APPLICATION NOTE 44459

Trace level quantification of multiple elements in meat and meat products using ICP-MS

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Goal

The objective of this application note is to demonstrate the applicability of the Thermo Scientific™ iCAP™ RQ ICP-MS for the quantification of trace elements in meat and meat products at trace levels in compliance with the AOAC 2015.01 guideline, the Food Safety and Standards Authority of India (FSSAI),[1] China Food and Drug Administration (CFDA),[2] and European Commission (EC)[3] MRLs.

Introduction

The Food Outlook Report from FAO (Food and Agricultural Organisation) contains detailed information on the production, consumption, and price indices of meat. As per the report, in the year 2018, total meat production, including poultry, bovine, ovine, and pig meat, was 335 million tons, which is a 1.5% increase compared to 2017. Meat is an essential nutritional source of protein for humans and provides high biological protein, vitamins, and essential minerals such as iron and zinc. A diet containing 20 g of protein per day is considered a healthy diet. Though India is the second-largest vegetarian country after Bangladesh, the consumption of meat is rising as the result of increasing average income and urbanization. It is expected that per capita consumption will grow to 50 kg in 2050 globally. Poultry meat production alone is increasing annually greater than 4%. The overall meat production in India is 7.5 million tons, which is 2.2% higher than the year 2017.[4]

It is crucial to assure food safety while reducing malnutrition by increasing meat consumption. Expanded industrialization and urbanization are introducing more pollution to water bodies, and livestock may be prone to heavy metal contamination through contaminated water and feeds that grown in contaminated soil.

There is a significant threat of toxicity in the consumption of meat through contamination with pesticide residues, veterinary drugs, and heavy metals, even if they are present at trace levels. In the case of heavy metals, bioaccumulation and biomagnification may cause contamination in meat. The maximum limits of elements that are set by FSSAI, EU, and CFDA regulations are shown in Table 1.

Table 1. Target analytes with the FSSAI, EU, and CFDA MRL values in mg.kg^{-1}

Elements	FSSAI		EU		CFDA	
	Meat	Offal[#]	Meat	Offal[#]	Meat	Offal[#]
Mercury	1.0	-	-	-	0.05	-
Arsenic	1.1	-	-	-	0.50	-
Lead	0.1	0.5	0.1	0.5	0.2	0.5
Cadmium	0.1	-	0.05	0.5 (liver) 1.0 (kidney)	0.1	0.5 (liver) 1.0 (kidney)
Copper	30	-	-	-	-	-
Tin	250	-	-	-	250	-
Chromium	-	-	-	-	1	-

For all other elements in the scope of this study, no MRLs are available in the FSSAI, CFDA, and EU regulations.

[#]Offal is the internal organs (e.g. liver, kidney) used as food for consumption.

ICP-MS is a powerful analytical tool for the analysis of trace elements in a wide variety of sample types, offering the selectivity and sensitivity needed for this application. However, there are several challenges associated with this analytical technique. One of the challenges is the removal of spectral interferences originating from the sample matrix, potentially leading to false positive results. For example, in the analysis of nickel (typically analyzed using the isotope ^{60}Ni), there are several possibilities for the formation of polyatomic species having equivalent mass to charge ratio such as ^{59}Co^{1}H or ^{44}Ca^{16}O in the likely presence of cobalt or calcium. In a similar way the presence of molybdenum, even in low concentrations, will lead to the formation of e.g., ^{95}Mo^{16}O, resulting in mass 111, which affects the quantification of cadmium (commonly accomplished using the isotope ^{111}Cd). To overcome these challenges, efficient removal of all (mostly polyatomic in nature) interferences is required. In state-of-the-art ICP-MS instruments, this is accomplished using kinetic energy discrimination (KED), which uses high purity helium gas as the collision gas in the collision/reaction cell (CRC). All ions passing through the CRC will undergo a series of collisions with helium atoms, and therefore lose part of their kinetic energy. However, polyatomic interferences have a larger size and therefore a larger collisional cross section compared to analytes of the same nominal mass, so that they suffer from a significantly higher number of collisions, and hence a higher loss of kinetic energy. The lower energy interferences are preferentially eliminated from the system by setting a positive potential at the exit of the CRC whilst higher energy analytes exit the cell and enter the analyzing quadrupole. Interferences may not only be formed in the ICP source, but may also be formed directly inside the CRC system, leading to an additional false positive contribution to the observed signal. To allow for a more efficient removal of interferences, KED can be combined with a low mass cut off, essentially removing all precursor ions of lower mass, potentially involved in the formation of additional interferences.

For ICP-MS analysis, it essential that the sample is in liquid form, so that sample preparation using a microwave-assisted digestion method is standard practice for the analysis of meat and meat products in most of the commercial laboratories. Demirezen, *et al.*;[5] Khan, *et al.*;[6] and Nordin, *et al.*[7] have reported microwave digestion for sample preparation of meat and meat products for ICP-MS analysis.

The aim of this application note is to provide a methodology for the determination of a wide range of key analytes at trace levels in meat using the iCAP RQ ICP-MS system in combination with microwave-assisted digestion for sample preparation. The proposed method was validated by following AOAC 2015.01.[8]

Experimental

Chemicals and reagents

- Nitric acid (65–69%), TraceMetal™ Grade, Fisher Chemical™ (A509-P212)
- Hydrogen peroxide (30–32%), TraceMetal™ Grade, Fisher Chemical™ (H/1820/15)
- Hydrochloric acid (35–37%), TraceMetal™ Grade, Fisher Chemical™ (A508-P500)
- Deionized water (18.20 MΩ·cm), Thermo Scientific™ Barnstead™ MicroPure™ Water Purification System
- Single element standard solutions (for all elements under study, each at 1000 µg·mL^{-1}, Inorganic™ Ventures (Christiansburg, Virginia, USA))

Standard preparation and calibration

All analytes under investigation and the internal standards are shown in Table 2. To assess the linearity range of the proposed method, individual calibration standards were prepared through serial dilution by diluting the four different groups of mixed working standards in appropriate concentration ranges. The respective concentrations prepared for each element are summarized in Table 3 and should cover at least three orders of magnitude of linear dynamic range of the method. Gold (200 µg·L^{-1}) was added to all standards and rinse solutions to facilitate the washout of mercury and to reduce memory effect. An internal standard mixture containing Sc, Ge, Y, Rh, In, Tb, Ir, and Bi (Table 2) was added to all samples at a concentration of 20 µg·L^{-1}.

Table 2. List of elements with their mass and internal standard elements

Name of element (Symbol)	Mass	Internal standard element	Mass
Lithium (Li)	7	Scandium (Sc)	45
Beryllium (Be)	9	Scandium (Sc)	45
Boron (B)	11	Scandium (Sc)	45
Aluminium (Al)	27	Scandium (Sc)	45
Vanadium (V)	51	Scandium (Sc)	45
Chromium (Cr)	53	Scandium (Sc)	45
Manganese (Mn)	55	Scandium (Sc)	45
Iron (Fe)	57	Scandium (Sc)	45
Cobalt (Co)	59	Scandium (Sc)	45
Nickel (Ni)	60	Scandium (Sc)	45
Copper (Cu)	65	Scandium (Sc)	45
Zinc (Zn)	66	Scandium (Sc)	45
Arsenic (As)	75	Germanium (Ge)	72
Selenium (Se)	77	Germanium (Ge)	45
Strontium (Sr)	88	Yttrium (Y)	89
Molybdenum (Mo)	98	Rhodium (Rh)	103
Cadmium (Cd)	111	Rhodium (Rh)	103
Tin (Sn)	118	Rhodium (Rh)	103
Antimony (Sb)	121	Indium (In)	115
Barium (Ba)	137	Terbium (Tb)	159
Mercury (Hg)	202	Bismuth (Bi)	209
Lead (Pb)	208	Bismuth (Bi)	209

Table 3. Calibration level standard preparation for elements

Levels	Linearity conc.	Final volume (mL)	Intermediate standard conc.(µg·L^{-1})	Required volume (mL)
Set 1	Hg, Cd, Co (µg·L^{-1})			
STD1	0.025	25	10	0.0625
STD2	0.05	25	10	0.125
STD3	0.10	25	10	0.25
STD4	0.25	25	10	0.625
STD5	0.50	25	10	1.25
STD6	5.00	25	1000	0.125
STD7	20.00	25	1000	0.50
Set 2	Pb, As, Sb, Sn (µg·L^{-1})			
STD1	0.05	25	10	0.125
STD2	0.10	25	10	0.25
STD3	0.20	25	10	0.50
STD4	0.40	25	100	0.10
STD5	2.00	25	100	0.50
STD6	10.00	25	1000	0.25
STD7	20.00	25	1000	0.50
Set 3	B, Ba, Be, Cu, Cr, Li, Mn, Mo, Ni, V (mg·L^{-1})			
STD1	0.001	25	1	0.025
STD2	0.002	25	1	0.05
STD3	0.004	25	1	0.10
STD4	0.01	25	1	0.25
STD5	0.05	25	10	0.125
STD6	0.1	25	10	0.25
STD7	0.2	25	10	0.50
Set 4	Al, Fe, Zn (mg·L^{-1})			
STD1	0.025	25	10	0.0625
STD2	0.05	25	10	0.125
STD3	0.10	25	10	0.25
STD4	0.25	25	10	0.625
STD5	0.50	25	100	0.125
STD6	1.00	25	100	0.25
STD7	2.00	25	100	0.50

Sample preparation

Red meat of wild water buffalo (*Bubalus arnee*), chicken (*Gallus gallus domesticus*), and goat (*Capra aegagrus hircus*) were purchased from the local market. The samples were homogenized using a heavy-duty mixer grinder Maharaja (Whiteline New Delhi, India). The homogenized samples were weighed to approximately 0.5 ± 0.05 g in a pre-cleaned, dry 75 mL capacity microwave digestion vessel. For the spike recovery experiment, a sample was spiked with all analytes assessed in the proposed method before the addition of any solvent. 200 µg·L^{-1} gold was added to the sample as a final concentration in the sample solution to stabilize mercury. Additionally, 2 mL nitric acid (HNO_3), 1 mL hydrogen peroxide (H_2O_2), and 0.2 mL hydrochloric acid (HCl) were added and the sample was kept in a fume hood for 60 min for pre-digestion. Then, 1 mL deionized water was added and the microwave digestion vessels were closed. The microwave digestion process was started with a set temperature program shown in Table 4. A Mars™ 6 microwave digestion system (CEM Corporation, Matthews, NC, USA) was used to perform microwave digestion.

Table 4. Temperature program for microwave digestion

	Ramp time (min)	Hold time (min)	Temperature	Power
Step 1	40	30	200 °C	1500 W

Note: Ramp time and microwave power settings may vary depending on the number of vessels.

After completion of the digestion process, the microwave digestion vessels were cooled by keeping the rotor at room temperature for 15 min. The vessels were opened slowly and carefully in a fume cupboard, as pressurized acid fumes could evaporate. The digested sample solution was quantitatively transferred to the pre-cleaned 50 mL volumetric flask with multiple rinsing with deionized water. The internal standards were added from a stock solution containing 10 mg·L^{-1} of each element (final concentration is 20 µg·L^{-1}), and the total volume was adjusted to 50 mL with deionized water. The prepared sample solutions were vortexed well for thorough mixing. A procedural blank was prepared by following the above protocol without a sample matrix.

ICP-MS analysis

To perform multi-element analysis in meat, a iCAP RQ ICP-MS system was used. KED analysis mode was selected to quantify all elements to ensure the complete removal of all possible polyatomic interferences. To enable high throughput analysis, a ASX 560 autosampler (Teledyne CETAC Technologies, Omaha, NE, USA) was used. A summary of all instrument conditions used for meat analysis is shown in Table 5.

Table 5. ICP-MS parameters

Parameter	Value
Forward power (RF)	1550 W
Nebulizer gas	1.0 L·min^{-1}
Auxiliary gas	0.8 L·min^{-1}
Cool gas flow (Argon)	14.0 L·min^{-1}
CCT gas flow (He gas)	5.4 mL·min^{-1}
KED bias potential	3 V
Sample uptake/wash time	45 s
Dwell time	0.05 s
Number of readings per sample	Three main runs with 10 sweeps each
Total acquisition time (3 repetitions including rinse)	150 s

Data acquisition and processing

Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software was used for data acquisition and processing. The Qtegra ISDS Software simplifies the process of method set up, starting from the selection of the most appropriate isotope for each element selected for analysis, supporting mathematical correction of isobaric interference (In and Sn 115), and straightforward selection of the measurement mode for all target elements. Setting up a sequence for analysis is accomplished in a so-called LabBook, a file format containing all relevant information about the acquired data, including method information and data evaluation settings.

Results and discussion

Sample preparation

Unlike dry powders such as spices, meat products react very slowly with acids. After the addition of acids, all samples were kept for 60 min for pre-digestion to support the initiation of reaction between the meat matrix and acids. A volume of 1 mL of deionized water was added just before placing the vessels in the microwave digester to avoid the dilution of acids in the pre-digestion period, which may further delay the reaction process. In the case of dry powder, deionized water was added before the addition of acids to avoid an exothermic reaction with the concentrated acids.

Linearity

All four groups of elements were mixed together in seven linearity standard solutions with different concentration levels (Table 3). Using these seven linear standard solutions, the calibration curves of each element were plotted against their intensity. As an example, the calibration curve of arsenic is shown in Figure 1. All target elements showed an excellent correlation coefficient over the assessed concentration range. The R^2 values of each analyte element and the %RSD values are shown in Table 6.

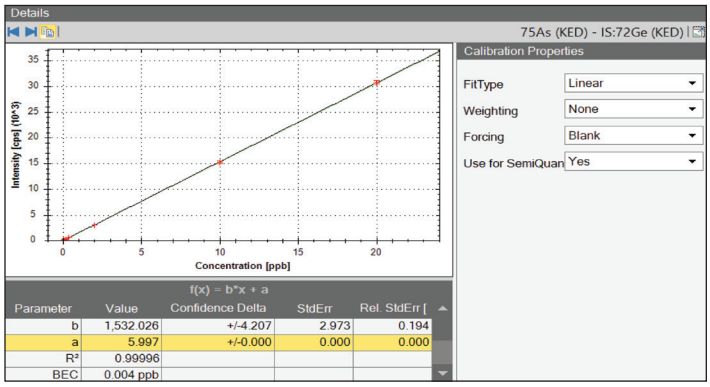

Figure 1. Calibration curve of arsenic obtained from Qtegra ISDS Software over a concentration range of 0.05–20 ppb

Table 6. R^2, %RSD of intensity, and instrument detection limit of all target analytes of the lowest standard obtained from Qtegra ISDS Software

Element	R^2	Intensity %RSD	IDL in ppb
Li	0.99985	0.3	0.06
Be	0.99999	4.1	0.02
B	0.99988	2.2	0.27
Al	0.99993	2.6	0.07
V	0.99998	4.0	0.01
Cr	>0.99999	3.9	0.05
Mn	0.99996	1.8	0.003
Fe	0.99999	2.0	0.75
Co	0.99994	5.3	0.0006
Ni	0.99998	2.2	0.01
As	0.99998	3.2	0.004
Se	0.99995	5.4	0.17
Sr	0.99983	4.0	0.004
Mo	0.99528	1.4	0.001
Cd	>0.99999	3.0	0.003
Sb	0.99999	4.1	0.002
Ba	0.99978	2.0	0.005
Hg	0.99943	2.7	0.0006
Cu	0.99973	0.7	0.01
Sn	0.99990	1.3	0.002
Pb	0.99996	2.4	0.002

Method performance with real samples

Microwave-digested real samples of red meat of buffalo, chicken, and goat purchased from the local market were analyzed in KED mode for all elements. It was observed that lead in chicken and goat meat samples was higher than the maximum limit of 0.1 $mg \cdot kg^{-1}$. All other elements were within the maximum limits of regulatory requirements as mentioned in Table 1. Red meat of buffalo was selected for spiking experiments. The measured values are shown in Table 7.

Table 7. Measured values of all analyte elements in buffalo red meat, chicken, and goat samples

Element	Buffalo meat (mg·kg^{-1})	Chicken meat (mg·kg^{-1})	Goat meat (mg·kg^{-1})
Li	BLQ	BLQ	BLQ
Be	BLQ	BLQ	BLQ
B	BLQ	0.21	BLQ
Al	BLQ	BLQ	BLQ
V	BLQ	BLQ	BLQ
Cr	BLQ	0.76	BLQ
Mn	BLQ	BLQ	BLQ
Fe	22.0	10.0	13.1
Co	BLQ	BLQ	BLQ
Ni	BLQ	BLQ	BLQ
Cu	1.5	2.3	3.5
Zn	38.9	6.1	26.8
As	BLQ	BLQ	BLQ
Se	0.15	0.15	0.07
Sr	BLQ	BLQ	BLQ
Mo	BLQ	BLQ	BLQ
Cd	BLQ	BLQ	BLQ
Sn	0.07	0.11	0.2
Sb	BLQ	0.01	BLQ
Ba	BLQ	BLQ	BLQ
Hg	BLQ	BLQ	BLQ
Pb	0.04	0.18	0.21

Note: Refer to Table 8 for limit of quantification. (BLQ: Below limit of quantification)

To calculate the limit of quantification (LOQ) of all target elements, 23 method blanks were prepared as described in the sample preparation section and analyzed for all target elements, as described in the AOAC 2015.01 guideline. The standard deviation was calculated from the response of all method blanks. The limit of quantification (LOQ) was calculated as six times the standard deviation of the response of each target element. To determine the practical LOQs for all target elements in the matrix, buffalo red meat was spiked with concentrations in decreasing order. The LOQs were calculated by taking into account the recovery values (elements with recovery values greater than 80%) as well as repeatability (as RSD values lower than 10%). The LOQs determined are given in Table 8.

Table 8. Limit of quantification of meat and meat products

Element	LOQ (mg·kg^{-1})
Li	0.20
Be	0.20
B	0.20
Al	5.0
V	0.20
Cr	0.40
Mn	0.20
Fe	10
Co	0.0050
Ni	0.20
As	0.01
Se	0.20
Sr	0.40
Mo	0.20
Cd	0.005
Sb	0.01
Ba	0.40
Hg	0.01
Cu	0.4
Sn	0.02
Pb	0.04

Accuracy and precision

To demonstrate the accuracy and precision of the method, a spike recovery experiment was conducted. A set of three different level spiked samples, each in six replicates, including the LOQ, were analyzed for all the target elements. Three different levels of spike concentrations of all target elements are given in Table 9. The percent recoveries were calculated against the spiked concentration, and relative standard deviation (RSD) was calculated from the standard deviation of each of the six replicates of the spiked sample. The average % of recoveries and RSDs are tabulated below in Table 10. The observed recoveries were within 89–110% with RSD of <10%. The results are in alignment with the acceptance criteria of the AOAC 2015.01 guidelines.

Table 9. Spiked concentrations at three different levels for accuracy (% recovery) and precision (%RSD). Level 1 (L1) (LOQ), Level 2 (L2), and Level 3 (L3), in mg·kg^{-1}.

Element	Spike concentration level in mg·kg^{-1}		
	L1	L2	L3
Li, Be, B, V, Mn, Ni, Se, Mo	0.2	0.4	10.0
Al	5.0	10.0	100.0
Cr, Sr, Ba	0.4	1.0	10.0
Fe	10	25.0	100.0
Co, Cd	0.005	0.01	1.0
As, Sb	0.01	0.02	1.0
Hg	0.01	0.025	1.0
Pb, Sn	0.02	0.04	1.0
Cu	0.2	0.4	10.0

*Note: All the concentration values given in the table are calculated back to sample taken with a dilution factor of 100-fold.

Table 10. Method performance data. Accuracy (% recovery) and precision (%RSD) calculated for three different spike levels in buffalo meat.

Element	L1		L2		L3	
	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
Li	90.0	5.0	92.3	4.1	94.1	3.4
Be	91.2	7.9	95.0	2.5	95.1	0.6
B	93.7	6.6	98.7	3.5	97.2	3.4
Al	99.7	4.3	98.2	2.9	98.2	1.6
V	93.8	1.7	96.4	1.8	96.2	1.3
Cr	89.7	1.8	91.4	0.5	93.8	1.1
Mn	89.0	1.9	95.0	2.1	95.7	1.9
Fe	90.7	1.8	90.5	2.6	91.5	1.6
Co	91.4	1.8	97.9	1.4	93.9	1.7
Ni	90.1	2.0	90.3	2.7	90.7	1.4
As	100.0	6.7	106.4	3.4	109.7	1.7
Se	90.5	4.3	100.8	2.9	109.6	1.3
Sr	106.3	6.1	100.0	3.0	99.2	4.3
Mo	90.7	2.0	90.2	1.2	91.4	2.4
Cd	98.2	5.1	102.2	4.1	99.2	1.7
Sb	90.2	3.8	106.5	9.4	101.7	1.9
Ba	107.3	7.5	99.9	4.3	102.0	3.8
Hg	89.2	3.4	93.5	2.8	104.2	1.4
Cu	NA	NA	89.4	1.6	89.1	0.7
Pb	NA	NA	98.7	2.4	98.3	2.9
Sn	NA	NA	109.3	5.3	99.1	3.4

*NA: Incurred sample concentration is greater than the spiked concentration.

The optimized method was applied to chicken samples to increase the scope of the method. A total of 18 individual preparations of chicken meat were spiked at three different concentrations (L1, L2, and L3 of Table 9) and verified for recovery and precision. The observed % recoveries were within 89–107%, and the %RSDs were <10%. The average % recovery and %RSD results for chicken samples are presented in Table 11. In the chicken sample, lead, tin, copper, and chromium were present at the concentrations of 0.19, 0.11, 2.3, and 0.76 mg.kg^{-1}, respectively.

Table 11. Method performance data. Accuracy (% recovery) and precision (%RSD) calculated for three different spike levels in red meat of chicken.

Element	L1		L2		L3	
	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
Li	96.1	4.7	95.3	5.0	93.8	3.0
Be	97.6	6.2	96.4	2.7	95.7	2.2
B	90.2	9.4	91.0	8.0	96.0	2.7
Al	90.6	1.4	101.7	2.6	99.3	3.3
V	91.2	1.5	94.5	2.0	96.2	2.3
Mn	94.9	2.5	90.7	2.1	95.2	1.5
Fe	90.8	3.9	80.3	4.6	92.7	2.7
Co	95.0	1.5	89.4	1.4	91.1	1.5
Ni	88.8	2.1	91.6	1.8	90.1	2.4
Zn	90.9	5.9	90.5	1.5	96.1	1.4
As	105.2	7.2	105.9	4.9	107.0	1.3
Se	89.4	6.5	103.9	5.6	102.7	2.4
Sr	90.4	6.4	101.1	4.8	98.8	3.2
Mo	89.1	5.7	90.3	2.3	93.2	3.5
Cd	98.3	7.0	101.7	2.9	101.5	3.0
Ba	89.9	2.6	103.7	4.6	99.6	4.5
Hg	101.2	5.3	104.7	4.1	104.8	3.2
Cu	NA	NA	99.6	0.9	89.0	2.7
Pb	NA	NA	95.6	7.9	96.9	2.1
Sn	NA	NA	104.4	6.4	102.3	1.9

*NA: Incurred sample concentration is greater than the spiked concentration.

The method was applied and the samples were analyzed for all the target elements. The measured results are shown in Table 12. It was observed that lead was above the maximum limit of 0.1 mg·kg^{-1} in the red meat (goat) and kidney of the goat. Chromium also exceeded the maximum limit of 1.0 mg·kg^{-1} mentioned as per the CFDA in red meat of goat.

Table 12. Measured values of all elements in red meat of goat analyzed with offal (goat kidney and goat liver)

Element	Goat meat (mg·kg^{-1})	Goat kidney (mg·kg^{-1})	Goat liver (mg·kg^{-1})
Li	BLQ	BLQ	BLQ
Be	BLQ	BLQ	BLQ
B	BLQ	BLQ	BLQ
Al	BLQ	BLQ	BLQ
V	BLQ	BLQ	BLQ
Cr	2.0	BLQ	BLQ
Mn	0.2	1.2	3.6
Fe	27.4	48.8	79.3
Co	BLQ	0.03	0.03
Ni	BLQ	BLQ	BLQ
Cu	5.1	3.6	11.1
Zn	17.6	20.0	24.4
As	BLQ	0.010	BLQ
Se	0.3	1.3	0.5
Sr	BLQ	0.6	BLQ
Mo	BLQ	0.3	0.9
Cd	BLQ	BLQ	BLQ
Sn	0.2	0.02	BLQ
Sb	BLQ	BLQ	BLQ
Ba	BLQ	0.3	BLQ
Hg	BLQ	0.01	0.01
Pb	0.59	0.14	0.07

Method robustness

The concentration of elements measured in the meat samples is prone to the influences of physical interferences, such as viscosity differences between the standard and samples, presence of undigested carbon, and matrix components that suppress or enhance the analyte signal, due to the presence of high dissolved salts. These effects should be compensated for throughout the analysis and corrected in the final results. The internal standard elements, which should be close to analyte elements in terms of mass and ionization potential, will behave similarly in the plasma and mass analyzer as compared to the analytes. The robustness of the method to the presence of such matrix effects was demonstrated by adding 0.02 mg·L^{-1} of internal standard elements as the final concentration in all standard and sample solutions. The % recovery of internal standard elements was obtained using the Qtegra ISDS Software and compared with the criteria set by AOAC. 2015.01.

The response of internal standard (0.02 mg·L^{-1}) during a sequence of meat samples showed excellent robustness. The internal standard elements showed recoveries in the range of 83–120% against the acceptance criteria of 60–125% (AOAC 2015.01). The internal standard recoveries oberserved for Sc, Ge, In, Rh, Tb, Ir, and Bi are shown in Figure 2.

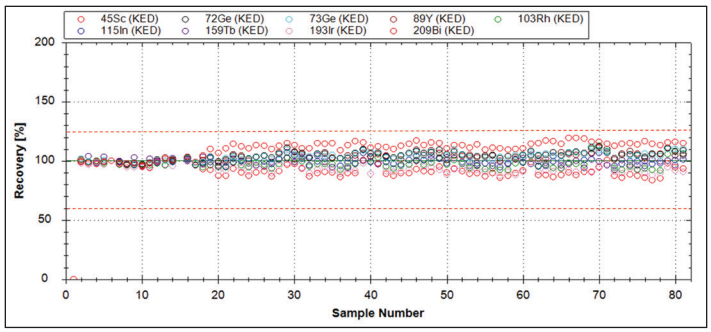

Figure 2. % Recovery of the internal standards in meat samples (n=42)

After every six replicates of spiked samples, CCV standards were analyzed to qualify the accuracy of sequence run and to ensure that there were no signal or intensity drifts throughout the analysis of recovery studies. One of the standard solutions from the seven linearity standards was analyzed (calibration point 3) as an intermediate quality control sample and % recovery was calculated. Using Qtegra ISDS Software the quality control criteria were set as 85–115% (red dotted lines in Figure 3) as per AOAC 2015.01 criteria. The results were calculated and the obtained recoveries were within the acceptance criteria of 85–115% of AOAC 2015.01. This showed that there is no signal drift beyond the limit of 85–115% and ensures the quality of data. The observed percentage recoveries of all elements are shown in Figure 3.

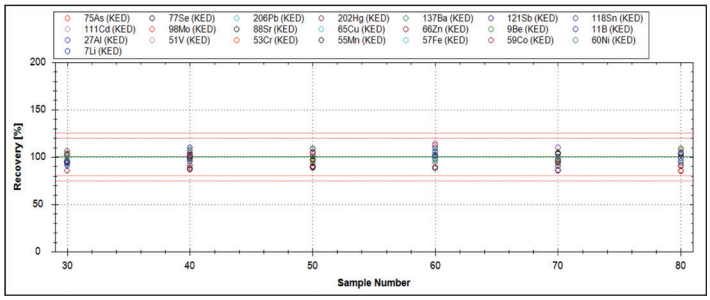

Figure 3. % Recovery of CCV standards in the meat method sequence (continuing calibration verification)

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Conclusions

- The experiments performed demonstrate that the Thermo Scientific iCAP RQ ICP-MS system operated in KED mode allows for robust and sensitive trace elemental analysis of meat and meat products in combination with microwave digestion for sample preparation.
- The method performance met all the analytical criteria of AOAC 2015.01 and demonstrated excellent linearity, specificity, the limit of quantifications, recovery, precision, and robustness.
- This analytical method provides an excellent solution for commercial laboratories aiming to achieve high-throughput sample analysis with no compromise in data quality.

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