mg/kg)**	**Measured Conc (mg/kg)**	**Recovery (%)***	**QC Criteria**	**Certified Conc (mg/kg)**	**Measured Conc (mg/kg)**	**Recovery (%)***	**QC Criteria**	**Certified Conc (mg/kg)**	**Measured Conc (mg/kg)**	**Recovery (%)***	**QC Criteria**
^{52}Cr					0.0026	<DL	**					
^{55}Mn	0.286	0.285	100	Pass	0.260	0.269	103	Pass	3.81	3.65	96	Pass
^{56}Fe	10.17	10.9	107	Pass	1.78	1.77	100	Pass	17.1	16.6	97	Pass
^{63}Cu	0.605	0.602	100	Pass	0.7	0.748	107	Pass	0.90^{R}	0.818	91	
^{66}Zn	17.88	19.0	106	Pass	46.1	42.4	92	Pass	8.37	7.94	95	Pass
^{78}Se	0.281	0.301	107	Pass	0.11	0.118	107	Pass				
^{98}Mo	0.016^{R}	0.019	119									
^{111}Cd					0.0005	<LOQ	**					
^{137}Ba	0.077^{R}	0.079	103									
^{201}Hg					0.0003	<LOQ	**					
^{208}Pb					0.019	0.022	116	Pass				

** FDA Elemental Analysis Manual (Section 3.4 Special Calculations) 3.4 Equation 20. ** Recoveries not calculated as the certified values are close to or below the method <LOQ. R = non-certified reference value.*

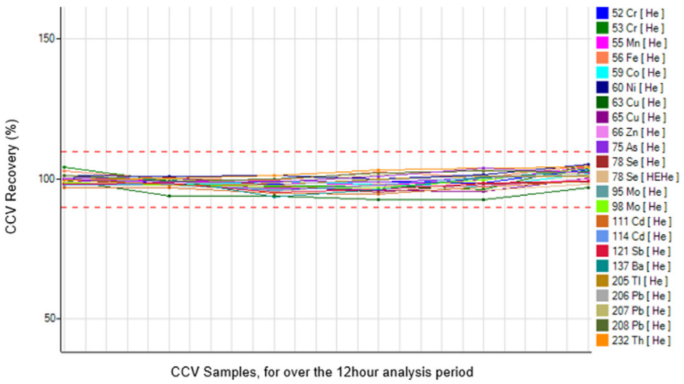

Figure 2. CCV recoveries over the course of the 12-hour sequence, including at the end of the analytical sequence.

To verify the sample digestion process, each of the three NIST SRMs was prepared in duplicate and each preparation was analyzed twice (with three replicates per analysis) using the 7800 ICP-MS. As shown in Table 5, the mean concentrations were in good agreement with the certified concentrations, meeting the QC criteria requirements of the FDA EAM method of 80–120%. Since not all SRMs are certified for all analytes, blank cells indicate the absence of a certified or reference value.

Matrix effects and spike recoveries

To test for non-spectral interferences (matrix effects), two fortified method blanks (FMB) were prepared by spiking the blank at 1 and 50 ppb. The higher-level spike was used for the elements Al, Fe, Cu, and Zn, while the low-level spike was used for the remaining trace elements. The FMB was analyzed periodically throughout the entire method procedure. All recoveries were within the EAM 4.7 method acceptable % recovery range of 90-110%, as shown in Table 6.

A spike recovery (FAP) test was carried out to check the recovery of the digestion and the accuracy of the 7800 ICP-MS method for food sample analysis. Two food samples (beef jerky and gummy bears) were selected at random. Both samples were spiked at two levels – 1 and 50 ppb – for all analytes and measured using ICP-MS. For samples that had naturally occurring elemental concentrations below 5 ppb, the 1 ppb spike recovery is reported. For samples with higher naturally occurring concentrations, the 50 ppb spike results are reported. The recoveries for all elements in the fortified food samples were within the EAM 4.7 method QC criteria of ±20%, as shown in Table 6.

Quantitative results for food samples

Quantitative results are given in Table 7 for six food products. In addition to the 12 elements specified in EAM 4.7, data is provided for Al, V, Fe, Sb, Ba, Th, and U. According to the IntelliQuant data for each sample, M^{2+} interferences from ^{150}Nd^{2+} and ^{150}Sm^{2+} on ^{75}As^{+}, and from ^{156}Gd^{2+} and ^{156}Dy^{2+} on ^{78}Se^{+} were unlikely to be a problem given the low concentrations of the REEs.

The measured concentrations of Cd and Pb in dark chocolate are high compared to the other foods. Chocolate is known to contain Cd, which is present in the soil of some cocoa plantations (*8*). The US doesn't have limits for Cd in chocolate, but the European Union (EU) introduced maximum levels on January 1, 2019 (*9*). The limit for dark chocolate with total dry cocoa solids above 50% is 0.80 mg/kg (800 ppb). The measured value of 205 ppb is well below the EU maximum level.

The FDA recommends a maximum Pb level of 0.1 ppm (100 ppb) in candy that is likely to be consumed frequently by small children (*10*). The measured concentrations for Pb in gummy bears and chocolate are well below the maximum guideline level.

Table 6. Mean recovery results based on the analysis of food sample digests. Mean calculated from two separate digests, each measured twice in triplicate.

	Quantitative Results		Fortified Method Blank		Fortified Analytical Portion (Beef Jerky)		Fortified Analytical Portion (Gummy Bears)	
Element	Beef Jerky	Gummy Bears	1.0 ppb spike		1.0 ppb spike		1.0 ppb spike	
	ppb		Mean Recovery ± 1σ (%)	QC Criteria	Mean Recovery ± 1σ (%)	QC Criteria	Mean Recovery ± 1σ (%)	QC Criteria
^{27}Al	3700	1390	101 *	Pass	117 *	Pass	114 *	Pass
^{51}V	<DL	<DL	96	Pass	104	Pass	113	Pass
^{52}Cr	107	162	101	Pass	110	Pass	102	Pass
^{55}Mn	2630	31.6	99	Pass	90	Pass	100	Pass
^{56}Fe	54200	1210	98 *	Pass	89 *	Pass	111 *	Pass
^{59}Co	1.0	1710	101	Pass	99	Pass	104	Pass
^{60}Ni	259	<DL	102	Pass	101	Pass	104	Pass
^{63}Cu	1250	157	100 *	Pass	110 *	Pass	94	Pass
^{66}Zn	82400	<DL	94 *	Pass	88 *	Pass	105	Pass
^{75}As	13.2	<DL	93	Pass	98	Pass	94	Pass
^{78}Se**	476	<DL	98	Pass	95	Pass	103	Pass
^{95}Mo	47.7	7.6	99	Pass	98	Pass	97	Pass
^{111}Cd	31.6	6.5	97	Pass	102	Pass	96	Pass
^{121}Sb	2630	<DL	90	Pass	93	Pass	89	Pass
^{137}Ba	577	9.7	96	Pass	95	Pass	106	Pass
^{201}Hg	<DL	<DL	97	Pass	84	Pass	82	Pass
^{205}Tl	<DL	<DL	94	Pass	88	Pass	100	Pass
^{208}Pb	9.5	<DL	97	Pass	101	Pass	102	Pass

*The dilution factor of both samples was ~200. *A 50 ppb spike was used. **Enhanced He mode used for Se.*

Table 7. Quantitative data for six food products. Units: µg/kg.

	Beef Jerky	Fortified Nutritional Shake	Gouda Cheese	Gummy Bears	Powdered Donuts	Dark Chocolate
^{27}Al	3700 ± 224	746 ± 10	705 ± 131	1390 ± 300	121000 ± 2900	33600 ± 1600
^{51}V	25.6 ± 5.9	7.1 ± 1.9	<DL	30.8 ± 2.8	33.1 ± 1.7	82.3 ± 8.8
^{52}Cr	107 ± 3	197 ± 2	71.9 ± 3.5	162 ± 4	135 ± 9	1540 ± 36
^{55}Mn	2630 ± 120	6400 ± 56	263 ± 4	31.6 ± 2.4	2500 ± 46	20400 ± 110
^{56}Fe	54200 ± 1400	23900 ± 227	1270 ± 1	1710 ± 27	15600 ± 200	127000 ± 2100
^{59}Co	1.0 ± 0.2	<DL	<DL	<DL	<DL	538 ± 4
^{60}Ni	259 ± 66	57.5 ± 6.4	34.6 ± 2.6	157 ± 5	193 ± 33	5080 ± 35
^{63}Cu	1200 ± 30	2590 ± 22	210 ± 4	<DL	543 ± 25	18700 ± 148
^{66}Zn	82400 ± 1890	21900 ± 189	43900 ± 260	<DL	4380 ± 50	39700 ± 320
^{75}As	13.2 ± 0.4	6.6 ± 0.5	3.4 ± 0.4	7.6 ± 0.6	6.8 ± 0.6	16.3 ± 0.7
^{78}Se*	476 ± 5	131 ± 1	50.6 ± 0.5	<DL	88.9 ± 2.5	98.4 ± 5.7
^{95}Mo	47.7 ± 2.6	227 ± 3	82.5 ± 1.4	9.7 ± 0.6	144 ± 10	218 ± 4
^{111}Cd	31.6 ± 0.7	19.6 ± 0.6	18.9 ± 0.1	18.7 ± 0.1	29.3 ± 0.6	205 ± 2
^{121}Sb	<DL	<DL	<DL	<DL	<DL	<DL
^{137}Ba	577 ± 40	116 ± 3	619 ± 12	77.1 ± 1.3	654 ± 24	7500 ± 28
^{201}Hg	<DL	<DL	<DL	<DL	<DL	<DL
^{205}Tl	<DL	<DL	<DL	<DL	<DL	<DL
^{208}Pb	9.5 ± 0.8	<DL	<DL	<DL	6.7 ± 0.5	29.8 ± 0.9
^{232}Th	<DL	<DL	<DL	<DL	<DL	<DL
^{238}U	<DL	<DL	<DL	<DL	<DL	<DL

**Enhanced He mode used for Se.*

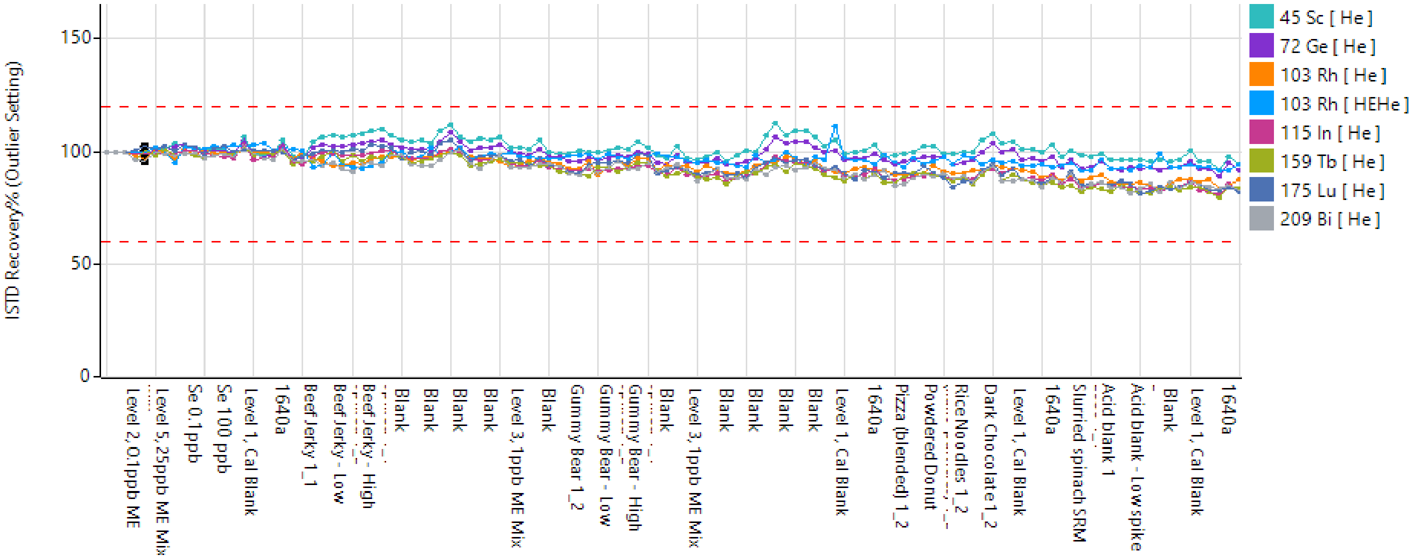

Figure 3. Stability of ISTD measurements over 12 hours. The ISTD recoveries have been normalized to the calibration blank for all samples.

ISTD recovery (%)

The analytical sequence outlined in Figure 1 was analyzed repeatedly over 12 hours. All the ISTD recovery plots were within ±20%, with no internal standard failures throughout the run, meeting the criteria specified in EAM 4.7 (Figure 3). The results demonstrate the robustness of the plasma and high matrix tolerance of the 7800 ICP-MS with HMI over long runs.

IntelliQuant data

When an analyst develops a quantitative method using an ICP-MS MassHunter preset method, an IntelliQuant Quick Scan acquisition is predefined in the He mode tune step. No special setup or separate calibration is needed for IntelliQuant, simplifying the analysis. IntelliQuant automatically acquires full mass-spectrum data in every sample with only 2 s additional measurement time, allowing the analyst to quickly see which elements are present in the samples. Because IntelliQuant data is acquired in He collision cell mode, analytes are free from common polyatomic ion overlaps, ensuring the quality of the data.

In this study, IntelliQuant data was acquired for each food sample with the 7800 ICP-MS operating in He mode. The data can be displayed in a periodic table heat map view, as shown in Figures 4 and 5. The color intensity heat map shows the approximate concentration of up to 78 elements in each sample, with a darker color indicating a higher concentration of that element.

The IntelliQuant data provides a complete picture of the elements present in the sample, as data can be reported for elements not included in the calibration standards. This benefit is demonstrated in the heat map display for donuts (Figure 4), which shows a relatively high concentration of Ti (labeled as food additive "TiO_2" on the packaging). The IntelliQuant semiquantitative result for Ti in the donut samples was ~90 ppm. Figure 5 shows the heat map results for dark chocolate, indicating a relatively high concentration of Ca, Cr, Ni, W, and Pb. Figure 6 shows the IntelliQuant Quick Scan spectrum for the dark chocolate, illustrating a good fit to the natural isotope template for W, confirming the presence of this element in dark chocolate.

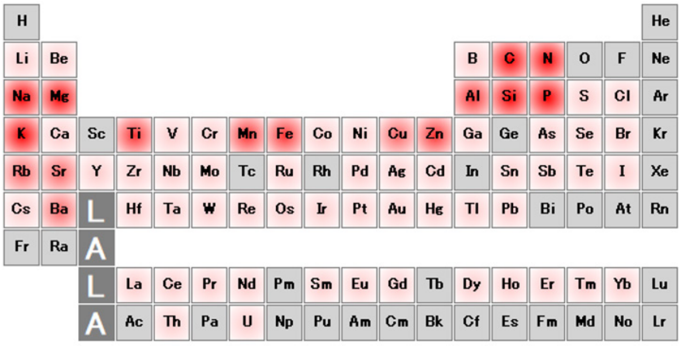

Figure 4. Periodic table heat map view of ICP-MS IntelliQuant data acquired for powdered donuts.

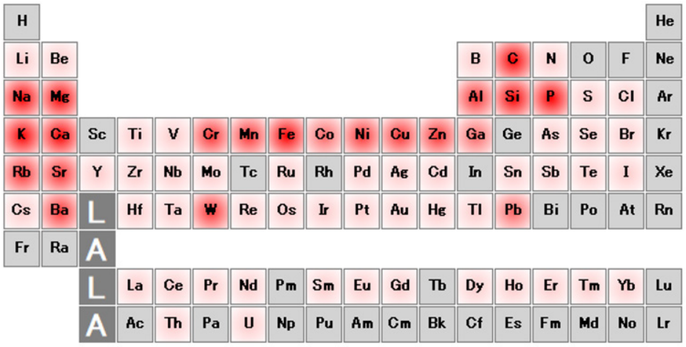

Figure 5. Periodic table heat map view of ICP-MS IntelliQuant data acquired for dark chocolate.

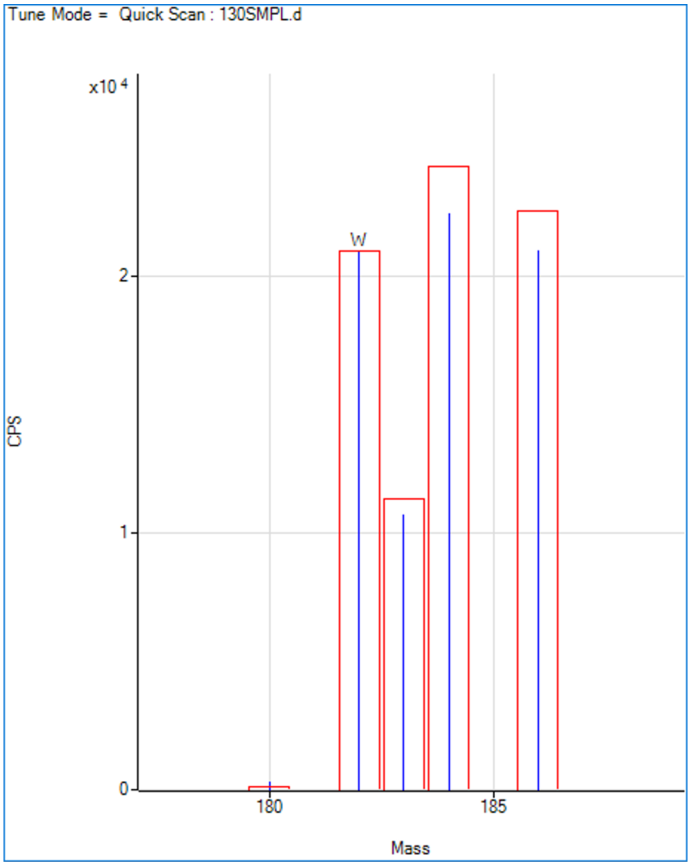

Figure 6. The unexpectedly high concentration of W reported in dark chocolate was confirmed by the isotope template fit in the IntelliQuant Quick Scan mass spectrum.

Conclusion

The Agilent 7800 ICP-MS was used to analyze multiple elements in a range of everyday foods in accordance with US FDA EAM method 4.7 for food and related products. All samples were prepared in the same batch using a single microwave digestion method.

In-built features of the ICP-MS and ICP-MS MassHunter software were used to simplify method development, improve instrument performance, and ensure high-quality data.

– A preset method and autotune were used to predefine and optimize instrument operating parameters, accelerating instrument setup.

– The ORS^4 cell was operated with a single gas (helium-KED mode), which is the standard operating mode for quantitative analysis of unknown samples. As indicated by the spike recovery data, He mode effectively removed polyatomic interferences, enabling high-quality multi-element data to be obtained for all elements, including As and Se.

– IntelliQuant was used to provide semiquantitative concentrations for all measurable elements present in each sample, including confirming the unexpected presence of nontarget elements. IntelliQuant also provided an estimate of the total matrix content of the food digests, helping with method optimization and scheduling of routine maintenance.

The accuracy of the method was evaluated by analyzing three food-based SRMs and conducting a spike recovery test for 12 elements in two food samples. Excellent recoveries were achieved in all cases. The instrument also exceeded the nominal detection limit requirements specified in the EAM method and showed excellent stability over a 12-hour run.

The study showed that Agilent ICP-MS instruments are suitable for the routine, multi-element screening of trace level elements in foods, making them ideal for food safety management programs.

References

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