



# Inductively Coupled Plasma – Mass Spectrometry

**Piyush Deokar, Ph. D.**  
Product Manager – TEA & IOMS (CHNS)

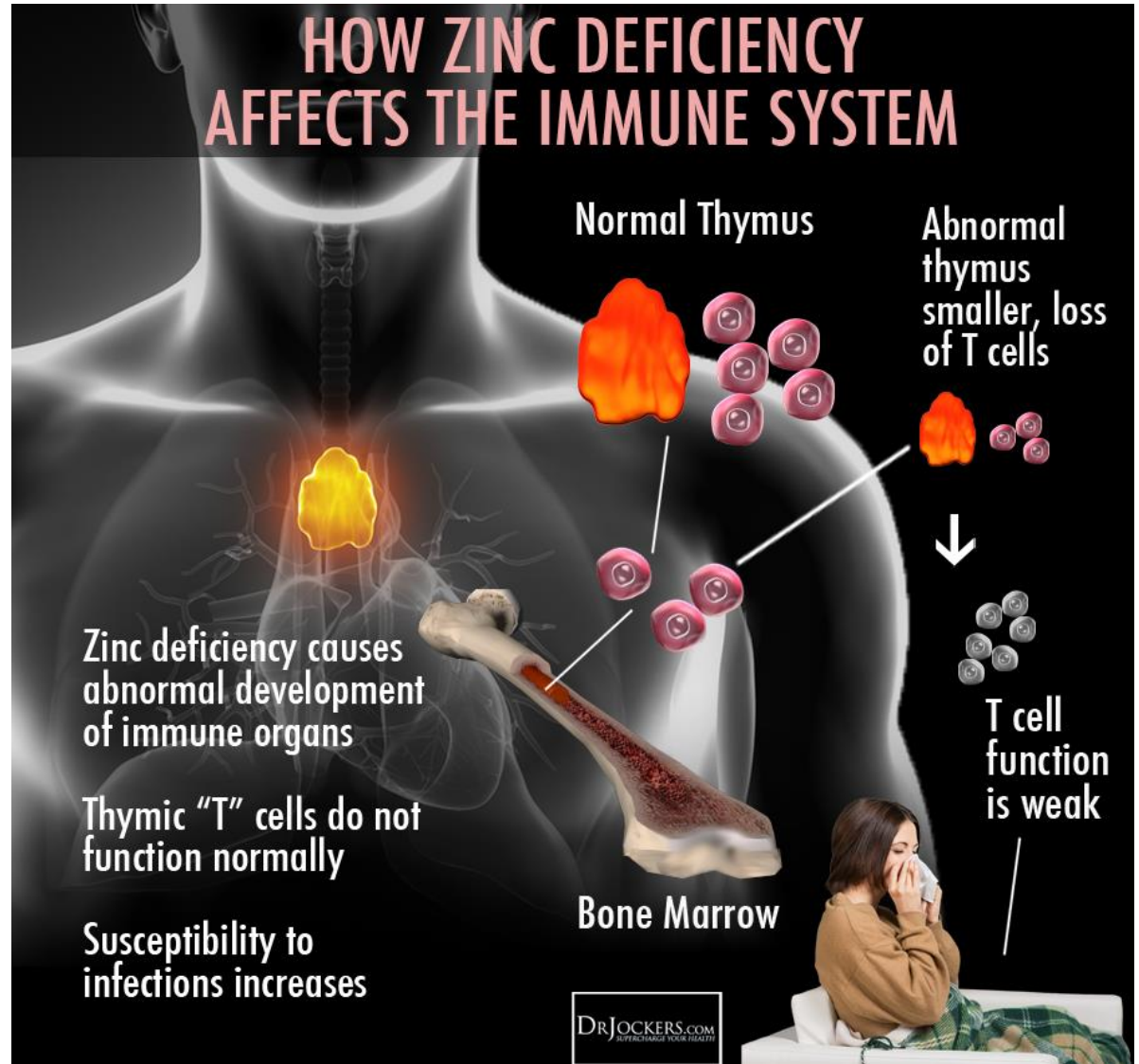
**Suresh Murugesan**  
Application Development Scientist,  
Food Safety Solution Center (FSSC) Ghaziabad- India

# Agenda

- 1 Elemental Analysis & Sample Preparation
- 2 Basics of ICP-MS
- 3 Interference Removal
- 4 Speciation Studies
- 5 Application

# Minerals in Day to Day Life

- **Calcium** helps to build strong bones and teeth and regulate your heartbeat. It also ensures your blood clots normally, important for healing.
- **Iron** helps your body make red blood cells to carry oxygen around your body.
- **Potassium** helps the body control the balance of fluids and keeps your heart healthy and functioning correctly.
- **Trace elements** are also essential nutrients, however, you need them in smaller amounts than vitamins and minerals.
- **Iodine** helps your body make the thyroid hormones that keep your cells and metabolic rate healthy.
- **Zinc** helps your body make new cells and enzymes, process carbohydrate, fat and protein in food and also with the healing of wounds

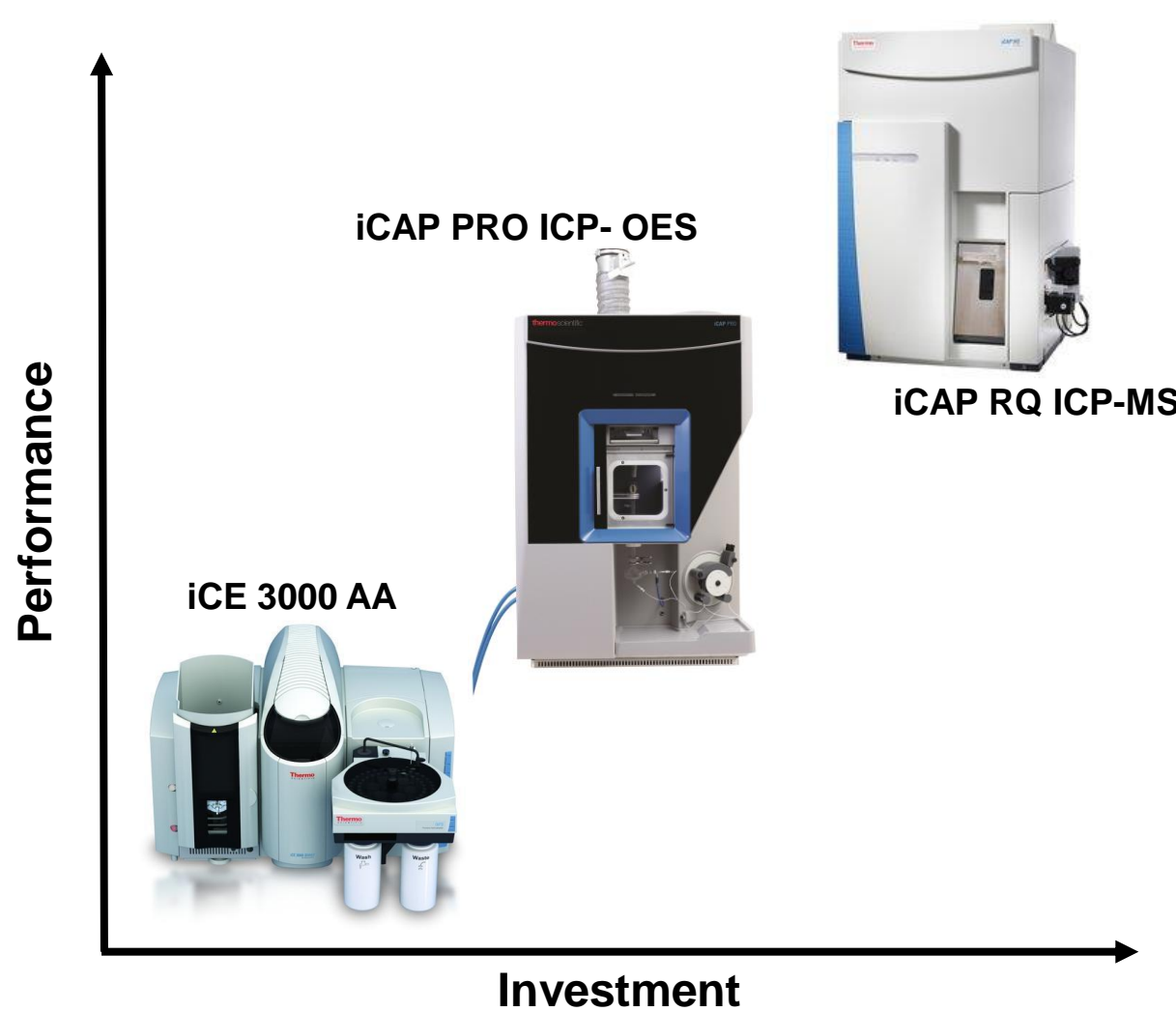


# Metal Toxicity

- Lead: Brain/ Kidney damage/Central Nervous system
- Cadmium: Lung Cancer/ Stomach Irritation/Prostate
- Chromium <sup>+6</sup>: Carcinogenic in lungs/Dermatitis
- Arsenic: Skin Damage/Carcinogenic/Chromosomes
- Mercury: Nervous Centre -Brain/Kidney/Fetuses



# Elemental Analysis & Sample Requirement



## How the Samples should be?

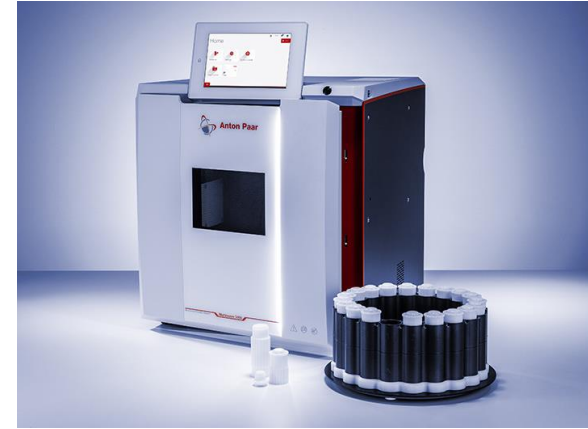
- Homogeneous
- Representative of Bulk
- Free of suspended particles
- Free flowing
- Uniform Introduction of Standards / Samples

## Acid Digestion



- Preferred for sample preparation for AAS – flame
- Larger Sample quantity
- Total digestion time is between 30 min. to 2 hours;
- Digestion temperature ~130deg c;
- Corrosive acidic fumes are evolved
- Volatiles may be lost
- Close supervision is normally required, to prevent charring of samples.

## Microwave Digestion



- Fast, about 30 - 40 minutes;
- Digestion Temp. up to 280 deg c
- Up to 48 samples can be digested at any time
- Minimum volatiles losses and contamination
- Pressures range from 20 - 80 bars
- Suitable for organics as well as Inorganics
- Fully automatic, user's supervision not required

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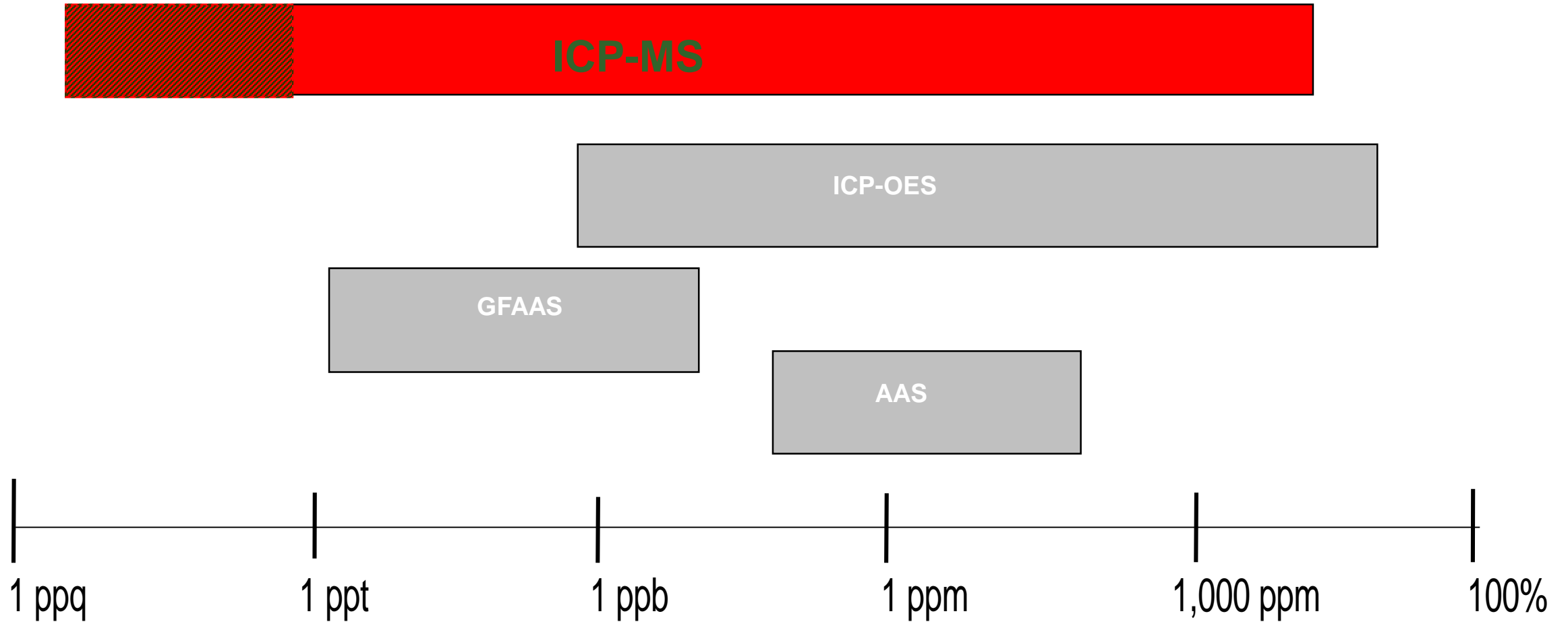
# The Periodic Table; Our Common Language

<i>H</i>																			<i>He</i>
<i>Li</i>	<i>Be</i>											<i>B</i>	<i>C</i>	<i>N</i>	<i>O</i>	<i>F</i>			<i>Ne</i>
<i>Na</i>	<i>Mg</i>											<i>Al</i>	<i>Si</i>	<i>P</i>	<i>S</i>	<i>Cl</i>			<i>Ar</i>
<i>K</i>	<i>Ca</i>	<i>Sc</i>	<i>Ti</i>	<i>V</i>	<i>Cr</i>	<i>Mn</i>	<i>Fe</i>	<i>Co</i>	<i>Ni</i>	<i>Cu</i>	<i>Zn</i>	<i>Ga</i>	<i>Ge</i>	<i>As</i>	<i>Se</i>	<i>Br</i>			<i>Kr</i>
<i>Rb</i>	<i>Sr</i>	<i>Y</i>	<i>Zr</i>	<i>Nb</i>	<i>Mo</i>	<i>Tc</i>	<i>Ru</i>	<i>Rh</i>	<i>Pd</i>	<i>Ag</i>	<i>Cd</i>	<i>In</i>	<i>Sn</i>	<i>Sb</i>	<i>Te</i>	<i>I</i>			<i>Xe</i>
<i>Cs</i>	<i>Ba</i>	<i>La</i>	<i>Hf</i>	<i>Ta</i>	<i>W</i>	<i>Re</i>	<i>Os</i>	<i>Ir</i>	<i>Pt</i>	<i>Au</i>	<i>Hg</i>	<i>Tl</i>	<i>Pb</i>	<i>Bi</i>	<i>Po</i>	<i>At</i>			<i>Rn</i>
<i>Fr</i>	<i>Ra</i>	<i>Ac</i>																	
			<i>Ce</i>	<i>Pr</i>	<i>Nd</i>	<i>Pm</i>	<i>Sm</i>	<i>Eu</i>	<i>Gd</i>	<i>Tb</i>	<i>Dy</i>	<i>Ho</i>	<i>Er</i>	<i>Tm</i>	<i>Yb</i>	<i>Lu</i>			
			<i>Th</i>	<i>Pa</i>	<i>U</i>	<i>Np</i>	<i>Pu</i>	<i>Am</i>	<i>Cm</i>	<i>Bk</i>	<i>Cf</i>	<i>Es</i>	<i>Fm</i>	<i>Md</i>	<i>No</i>	<i>Lw</i>			

- AA/ICP/ICP-MS
- ICP/ICP-MS
- ICP-MS
- Beyond the scope of this technique
- Unstable elements

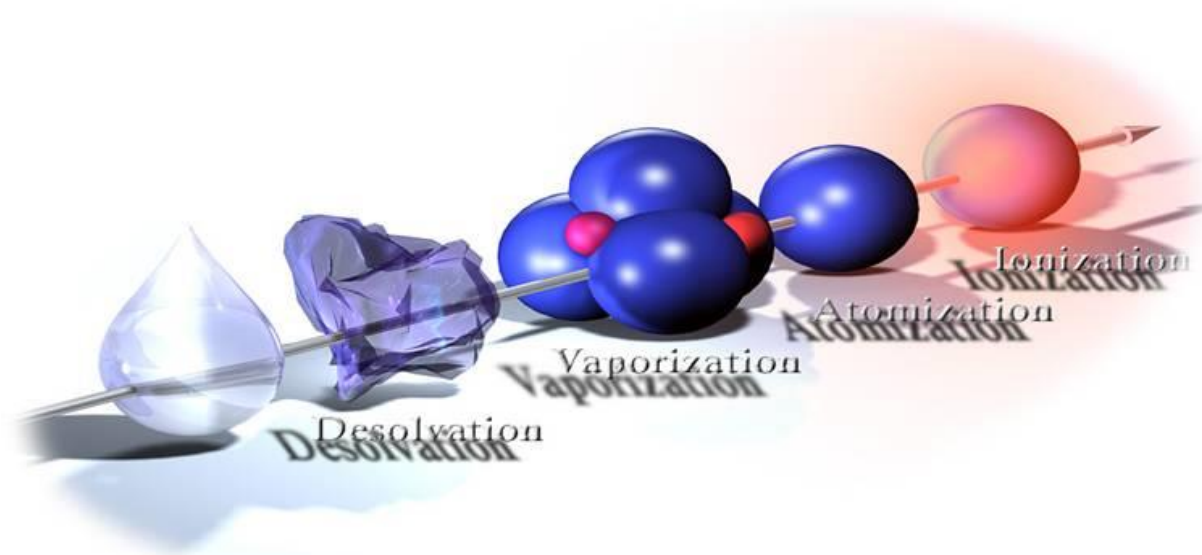


# Dynamic Range Comparison



# Inductively Coupled Plasma

- High voltage spark acts as initial ion generator
- The RF field causes the gaseous ions to oscillate with the field which results in heat.
- The temperature developed inside a plasma reaches 10,000 K.
- A plasma is a stream of highly ionized gas containing an equal number of electrons and positive ions



Nebulizer Gas

Coolant Gas

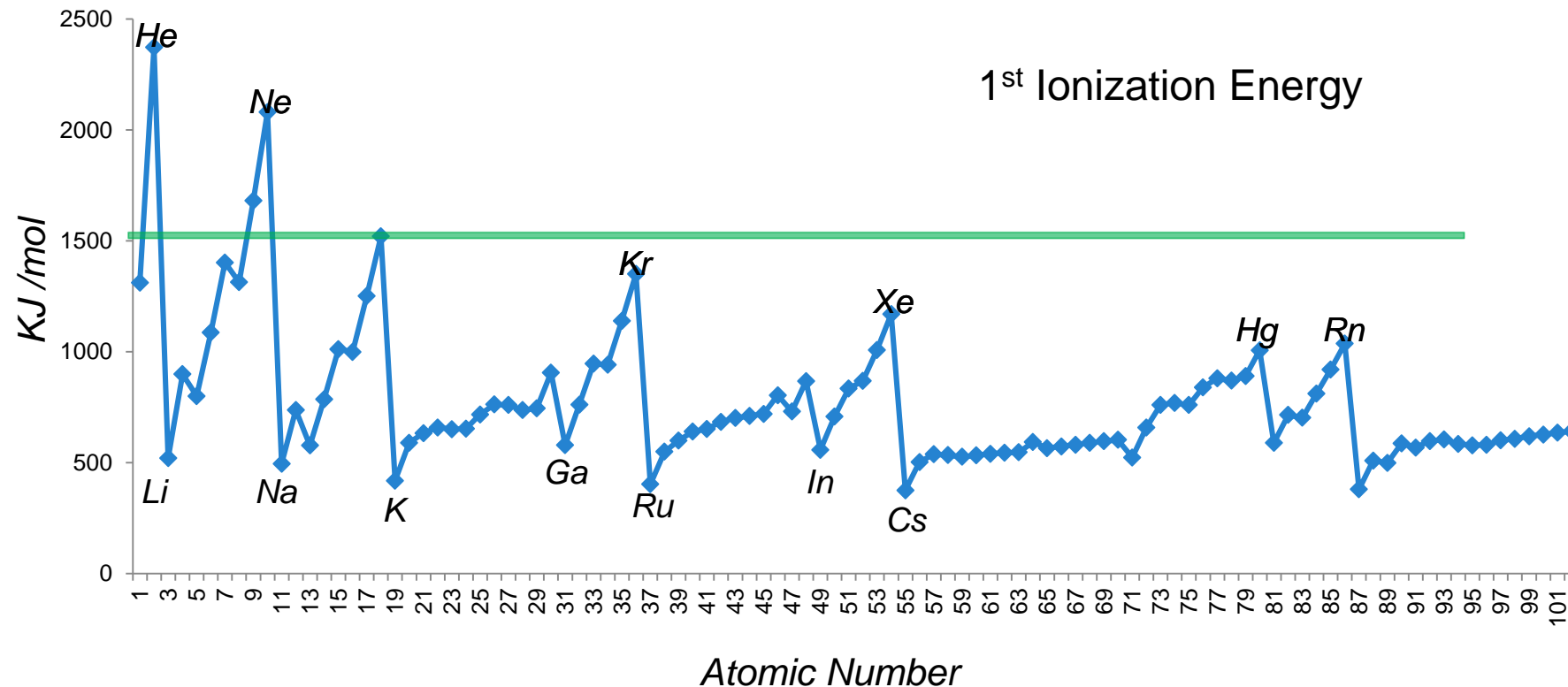
Auxiliary Gas



# Why Argon?

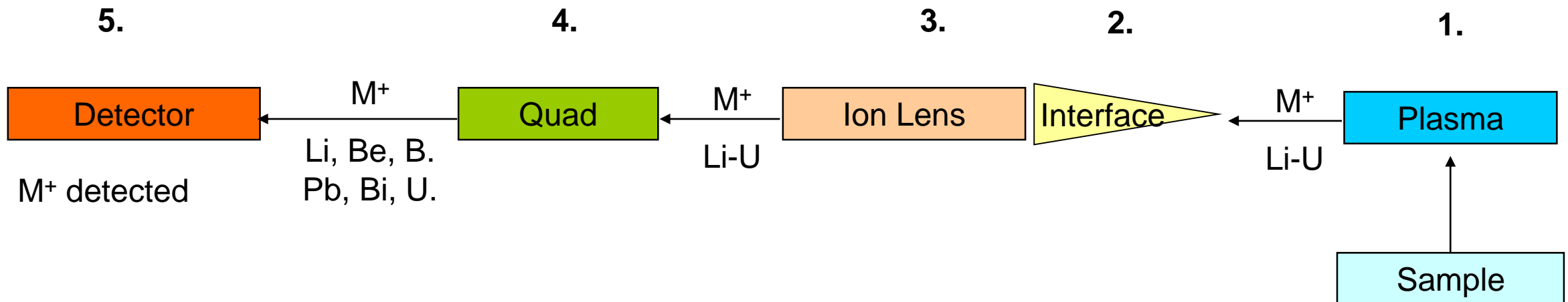
Argon has an ionization energy of ~ 1500 KJ/mol

4<sup>th</sup> highest ionization energy in the periodic table after helium, neon, & fluorine



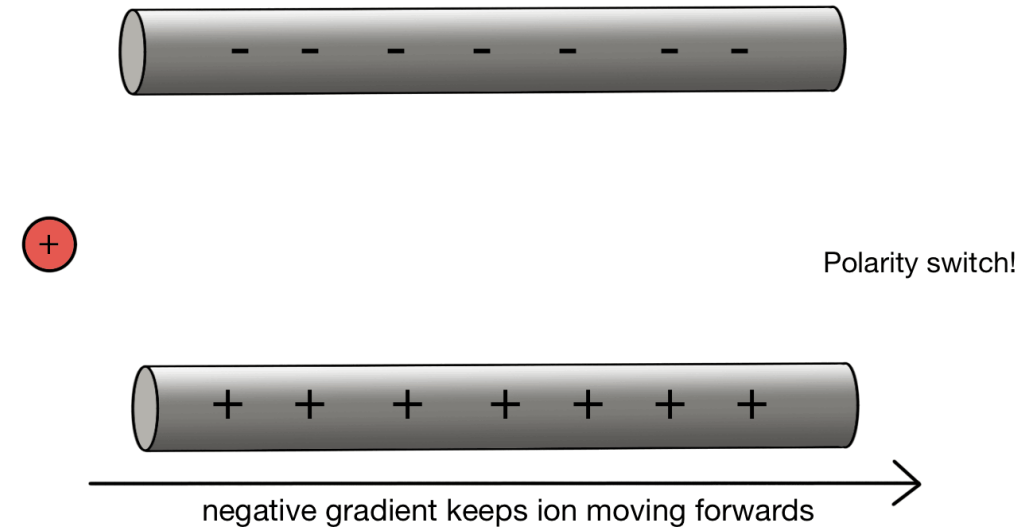
# The ICP-MS Technique

- 5 Basic Stages
  - 1. Sample Introduction and Ion Generation
  - 2. Ion Sampling (Ion Extraction)
  - 3. Ion Focusing
  - 4. Separation of Analyte Ions in Quadrupole Mass Filter
  - 5. Ion Detection

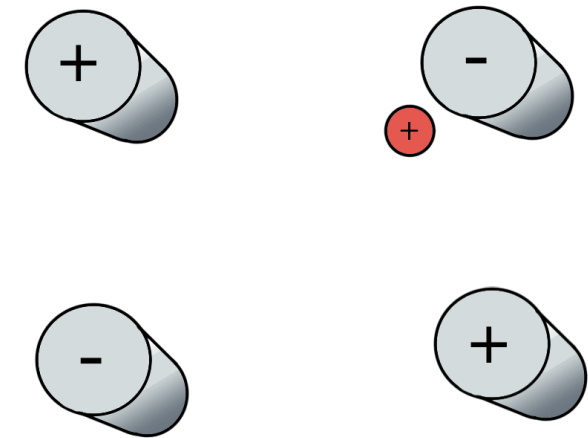


# Quadrupole Mass Filter

If we have two poles and switch polarity as the ion approaches, then we control how the ion moves.



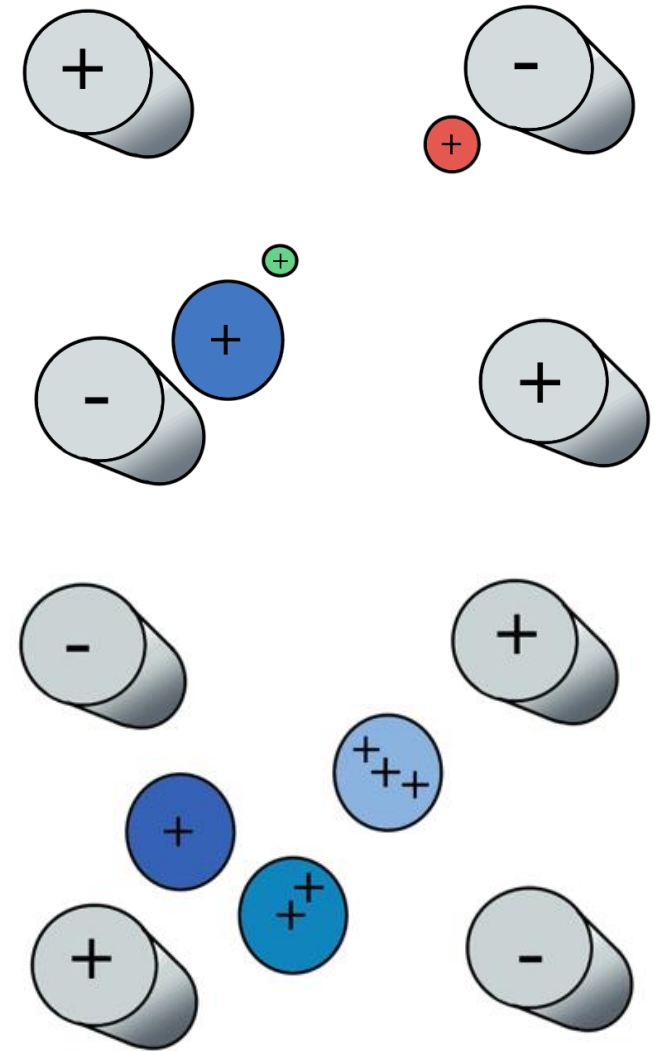
But we need to control how an ion moves in 3D space,  
Four poles work well for this



# Quadrupole Mass Filter

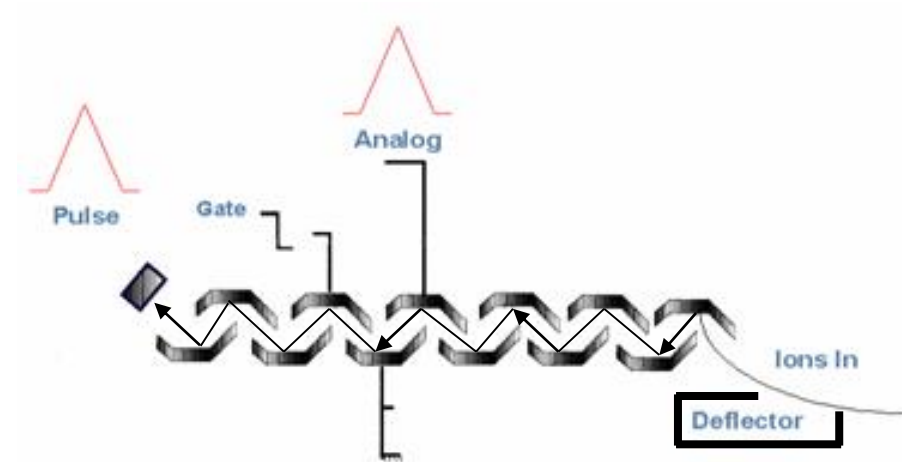
So if we have three ions of different  $m/z$  in the quadrupole, they will have different stable paths

It is the mass to charge ratio ( $m/z$ ) that determines how an ion moves in an electromagnetic field

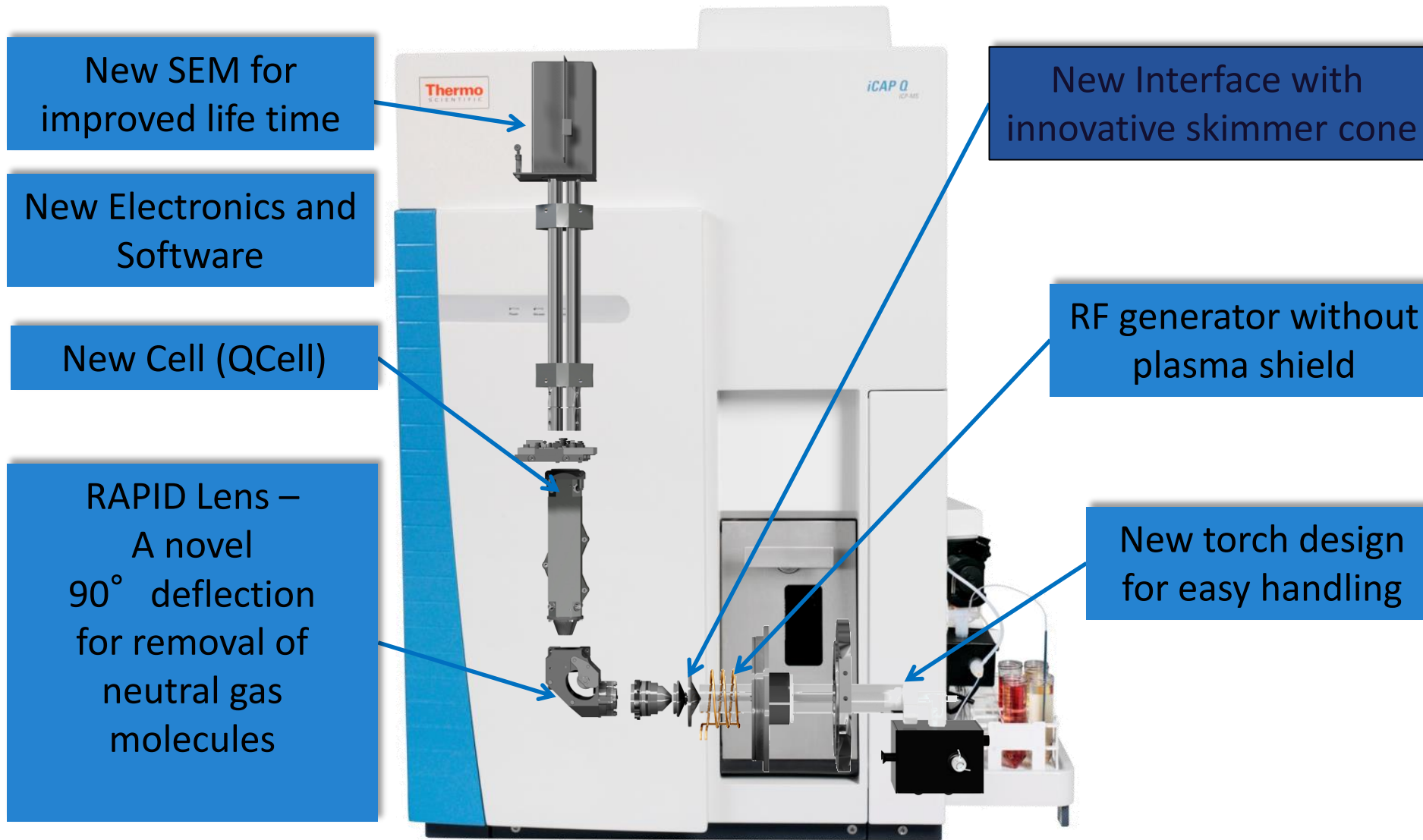


# Ion detection

- Ions leaving the quadrupole can't be detected directly - their signal must first be amplified
- Most instruments use 'discrete dynode' ion multipliers to achieve this



# Schematics



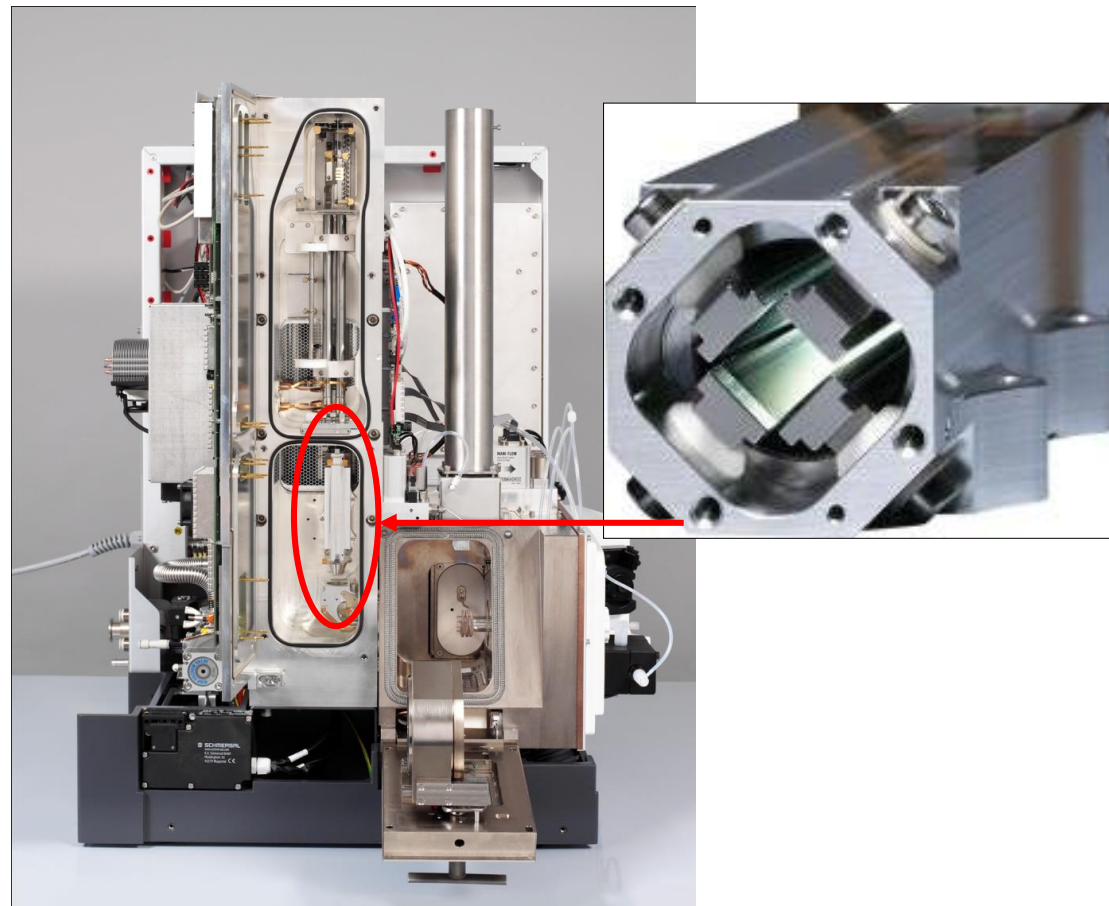
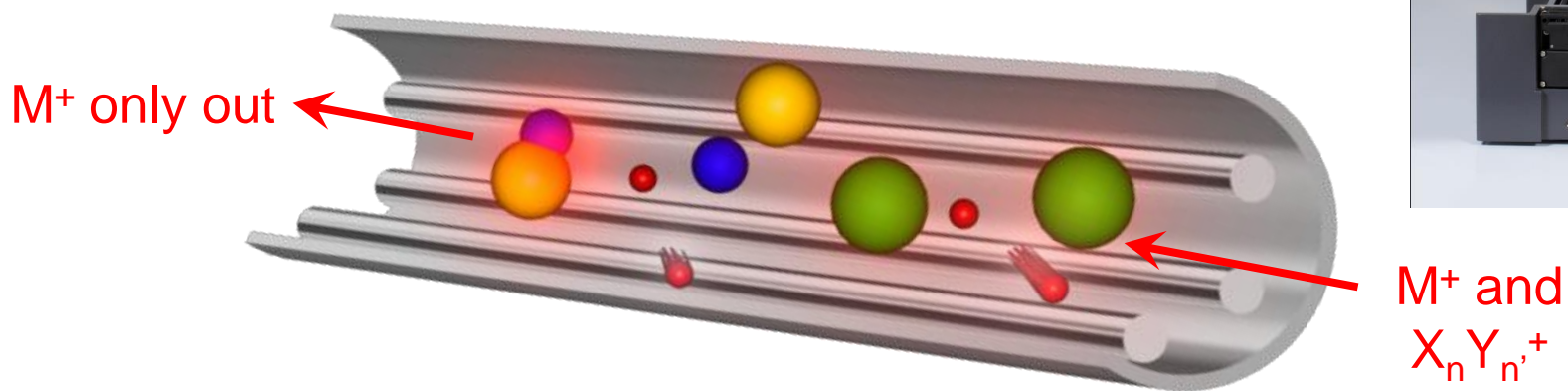


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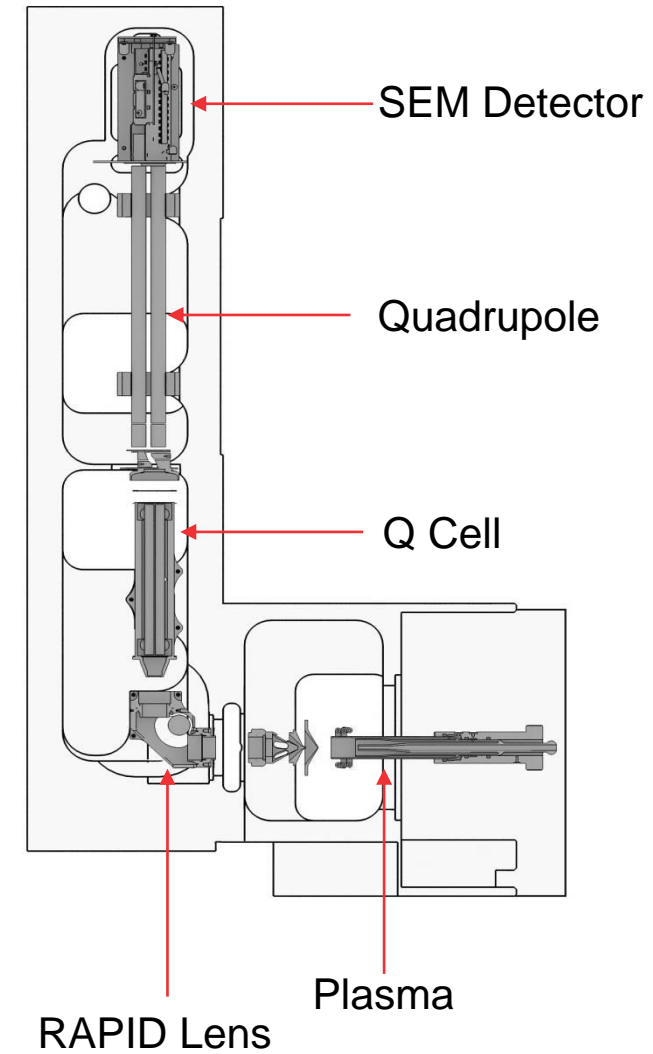
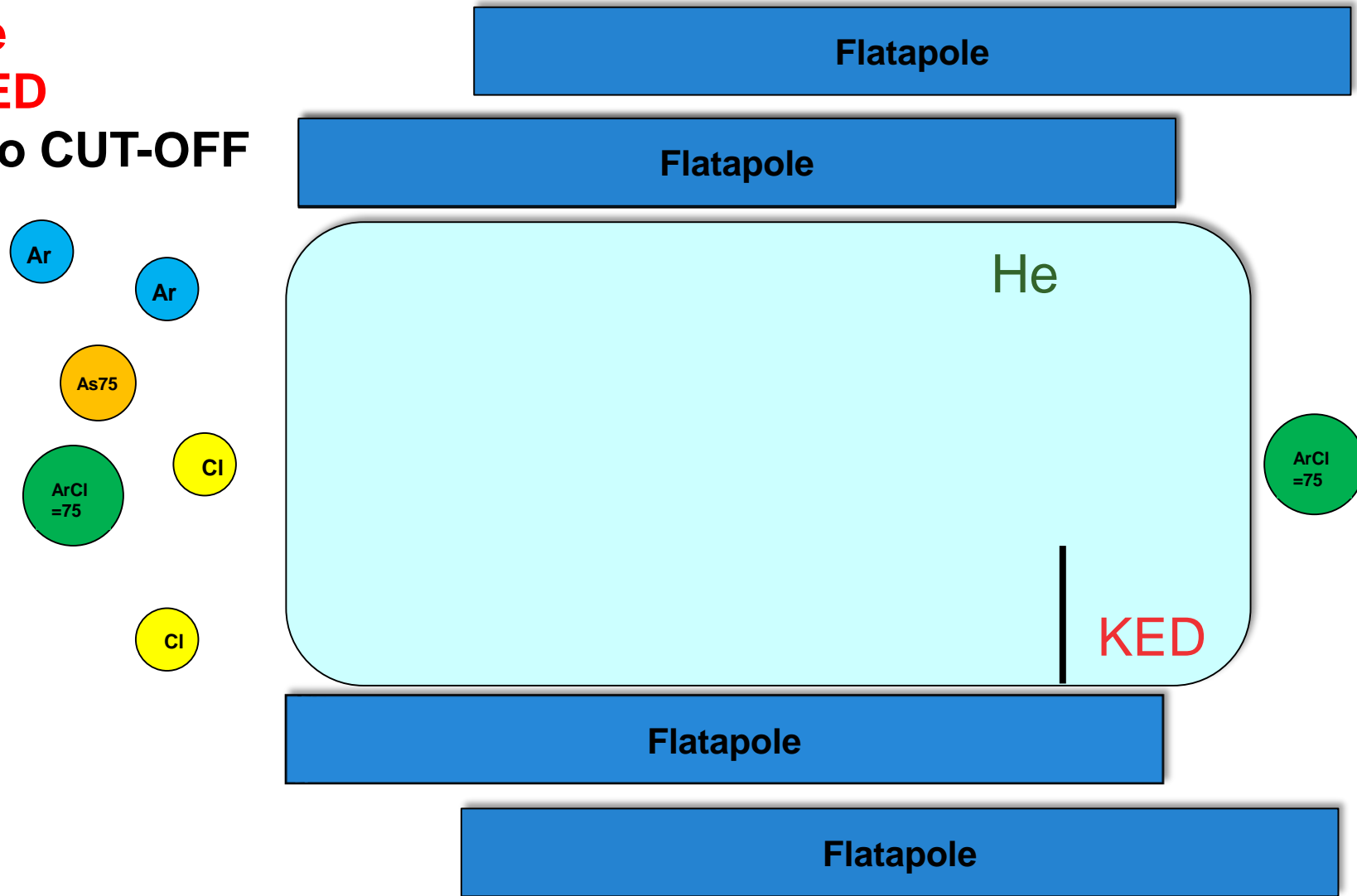
# Collision-Reaction Cell (CRC) Technology

- A multipole enclosed in a cylinder
- Controlled flow of gas into the cell
- Interaction of ions with the gas
- If reactive gas used, reactions occur
- All cells are reaction cells



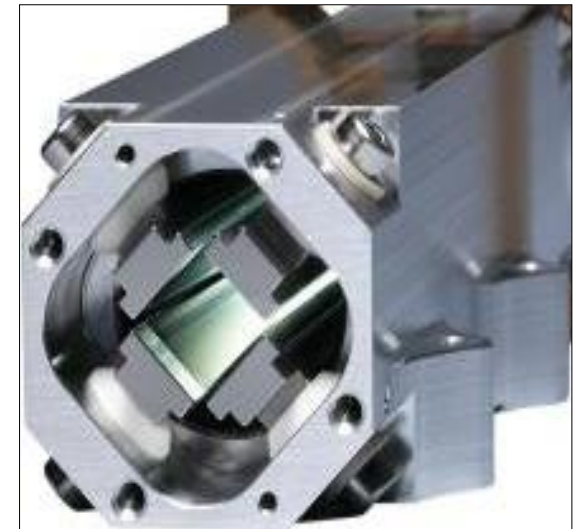
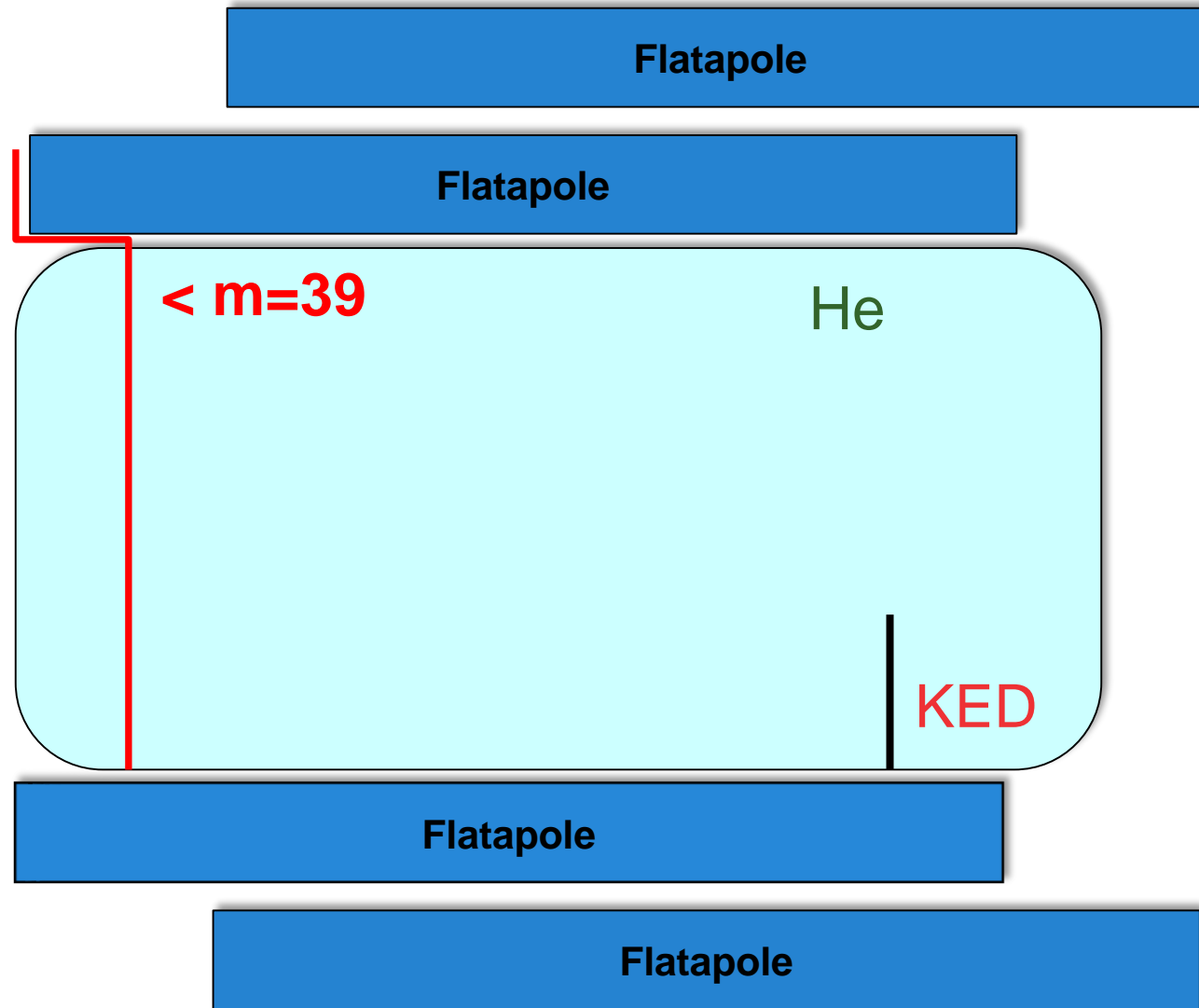
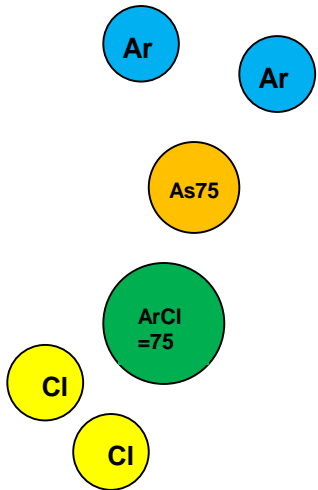
# Collision Cell Without Low-Mass Cut Off

He  
KED  
W/o CUT-OFF



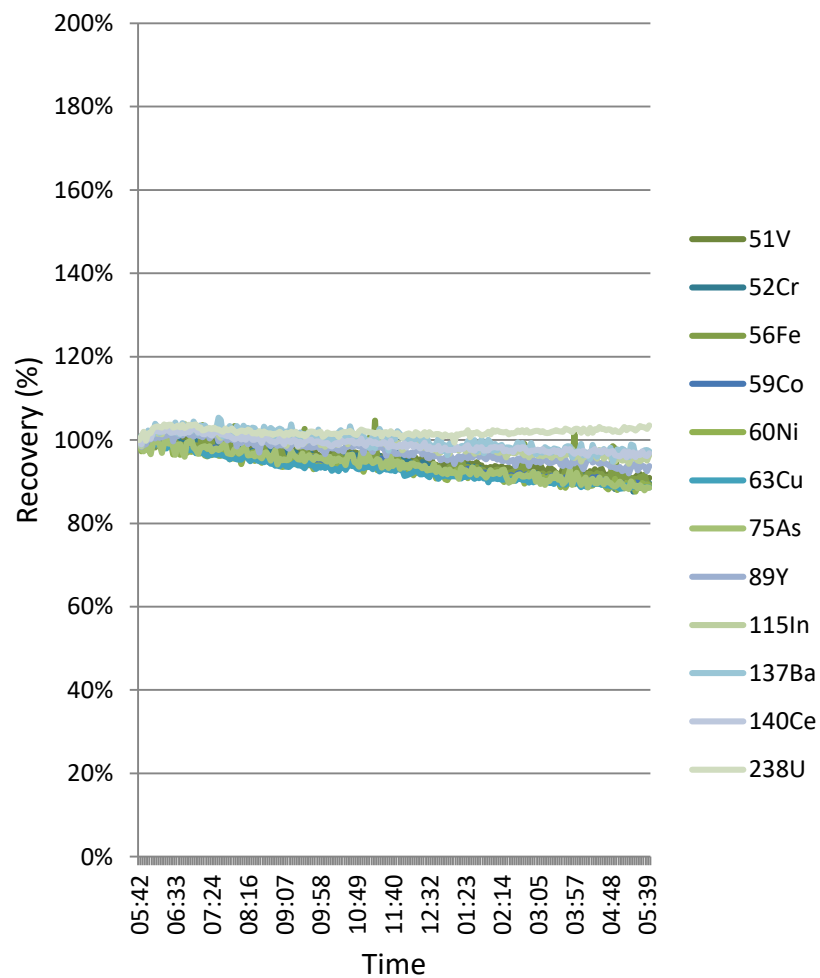
# Collision Cell Without Low-Mass Cutoff

He  
KED  
CUT-OFF

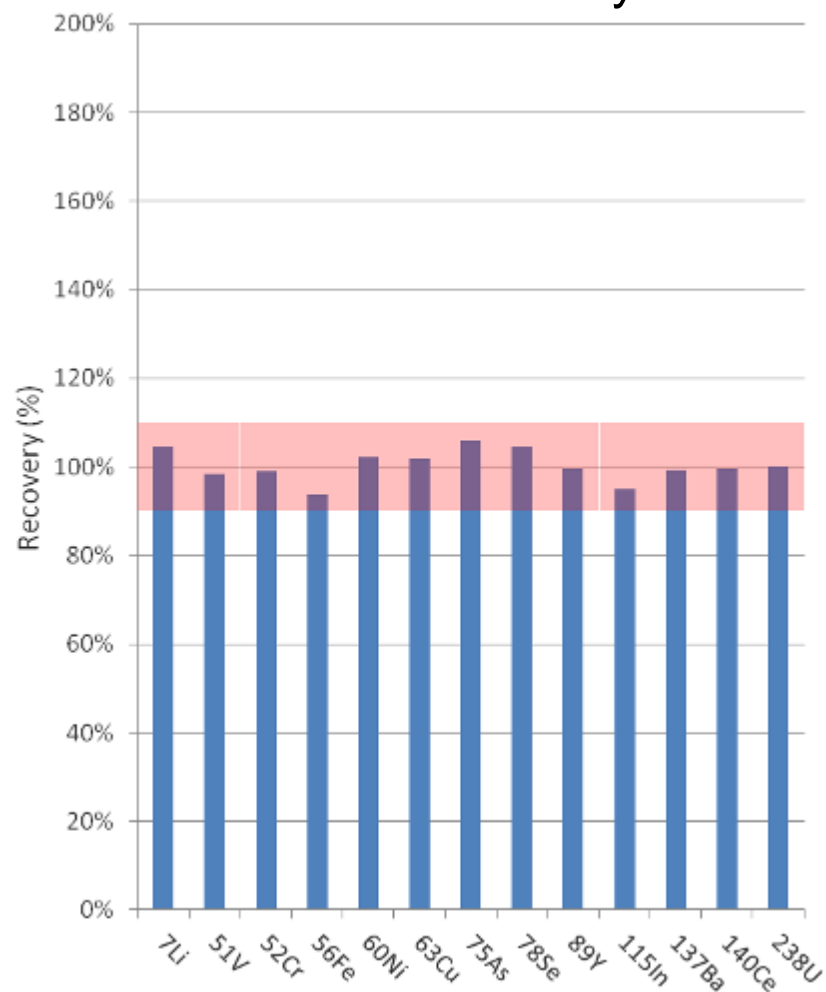


# Use of 100% Organic Solvents

## Stability over 12 Hours All <5% RSD



## Spike Recovery (1ppb) in 100% ACN) All 90-110% Recovery



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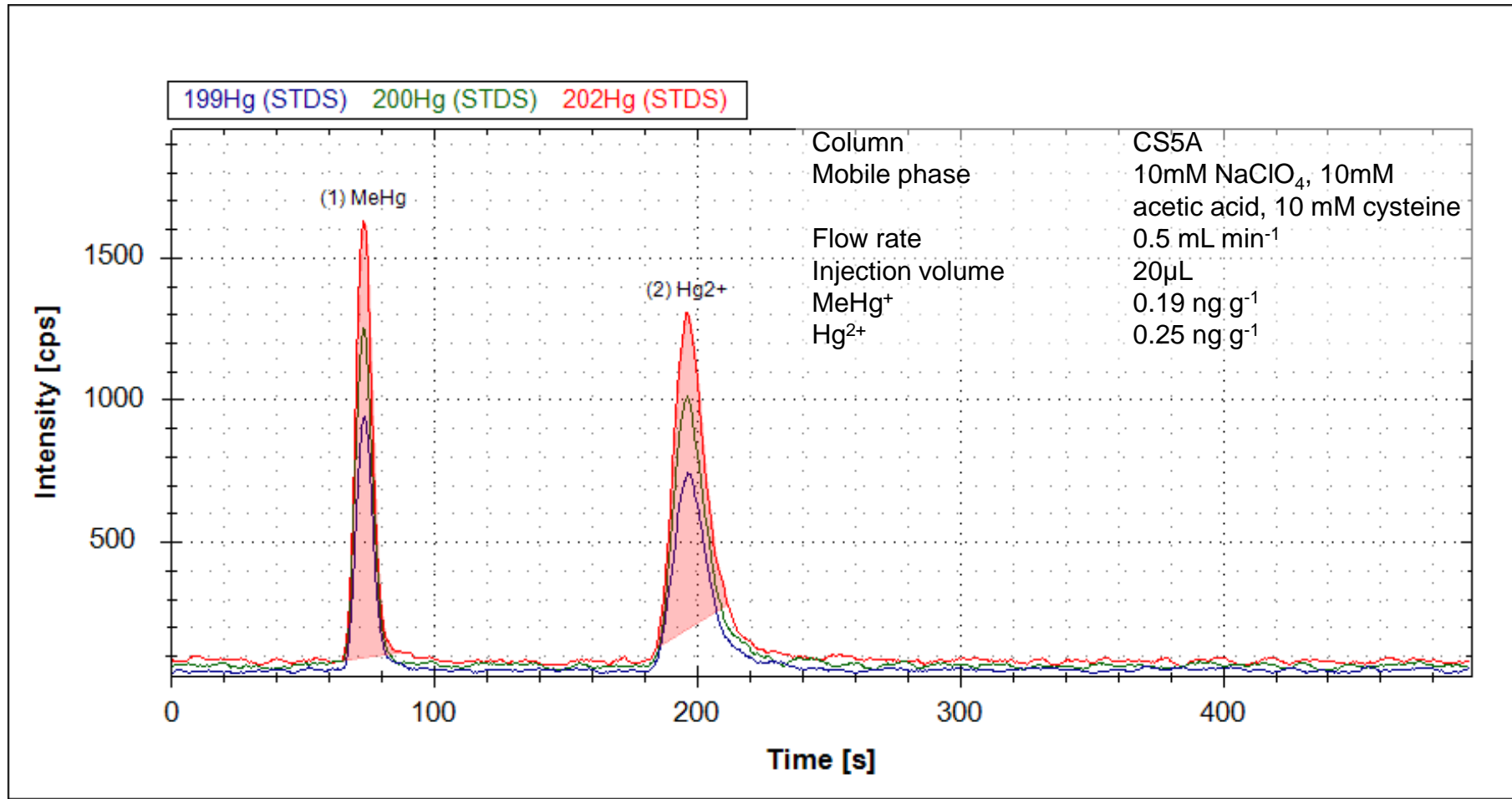
# Hyphenation Technique: IC-ICP-MS



- Metal-free systems
- Powerful separation chemistries
- Reagent-Free Ion Chromatography (RFIC)
- Extensive IC product line for full flexibility



- **MeHg<sup>+</sup> and Hg<sup>2+</sup> standards**





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# Method Performance in Different food matrices



**Rice**



**Wheat**



**Milk**



**Paneer**

**Butter**



**Meat**



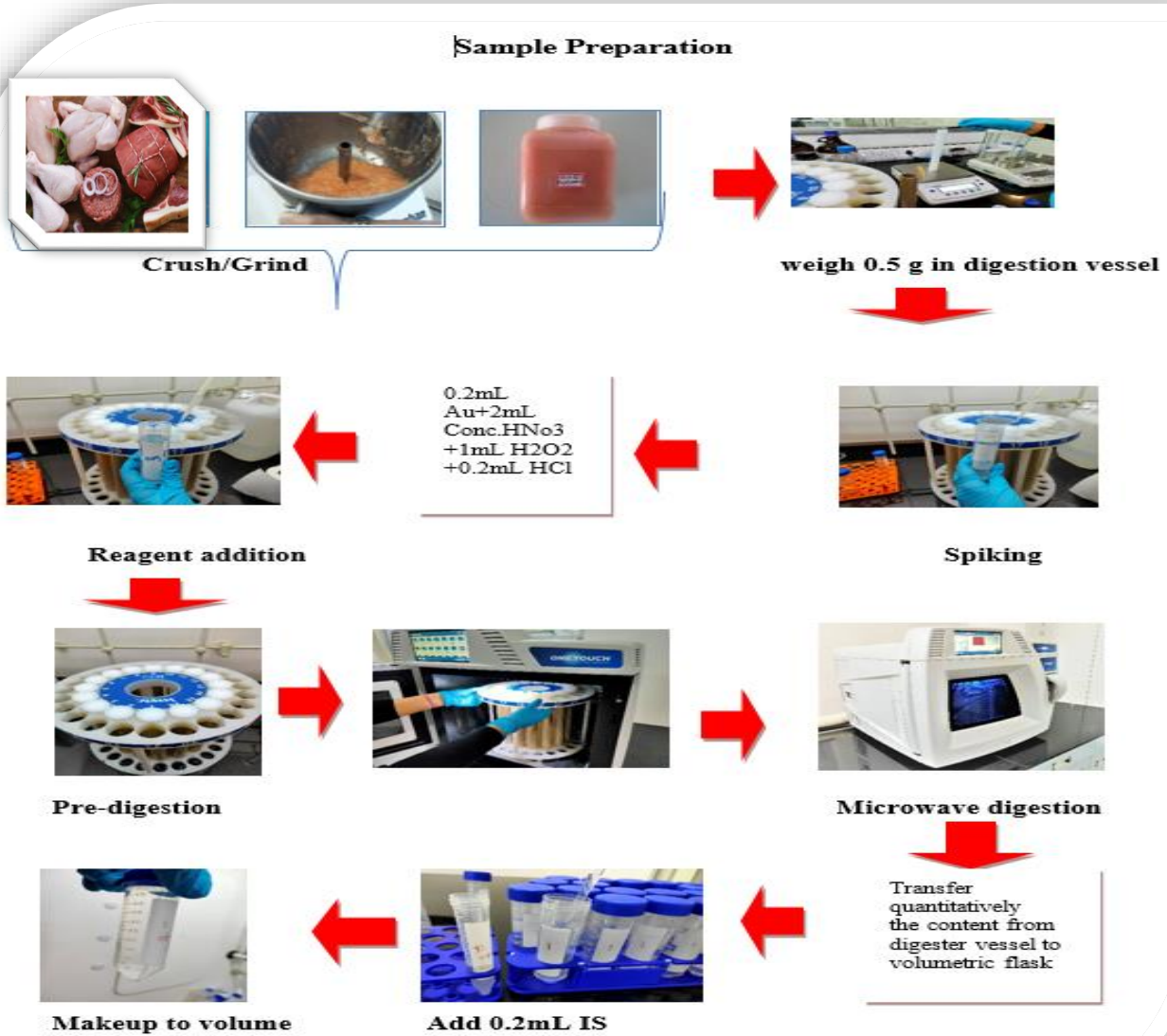
**Spices**



**Fruits and Vegetables**



# Sample Preparation



Parameters	Rice	Wheat	Milk and milk products	Meat and Meat products	Spices	Fruits and vegetables
sample weight (g)	0.25	0.25	0.5	0.5	0.2	0.5
Final volume made up (mL)	50	50	50	50	50	50
DI water (mL)	1	1	1	NA	1	NA
HNO <sub>3</sub> (mL)	4	2	2	2	2	2
H <sub>2</sub> O <sub>2</sub> (mL)	2	1	1	1	1	1
HCl (mL)	0.4	0.2	0.2	0.2	0.2	0.2
Gold solution (ug/L)	200	200	200	200	200	200
Internal standard mix	20	20	20	20	20	20
Pre-digestion time (min)	10	10	10	60	60	60

# Target Analytes

The periodic table is color-coded by groups: Group 1 and 2 (blue), Groups 13-18 (green), Groups 3-10 (red), Groups 11-12 (pink), and Groups 3-10 (yellow). Target analytes are highlighted with red circles: Li, Be, Na, Mg, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Cd, Hg, B, C, N, O, F, Ne, Al, Si, P, S, Cl, Ar, As, Se, Br, Kr, Sr, Mo, Sb, Te, I, Xe, Ba, Pb, Bi, Po, At, Rn, and Fr, Ra, Ac. Other target analytes are highlighted with blue circles: Cr, Cu, Cd, Hg, Sn, and Pb.

H																			He
Li	Be											B	C	N	O	F		Ne	
Na	Mg											Al	Si	P	S	Cl		Ar	
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br		Kr	
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I		Xe	
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At		Rn	
Fr	Ra	Ac																	
			Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb			Lu	
			Th	Pa	U	Np	Pu	Am	C	Bk	Cf	Es	Fm	Md	No			Lr	

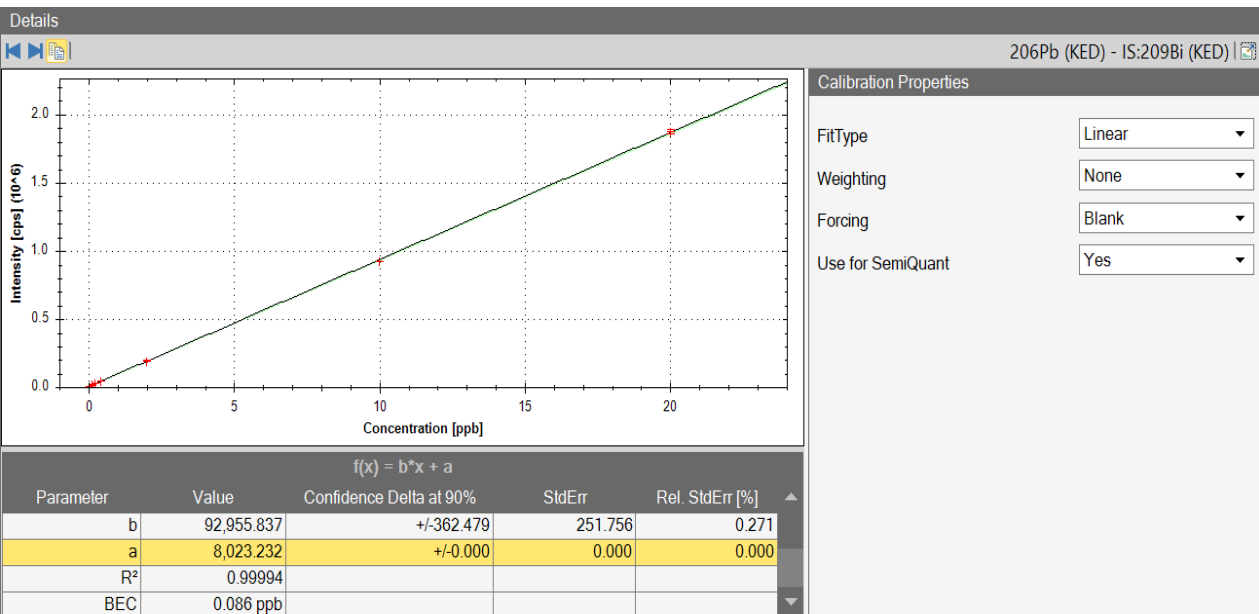
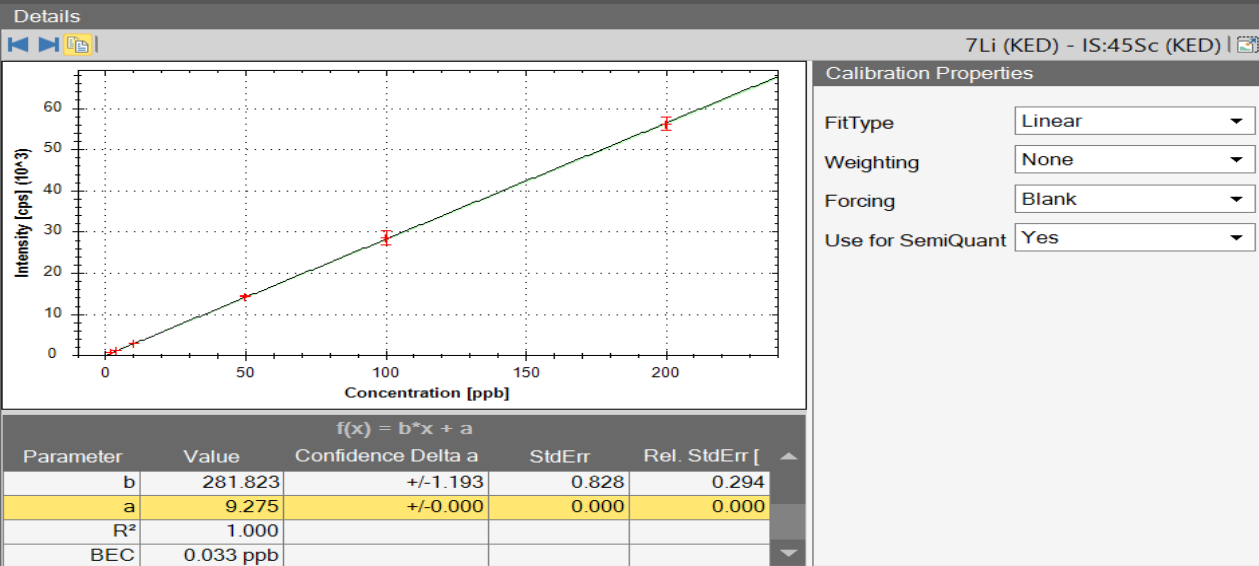
# Linearity

Elements	Working standard concentration (mg.L <sup>-1</sup> )	Volume made up (mL)	Stock solution/certified standard (mg.L <sup>-1</sup> )	Volume taken (mL)
Pb, Sb	0.1	25	10	0.25
Al, Fe, Cu, Zn, Sn	30		1000	0.75
Hg, Co	0.02		1	0.5
Cr, As, Se	0.5		10	1.25
Li, B, Be, V, Mn, Ni, Mo, Ba	1		10	2.5
Cd	0.05		10	0.125



Elements	Std-1	Std-2	Std-3	Std-4	Std-5	Std-6	Std-7
Pb, Sb	0.05	0.1	0.25	0.5	1	2	4
Al, Fe, Cu, Zn, Sn	15	30	75	150	300	600	1200
Hg, Co	0.01	0.02	0.05	0.1	0.2	0.4	0.8
Cr, As, Se	0.25	0.5	1.25	2.5	5	10	20
Li, B, Be, V, Mn, Ni, Mo, Ba	0.5	1	2.5	5	10	20	40
Cd	0.025	0.05	0.125	0.25	0.5	1	2
Volume prepared (mL)	25	25	25	25	25	25	25
Volume taken from mixed working standard (µL)	12.5	25	62.5	125	250	500	1000

# Linearity

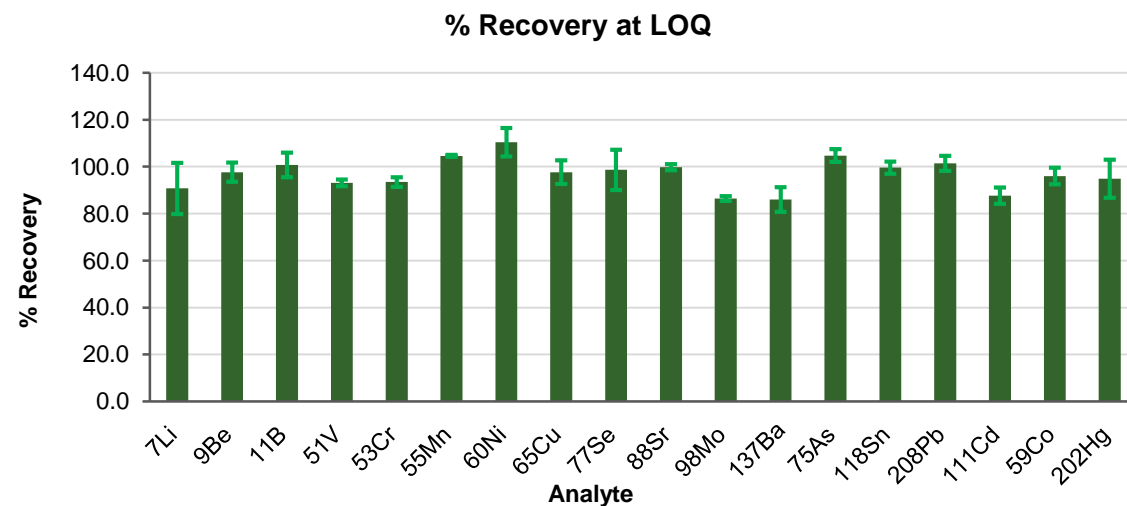


Element	R2	Intensity %RSD	IDL in ppb
Li	0.99985	0.3	0.06
Be	0.99999	4.1	0.02
B	0.99988	2.2	0.27
Al	0.99993	2.6	0.07
V	0.99998	4	0.01
Cr	1.00000	3.9	0.05
Mn	0.99996	1.8	0.003
Fe	0.99999	2	0.75
Co	0.99994	5.3	0.0006
Ni	0.99998	2.2	0.01
As	0.99998	3.2	0.004
Se	0.99995	5.4	0.17
Sr	0.99983	4	0.004
Mo	0.99528	1.4	0.001
Cd	1.00000	3	0.003
Sb	0.99999	4.1	0.002
Ba	0.99978	2	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

# Accuracy & Precision-Rice

Mass	Element	Level 1 (LOQ) mg·kg <sup>-1</sup>	% Rec.	% RSD	Level 2 mg·kg <sup>-1</sup>	% Rec.	% RSD	Level 3 mg·kg <sup>-1</sup>	% Rec.	% RSD
7	Li	0.4	91	10.9	0.8	98	6.2	2	101	2.4
9	Be	0.4	98	4.1	0.8	99	1.6	2	99	3.4
11	B	0.4	101	5.3	0.8	110	3.7	2	95	2.4
51	V	0.4	93	1.5	0.8	95	2.6	2	93	1.7
53	Cr	0.4	93	2	0.8	103	5.8	2	99	3.2
55	Mn	0.4	105	0.4	0.8	112	1.3	2	98	1.7
59	Co	0.01	96	3.5	0.02	96	1.8	0.05	105	1.1
60	Ni	0.4	110	6.1	0.8	98	2.2	2	94	1.6
65	Cu	0.4	98	5	0.8	113	1.8	2	90	1.2
75	As	0.1	105	2.7	0.2	103	1.7	2	104	1.6
77	Se	0.4	99	8.6	0.8	101	5.8	2	102	3.8
88	Sr	0.4	99	1.3	0.8	105	1.8	2	99	1.2
98	Mo	0.4	86	1.1	0.8	89	2.5	2	90	2.4
111	Cd	0.01	88	3.5	0.02	94	6.1	0.05	98	1.2
118	Sn	0.1	99	2.6	0.2	97	1.2	2	103	2.3
137	Ba	0.4	86	5.2	0.8	99	1.5	2	97	2
202	Hg	0.01	95	8.1	0.02	84	2.9	0.05	86	1.6
208	Pb	0.1	101	3.2	0.2	97	2.3	2	101	1.1

Accuracy @ LOQ is 86-110%  
Precision @ LOQ is <11%

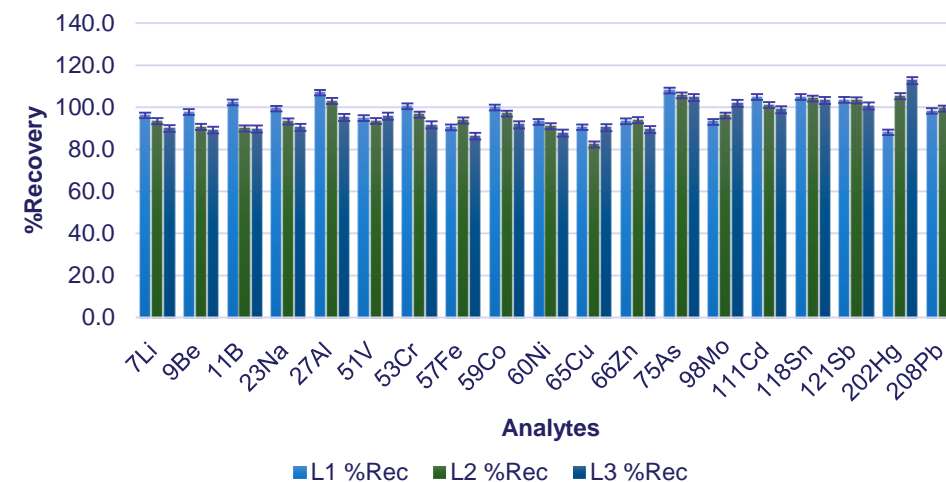


# Accuracy & Precision-Wheat

Element	L1 (LOQ) mg·kg <sup>-1</sup>	%Rec	%RSD	L2 mg·kg <sup>-1</sup>	%Rec	%RSD	L3 mg·kg <sup>-1</sup>	%Rec	%RSD
7Li	0.4	96.1	3.1	2	93.4	1.7	20	89.9	1.4
9Be	0.4	97.8	4.2	2	90.7	1.8	20	89.1	1.1
11B	0.4	102.3	3.5	2	89.9	2.5	20	89.7	1.7
23Na	10	99.3	0.7	50	93.3	1.6	200	90.5	0.7
27Al	10	106.9	0.7	50	103.0	2.3	200	95.2	1.0
51V	0.4	94.9	1.1	2	93.5	1.2	20	95.8	0.7
53Cr	0.4	100.5	1.0	2	96.4	1.6	20	91.6	0.7
57Fe	20	90.4	2.1	50	93.7	0.8	200	86.3	1.0
59Co	0.01	99.9	1.1	0.05	97.0	2.0	1	91.7	0.8
60Ni	0.4	93.0	1.4	2	90.9	0.9	20	87.7	0.9
65Cu	0.4	90.5	3.2	2	82.3	1.8	20	90.3	0.5
66Zn	10	93.4	0.4	50	93.8	1.0	200	89.4	0.9
75As	0.1	107.9	3.9	0.2	105.7	1.4	2	104.6	1.0
98Mo	0.4	93.1	0.6	2	96.1	1.1	20	101.9	0.7
111Cd	0.01	104.9	1.8	0.05	101.1	2.2	1	98.8	0.7
118Sn	0.1	104.9	0.9	0.2	104.1	1.3	2	103.2	1.0
121Sb	0.1	103.5	1.4	0.2	103.3	1.7	2	100.6	1.1
202Hg	0.01	88.1	1.6	0.05	105.2	2.3	1	112.7	0.3
208Pb	0.1	98.3	2.0	0.2	99.3	5.1	2	102.0	1.0

Accuracy @ LOQ is 88-108%  
Precision @ LOQ is <5%

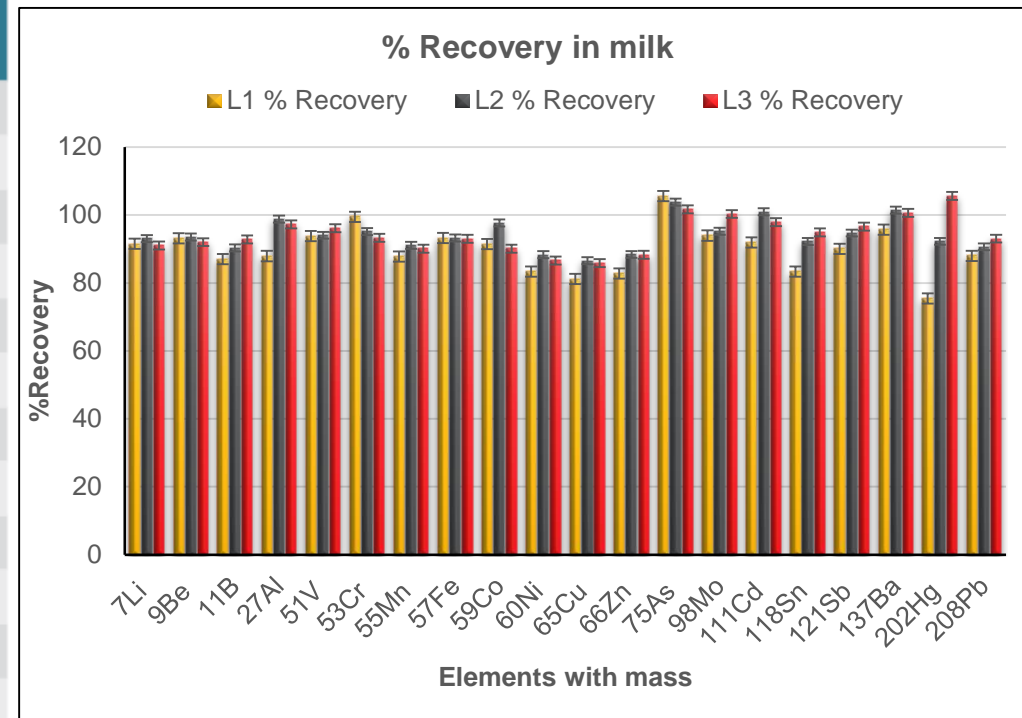
%Recovery at 3 different level spike in Wheat





# Accuracy & Precision-Milk and milk products

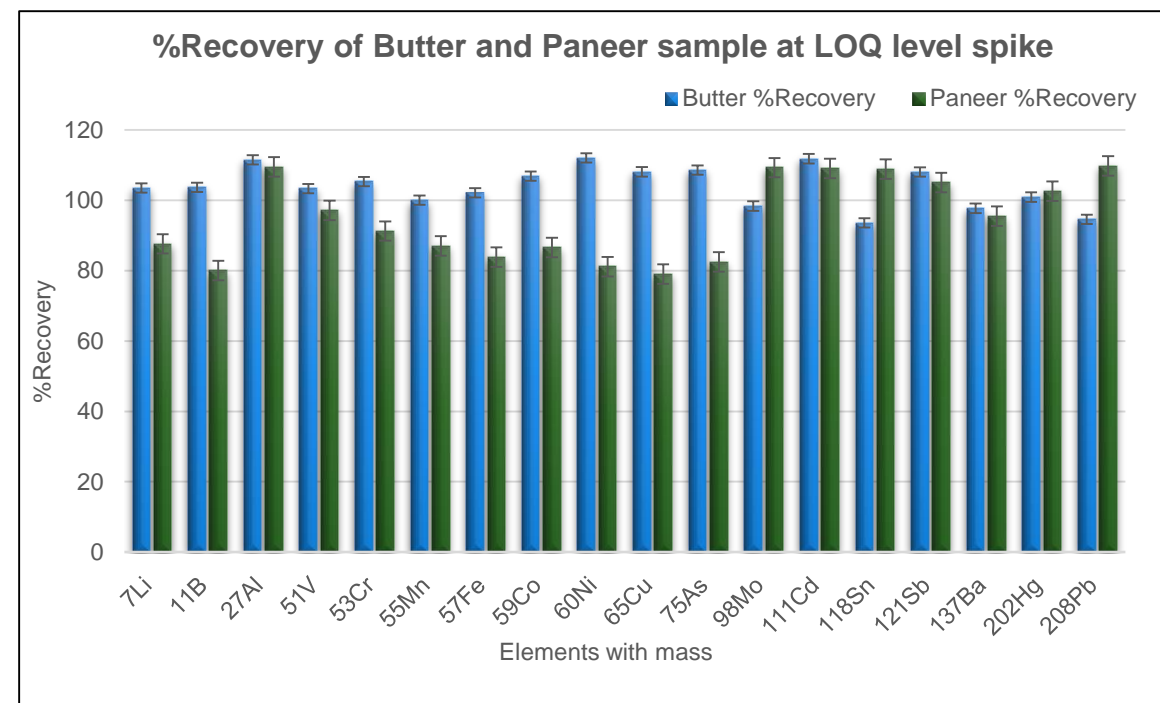
Isotope	L1 mg·kg <sup>-1</sup>	% Recovery	% RSD	L2 mg·kg <sup>-1</sup>	% Recovery	% RSD	L3 mg·kg <sup>-1</sup>	% Recovery	% RSD
<sup>7</sup> Li	0.10	92	2.3	0.50	93	2.4	5.00	91	1.5
<sup>9</sup> Be	0.10	93	4.7	0.50	94	1.7	5.00	92	1.9
<sup>11</sup> B	0.20	87	2.4	0.50	90	2.4	5.00	93	2.1
<sup>27</sup> Al	2.50	88	1.2	12.50	99	1.5	50.00	97	1.9
<sup>51</sup> V	0.10	94	2.0	0.50	94	1.7	5.00	96	1.2
<sup>53</sup> Cr	0.20	99	6.4	0.50	95	2.6	5.00	93	1.4
<sup>55</sup> Mn	0.10	88	1.8	0.50	91	2.3	5.00	90	1.7
<sup>57</sup> Fe	2.50	93	3.6	12.50	93	1.5	50.00	93	1.7
<sup>59</sup> Co	0.005	91	0.9	0.0125	98	2.6	0.50	90	1.4
<sup>60</sup> Ni	0.10	83	1.5	0.50	88	1.5	5.00	87	1.5
<sup>65</sup> Cu	0.10	81	1.5	0.50	87	1.7	5.00	86	1.8
<sup>66</sup> Zn	2.50	83	1.4	12.50	88	1.4	50.00	88	1.5
<sup>75</sup> As	0.005	106	2.3	0.02	104	1.8	0.50	102	2.3
<sup>98</sup> Mo	0.10	94	1.2	0.50	95	2.0	5.00	100	1.5
<sup>111</sup> Cd	0.003	92	5.1	0.0125	101	1.4	0.50	98	1.0
<sup>118</sup> Sn	0.005	83	2.7	0.02	92	1.6	0.50	95	1.9
<sup>121</sup> Sb	0.005	90	6.8	0.02	95	1.8	0.50	97	1.7
<sup>137</sup> Ba	0.10	96	1.0	0.50	101	1.9	5.00	101	1.7
<sup>202</sup> Hg	0.005	75	4.5	0.0125	92	2.0	0.50	106	1.1
<sup>208</sup> Pb	0.01	88	4.8	0.02	91	2.0	0.50	93	2.7



**Milk**  
**Accuracy @ LOQ is 81-108%**  
**Precision @ LOQ is <7%**

# Accuracy & Precision-Milk and Milk Products

Isotope	Measured spiked sample conc. in Qtegra ISDS Software (mg·kg <sup>-1</sup> )	Corrected conc. with dilution factor (mg·kg <sup>-1</sup> )	Butter (n=6)		Cheese (n=6)	
			% Recovery	% RSD	% Recovery	% RSD
<sup>7</sup> Li	0.0020	0.1000	103	0.8	88	4.6
<sup>11</sup> B	0.0020	0.1000	104	3.0	80	1.2
<sup>27</sup> Al	0.1000	5.0000	111	2.8	109	1.4
<sup>51</sup> V	0.0020	0.1000	103	1.1	97	1.3
<sup>53</sup> Cr	0.0040	0.2000	105	3.2	91	1.9
<sup>55</sup> Mn	0.0040	0.2000	100	2.5	87	1.0
<sup>57</sup> Fe	0.0500	2.5000	102	1.5	84	1.7
<sup>59</sup> Co	0.0001	0.0025	107	1.0	87	1.5
<sup>60</sup> Ni	0.0020	0.1000	112	1.1	81	1.9
<sup>65</sup> Cu	0.0040	0.2000	108	2.3	79	1.2
<sup>75</sup> As	0.0003	0.0150	109	2.0	82	3.4
<sup>98</sup> Mo	0.0020	0.1000	98	1.3	109	1.4
<sup>111</sup> Cd	0.0001	0.0050	112	2.6	109	2.3
<sup>118</sup> Sn	0.0001	0.0050	94	2.2	109	3.0
<sup>121</sup> Sb	0.0002	0.0100	108	1.2	105	1.4
<sup>137</sup> Ba	0.0020	0.1000	98	1.0	95	1.9
<sup>202</sup> Hg	0.0001	0.0050	101	2.4	103	1.3
<sup>208</sup> Pb	0.0001	0.0050	95	1.7	110	1.5



## Butter

Accuracy @ LOQ is 94-112%

Precision @ LOQ is <4%

## Paneer

Accuracy @ LOQ is 80-110%

Precision @ LOQ is <5%

# Spike concentrations in Meat products

Element	Spike concentration level in mg.kg <sup>-1</sup>		
	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



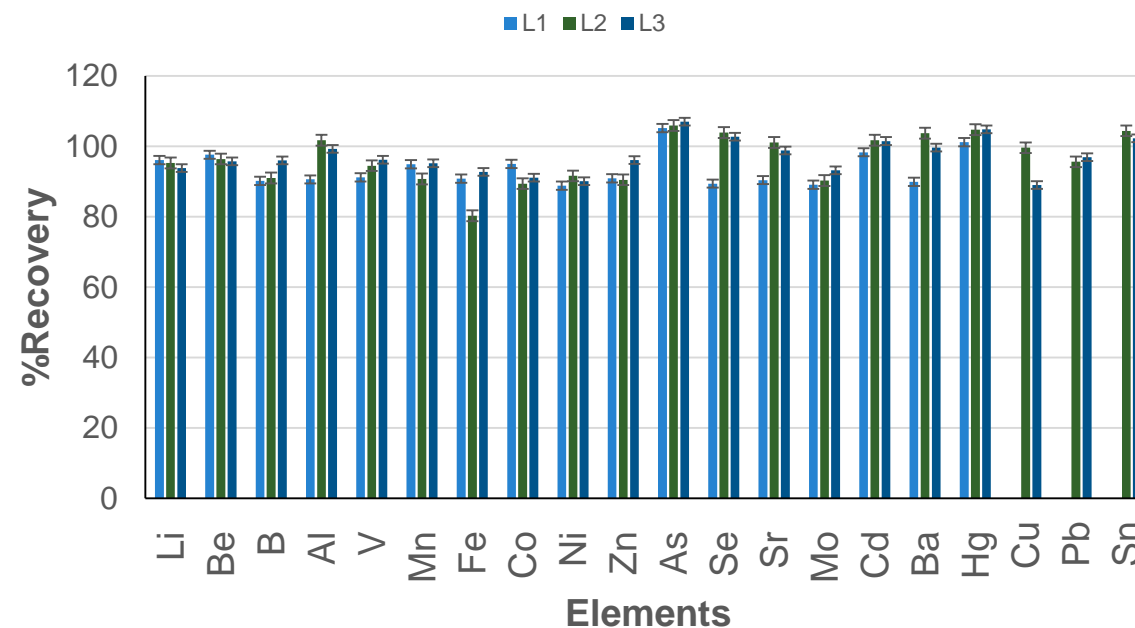
*\*Note: All the concentration values given in the table are calculated back to sample taken with a dilution factor of 100 fold.*

# Accuracy and precision in Meat products

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

## Chicken meat

Accuracy at 3 different level in red meat of chicken



Chicken meat  
 Accuracy @ LOQ is 89-107%  
 Precision @ LOQ is <10%

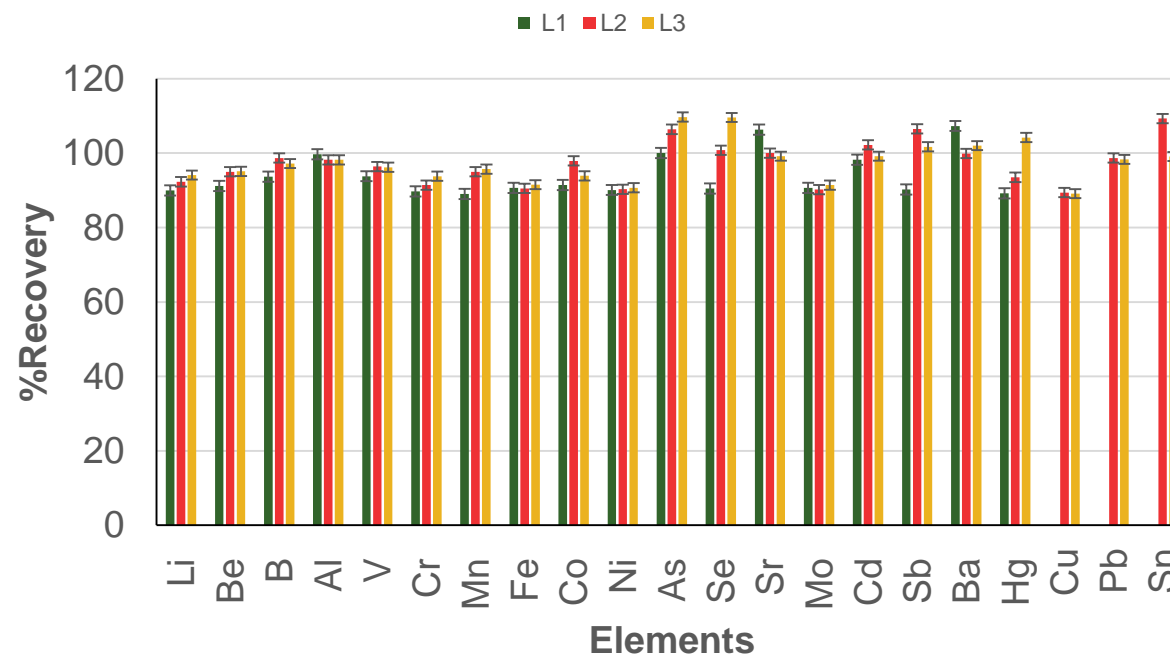
# Accuracy and Precision in Meat products

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

## Buffalo meat

Accuracy at 3 different level in red meat of buffalo



Buffalo meat

Accuracy @ LOQ is 89-110%

Precision @ LOQ is <10%

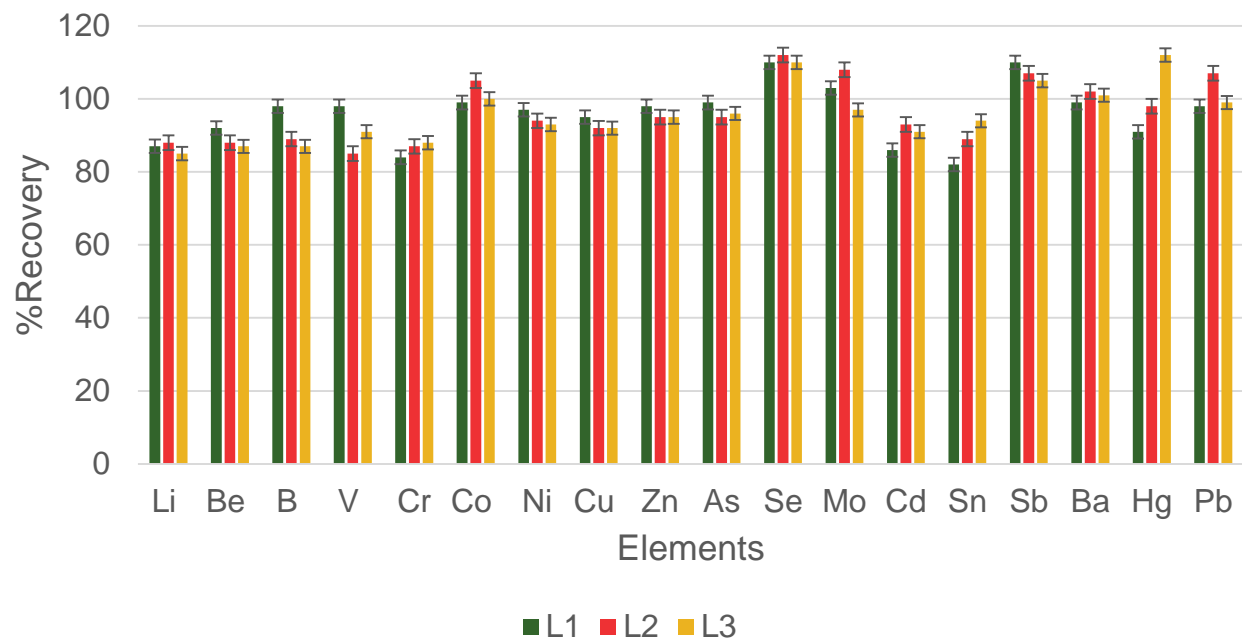
# Spike Concentrations in Spices

Elements	Spike concentrations in mgkg <sup>-1</sup>		
	L1 (LOQ)	L2	L3
Li, Be, B, V, Cr, Ni, Se, Mo	0.40	0.8	20.0
Co	0.125	0.25	1.0
Cu	1.5	3.75	30
Zn	10	20	200
As, Pb, Sb	0.02	0.04	2
Hg	0.02	0.05	1.0
Cd, Sn	0.01	0.02	1.0
Ba	0.8	2.0	20



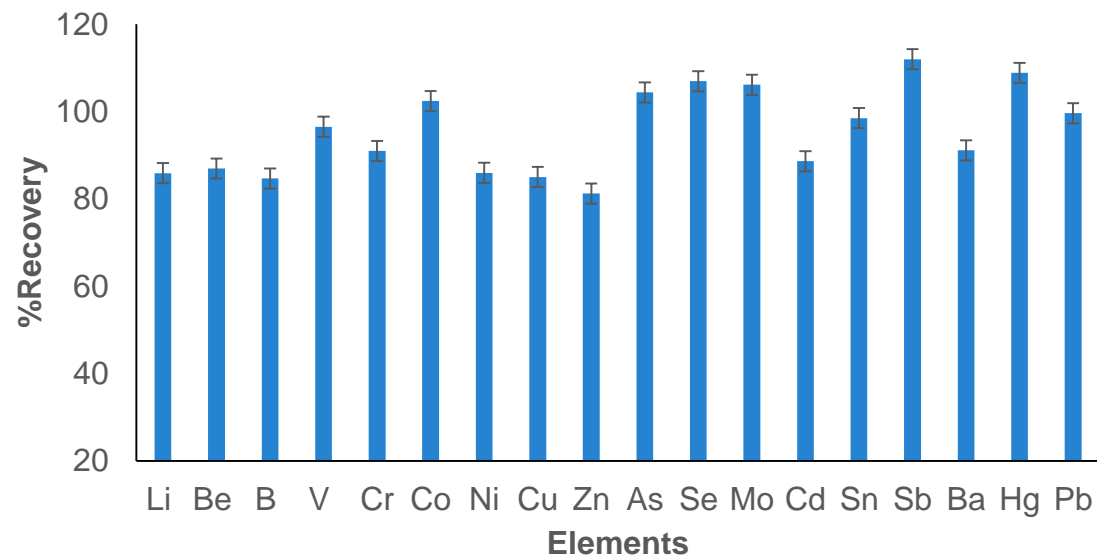
# Spike Concentrations in Spices

## Accuracy at three level spike in Turmeric powder

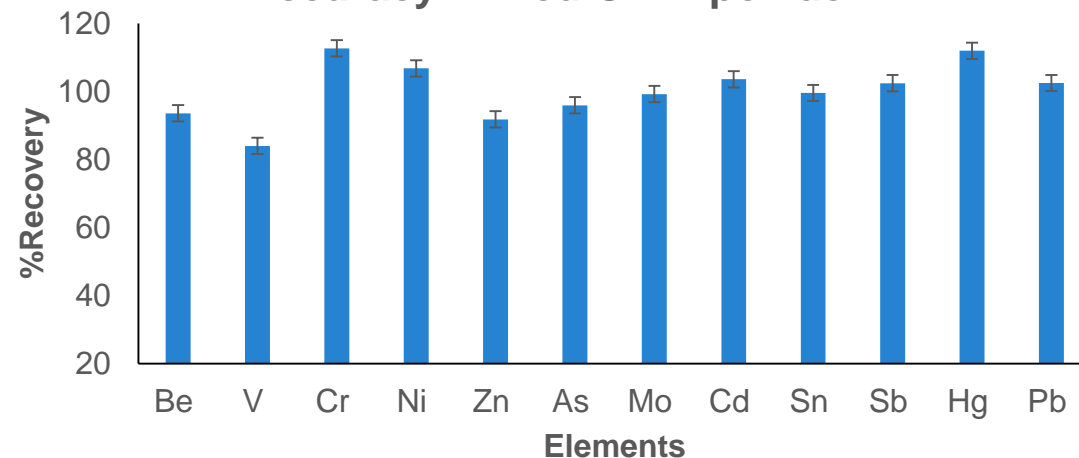


Turmeric powder, Black pepper powder, Red chilli powder  
 Accuracy @ LOQ is 80-112%  
 Precision @ LOQ is <10%

## Accuracy in Black pepper powder



## Accuracy in Red Chilli powder



# Spike Concentrations in Fruits and Vegetables

Element	Spike Conc (mg/kg)		
	L1	L2	L3
Li, Be, Ni, Mo, B, Ba	0.1	0.25	2
As	0.05	0.125	1
Al, Sn	3	7.5	60
V	0.25	0.5	2
Cr, Se	0.125	0.25	1
Fe, Cu, Zn	7.5	15	60
Co, Hg	0.002	0.005	0.04
Cd	0.005	0.0125	0.1
Sb, Pb	0.01	0.025	2

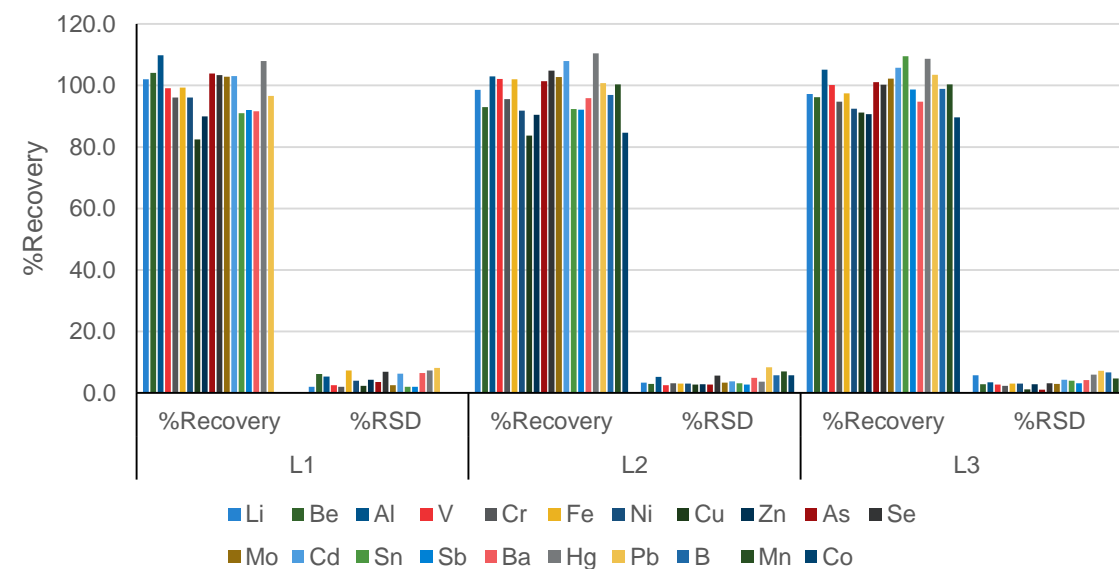




# Spike Concentrations in Fruits and Vegetables

Elements	L1		L2		L3	
	%Recovery	%RSD	%Recovery	%RSD	%Recovery	%RSD
Li	102.0	2.0	98.6	3.4	97.2	5.7
Be	104.0	6.2	93.0	2.9	96.2	2.8
Al	109.9	5.4	103.0	5.3	105.1	3.4
V	99.1	2.6	102.1	2.5	100.1	2.8
Cr	96.1	2.0	95.6	3.1	94.7	2.3
Fe	99.3	7.3	102.0	3.0	97.5	3.0
Ni	96.1	4.0	91.8	3.0	92.4	3.0
Cu	82.4	2.3	83.7	2.7	91.2	1.2
Zn	89.9	4.3	90.5	2.8	90.7	2.9
As	103.9	3.5	101.3	2.7	101.1	1.1
Se	103.4	6.8	104.8	5.6	100.3	3.1
Mo	102.8	2.5	102.8	3.3	102.2	2.9
Cd	103.1	6.2	107.9	3.8	105.8	4.3
Sn	91.0	2.0	92.3	3.2	109.5	4.0
Sb	92.0	2.0	92.1	2.7	98.7	3.2
Ba	91.6	6.5	95.9	4.9	94.7	4.2
Hg	108.0	7.3	110.5	3.6	108.7	5.9
Pb	96.6	8.1	100.7	8.3	103.5	7.3
B	NA	NA	96.9	5.7	98.9	6.7
Mn	NA	NA	100.4	7	100.3	4.7
Co	NA	NA	84.6	5.7	89.7	2.0

Accuracy and precision at three different level spikes in Tomato samples

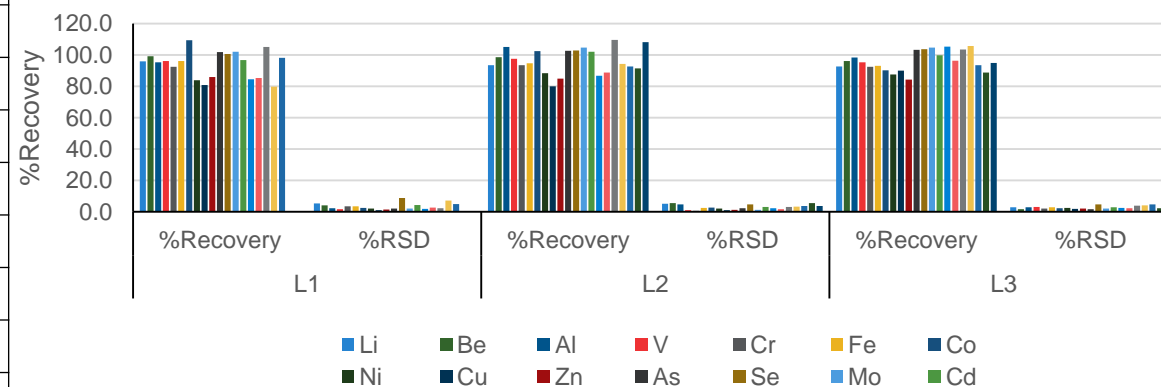


Accuracy @ LOQ is 80-111%  
Precision @ LOQ is <10%

# Spike Concentrations in Fruits and Vegetables

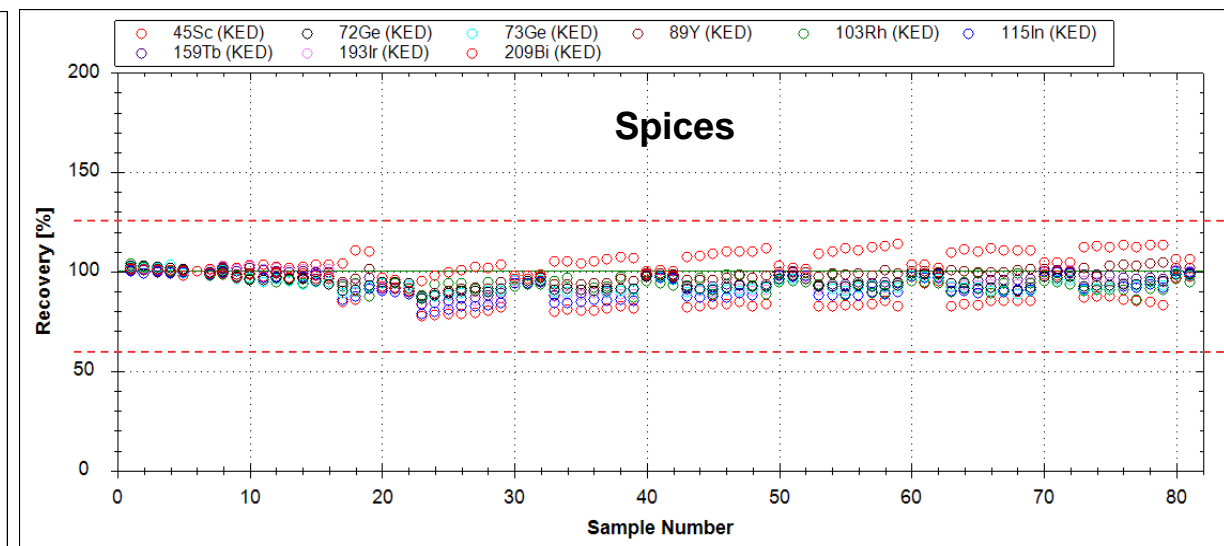
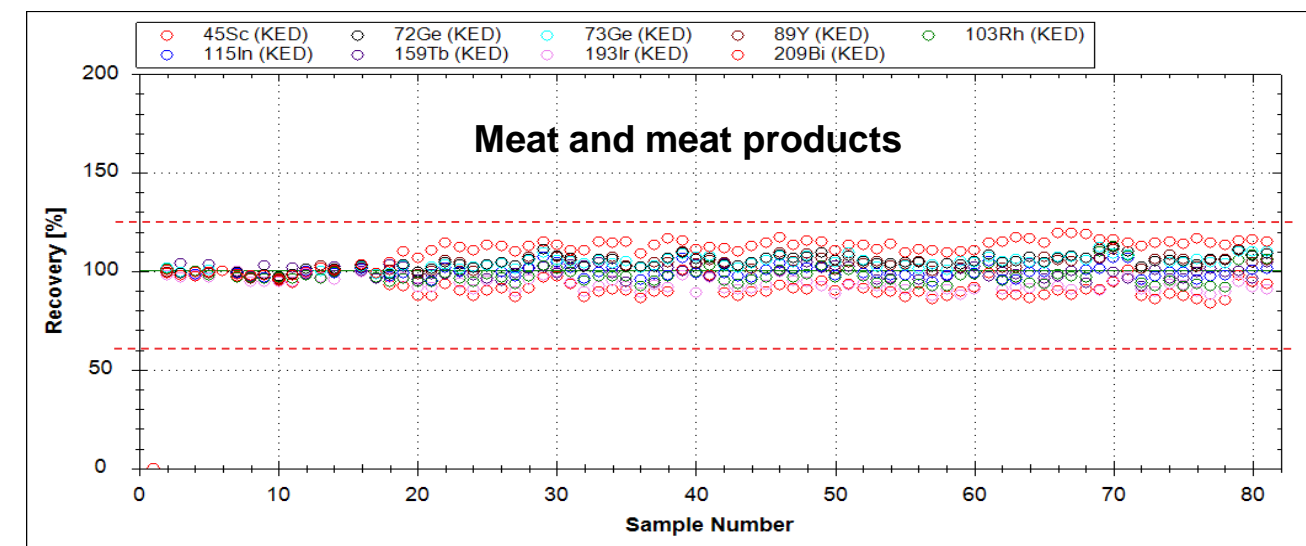
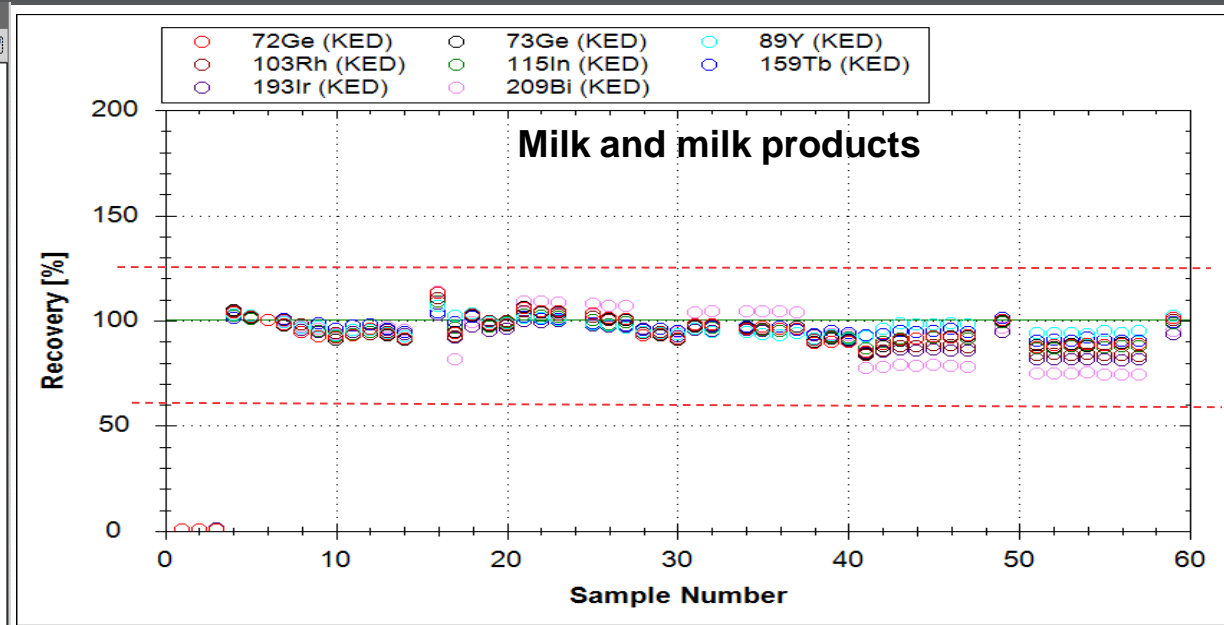
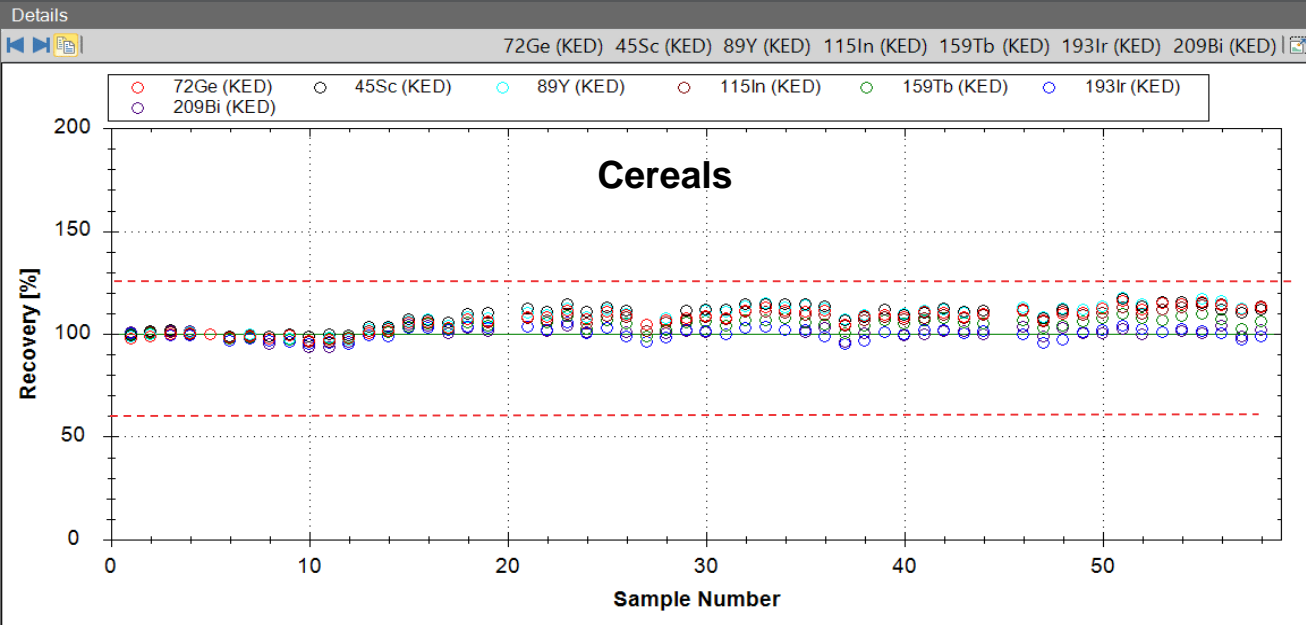
Ladies finger	L1		L2		L3	
Element	%Recovery	%RSD	%Recovery	%RSD	%Recovery	%RSD
Li	96.0	5.3	93.4	5.1	92.7	2.9
Be	99.3	4.1	98.6	5.5	96.3	1.7
Al	95.4	2.4	105.1	4.8	98.5	3.0
V	96.2	1.7	97.7	1.0	95.4	3.0
Cr	92.6	3.6	93.5	0.7	92.4	2.1
Fe	96.3	3.5	94.8	2.6	93.2	2.8
Co	109.4	2.4	102.5	2.8	90.3	2.3
Ni	84.0	2.1	88.5	2.0	87.7	2.5
Cu	81.0	1.2	80.0	1.1	90.1	1.9
Zn	86.0	1.5	84.9	1.2	84.3	2.0
As	101.9	2.2	102.7	2.2	103.4	1.8
Se	100.7	8.9	103.0	4.8	103.8	4.9
Mo	102.2	2.1	104.7	1.3	104.8	2.1
Cd	96.9	4.4	102.1	3.1	99.9	2.9
Sn	84.6	1.9	86.8	2.3	105.4	2.5
Sb	85.5	2.6	88.8	1.7	96.5	2.3
Ba	105.2	2.3	109.7	3.0	103.6	3.9
Hg	80	7.1	94.4	3.3	105.8	4.2
Pb	98.2	4.9	92.7	3.8	93.6	4.8
B	NA	NA	91.5	5.7	88.9	2.3
Mn	NA	NA	108.3	3.6	94.9	2.5

Accuracy and precision at three different level spikes in Ladies finger

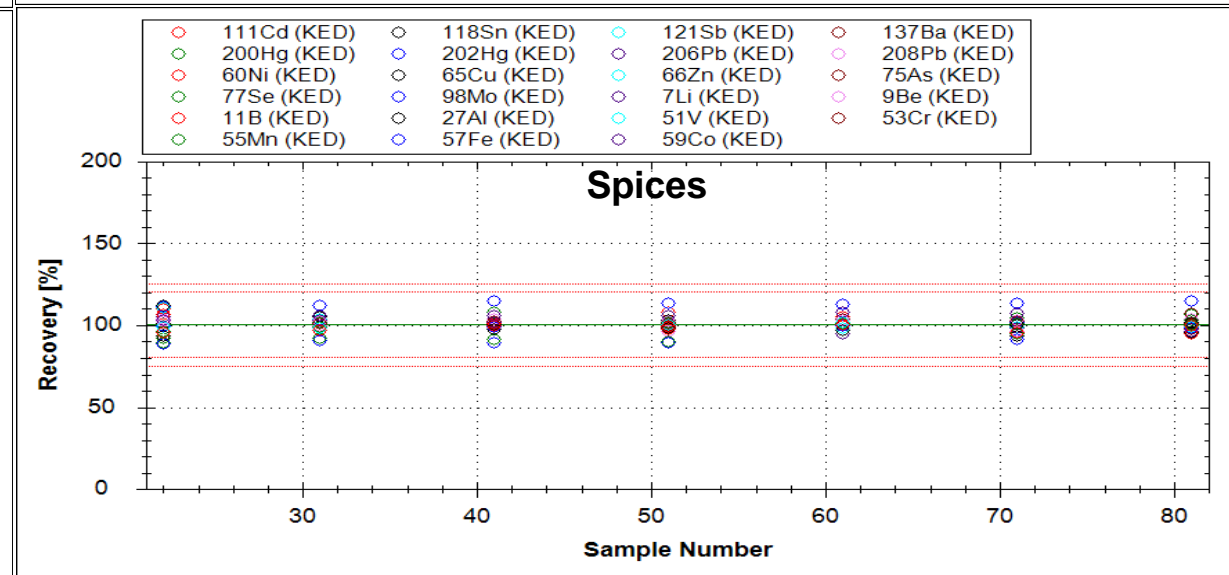
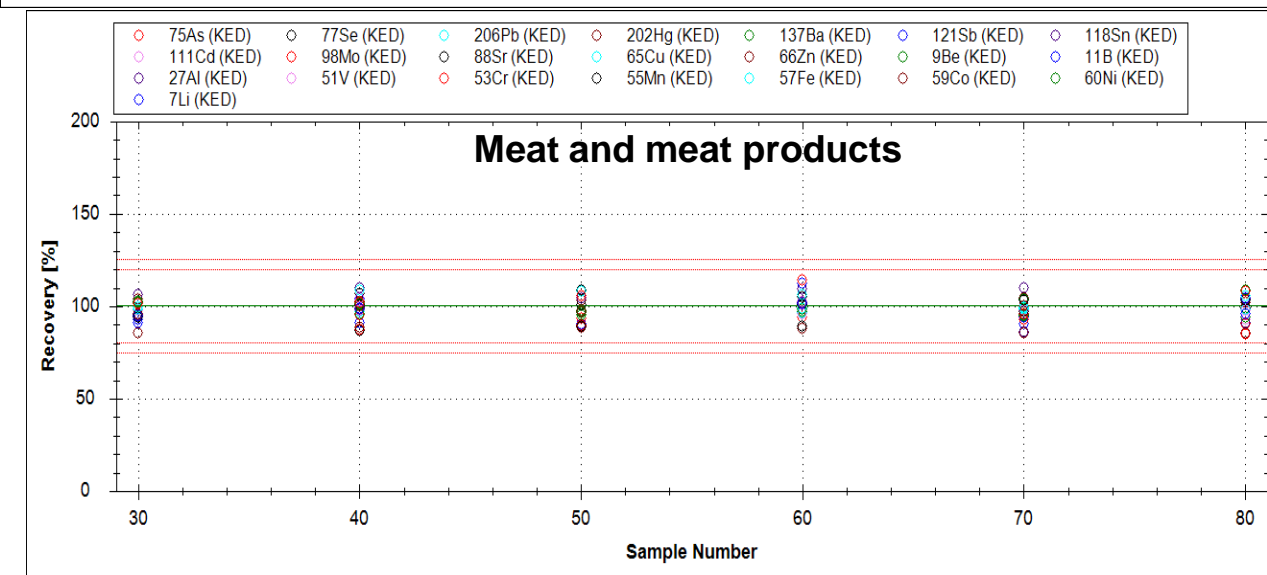
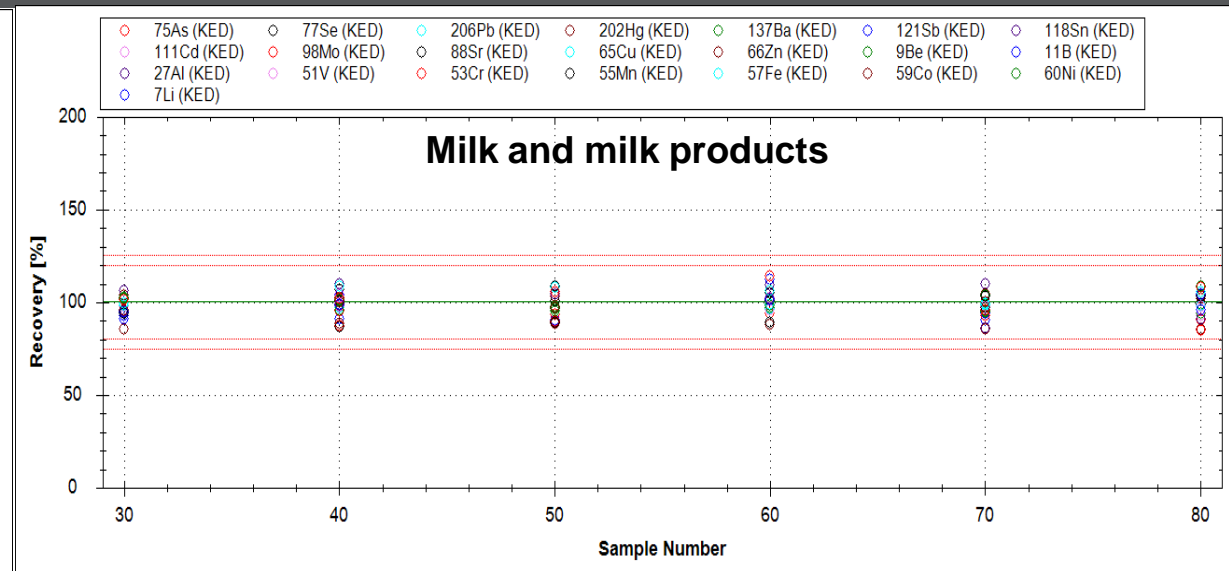
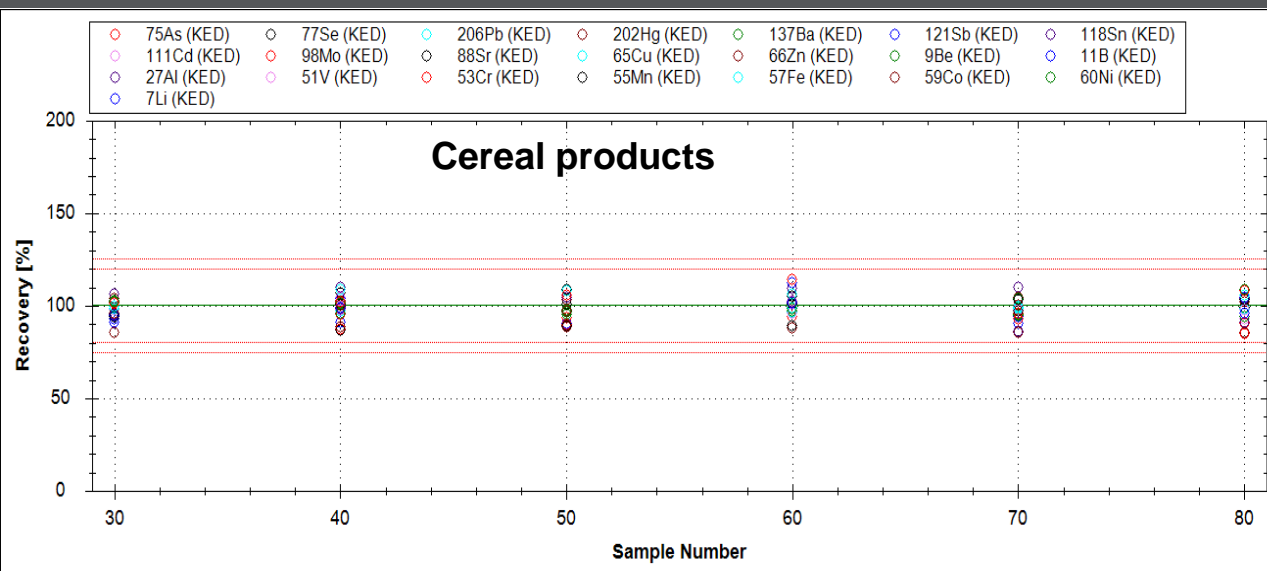


Accuracy @ LOQ is 80-110%  
Precision @ LOQ is <10%

# Internal Standard Response



# Sequence Accuracy with CCV Standard





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APPLICATION NOTE 73020

## Low level quantification of trace metals in rice using ICP-MS

**Authors**  
Suresh Murugesan and Dashaarath Oulker  
Customer Solution Center,  
Ghaziabad,  
Thermo Fisher Scientific, India

**Goal**  
To demonstrate the capability of an ICP-MS for trace elemental analysis in rice in compliance with the AOAC Official Method 2015.01 validation guidelines and the Food Safety and Standards Authority of India (FSSAI), China Food and Drug Administration (CFDA), and European Union (EU) MRL requirements.


**Keywords**  
Trace metals, rice, ICP-MS, KED, iCAP RQ, Qtegra

**Introduction**  
Rice is an important staple food with global consumption of 518 million tons in 2016.<sup>1</sup> The main countries in which rice is a major part of the human diet are India and China. Consequently, the FSSAI, CFDA, and the European Commission (EC), have set maximum residue limits (MRLs) for heavy metals in rice as listed in Table 1.<sup>2-4</sup> Heavy metals can occur in rice as a consequence of uptake from agricultural soils contaminated by mining activities and sludge disposal. Processes such as milling and polishing, packaging, and transportation may also be a source of contamination. Contamination with toxic heavy metals in rice is a concern because of the possible adverse effects on human health. Scientists have reported health issues including coughing, anemia, and kidney failure, ultimately leading to death due to heavy metal contamination.<sup>4,6</sup> To verify and quantify the amount of toxic elements in rice, commercial food testing laboratories widely use a procedure based on

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<https://www.revbase.com/tt/sl.ashx?z=73090c66&DataID=370711&ft=1>



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APPLICATION NOTE 73165

## Trace elemental quantification, including heavy metals in wheat, using ICP-MS

**Authors**  
Suresh Murugesan and Dashaarath Oulker  
Customer Solution Center,  
Ghaziabad,  
Thermo Fisher Scientific, India

**Goal**

- To develop and validate an analytical solution for routine multielemental analysis in wheat using inductively coupled plasma mass spectrometry (ICP-MS) in accordance with AOAC 2015.01 guidelines
- To assess method performance for compliance with the Food Safety Standards Authority of India (FSSAI), China Food and Drug Administration (CFDA), and European Commission (EC) MRL requirements in wheat

**Keywords**  
Trace metals, wheat, ICP-MS, iCAP RQ, Qtegra ISDS Software, KED

**Introduction**  
Wheat is an important cereal that is rich in carbohydrates and protein and is consumed worldwide as a staple food, with global wheat production for 2018-2019 at 732 million tons.<sup>1</sup> Wheat flour is processed from wheat grain and is the primary raw material for many bakery products like biscuits and bread. The introduction of toxic elements in wheat may occur through heavy metal deposition in soil and water by industrial and mining processes or agricultural and food processing practices. FSSAI, CFDA, and EC have established maximum residual limits (MRLs) in cereal grains, as listed in Table 1.<sup>2-4</sup> Some trace elements such as zinc or iron are important nutrients and are essential to a healthy and balanced diet; however, other elements like lead, cadmium, mercury, and arsenic pose a serious risk when consumed

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<https://www.revbase.com/tt/sl.ashx?z=73090c66&DataID=3971293&ft=1>

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APPLICATION NOTE 73117



Multielemental quantification of trace metals in milk and milk products using ICP-MS

**Authors**  
Suresh Murugesan and  
Dasharath Oulkar  
Customer Solution Center,  
Ghaziabad,  
Thermo Fisher Scientific, India

**Keywords**  
Trace metals, milk, milk products,  
ICP-MS, iCAP IQ, Qtegra ISDS  
Software, KED

**Goal**

- To demonstrate the efficiency of Thermo Scientific™ iCAP™ RQ ICP-MS to provide a single method for trace elemental analysis in milk and milk products, such as butter and cheese
- To assess method performance for compliance with AOAC 2015.01 guidelines, the Food Safety and Standards Authority of India (FSSAI), China Food and Drug Administration (CFDA), and European Commission (EC) MRLs

**Introduction**  
Milk is considered an essential and almost complete dietary food for children and adults. It provides necessary macronutrients such as carbohydrates, proteins, and fats as well as micronutrients such as vitamins (e.g., riboflavin, B5, B12) and minerals. India is the largest milk producer in the world, with a 20% contribution to total world milk production.<sup>1</sup> Secondary milk products like cheese and butter are used in a variety of processed foods.

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APPLICATION NOTE 44459

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

Authors: Suresh Murugesan,  
Piyush Deokar, and Dasharath Oulkar  
Customer Solution Center,  
Thermo Fisher Scientific, Ghaziabad, India



Keywords: Trace metals, meat, red meat, beef, chicken, goat meat, ICP-MS, Qtegra ISDS Software

**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),<sup>1</sup> China Food and Drug Administration (CFDA),<sup>2</sup> and European Commission (EC)<sup>3</sup> 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.<sup>4</sup>

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.

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**Raise** the Bar

# Thank you

[piyush.deokar@thermofisher.com](mailto:piyush.deokar@thermofisher.com)

[suresh.murugesh@thermofisher.com](mailto:suresh.murugesh@thermofisher.com)

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