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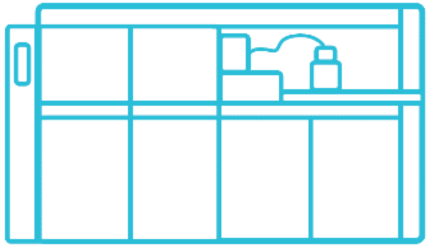
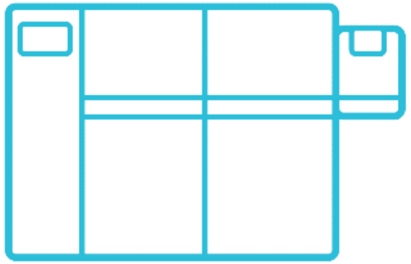
Outsmart Interferences with Triple Quadrupole ICP-MS

ICP-MS Performance for All Applications

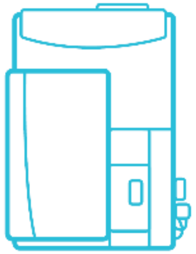
Technology for All Challenges



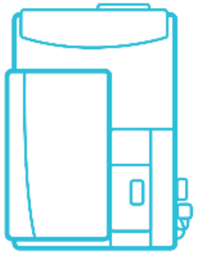
Thermo Scientific™ NEPTUNE™ Plus MC-ICP-MS
Multicollector ICP-MS



Thermo Scientific™ ELEMENT 2/XR™ HR-ICP-MS
High Resolution ICP-MS



Thermo Scientific™ iCAP™ TQ ICP-MS
Triple quadrupole ICP-MS



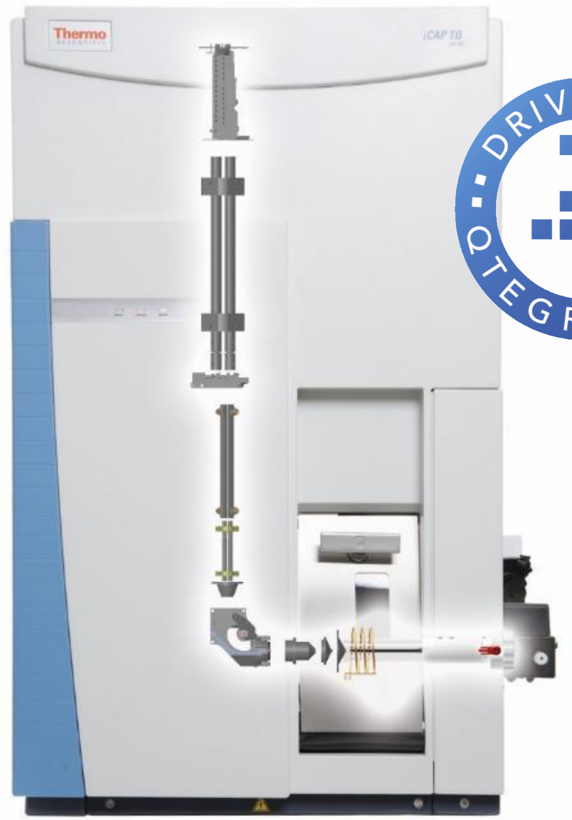
Thermo Scientific™ iCAP™ RQ ICP-MS
Single quadrupole ICP-MS

ThermoFisher SCIENTIFIC

All the Power,
None of the Complexity

- ✓ Robust design for routine analysis
- ✓ Integrated automation options
- ✓ Flexible for advanced applications
- ✓ Advanced interference removal
- ✓ Unique ease of use

Triple quadrupole accuracy with single quadrupole ease of use



DRIVEN BY QTEGRA ISDS

What’s the difference between a SQ and TQ-ICP-MS?

iCAP RQ ICP-MS

iCAP TQ ICP-MS

Additional Q1 mass filter quadrupole

Additional electronics

Additional gases

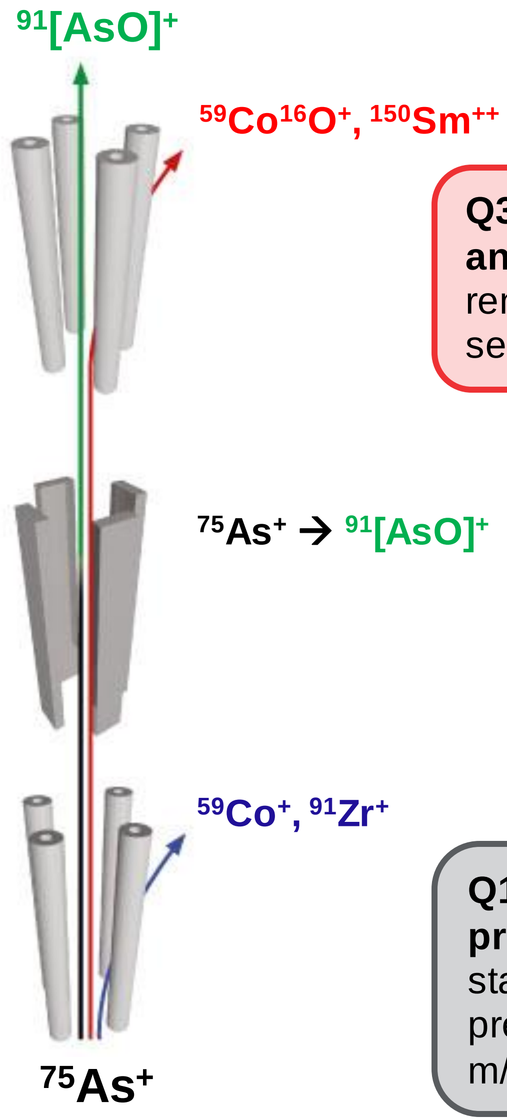
Enhanced software

Thermo Scientific iCAP TQ ICP-MS – How it Works

Q3 set to production mass (*m/z* 91)

Q2 filled with reactive gas (O_2)

Q1 set to analyte mass (*m/z* 75)



Q3 isolates the production of the analyte and removes any remaining interferences through a second stage of mass filtration

Optimal reaction conditions in Q2 are achieved through the selection of the appropriate measurement mode in Reaction Finder

Q1 rejects unwanted ions and preselects the analyte. This first stage of mass filtration rejects precursors and ions with the same m/z ratio as the production.

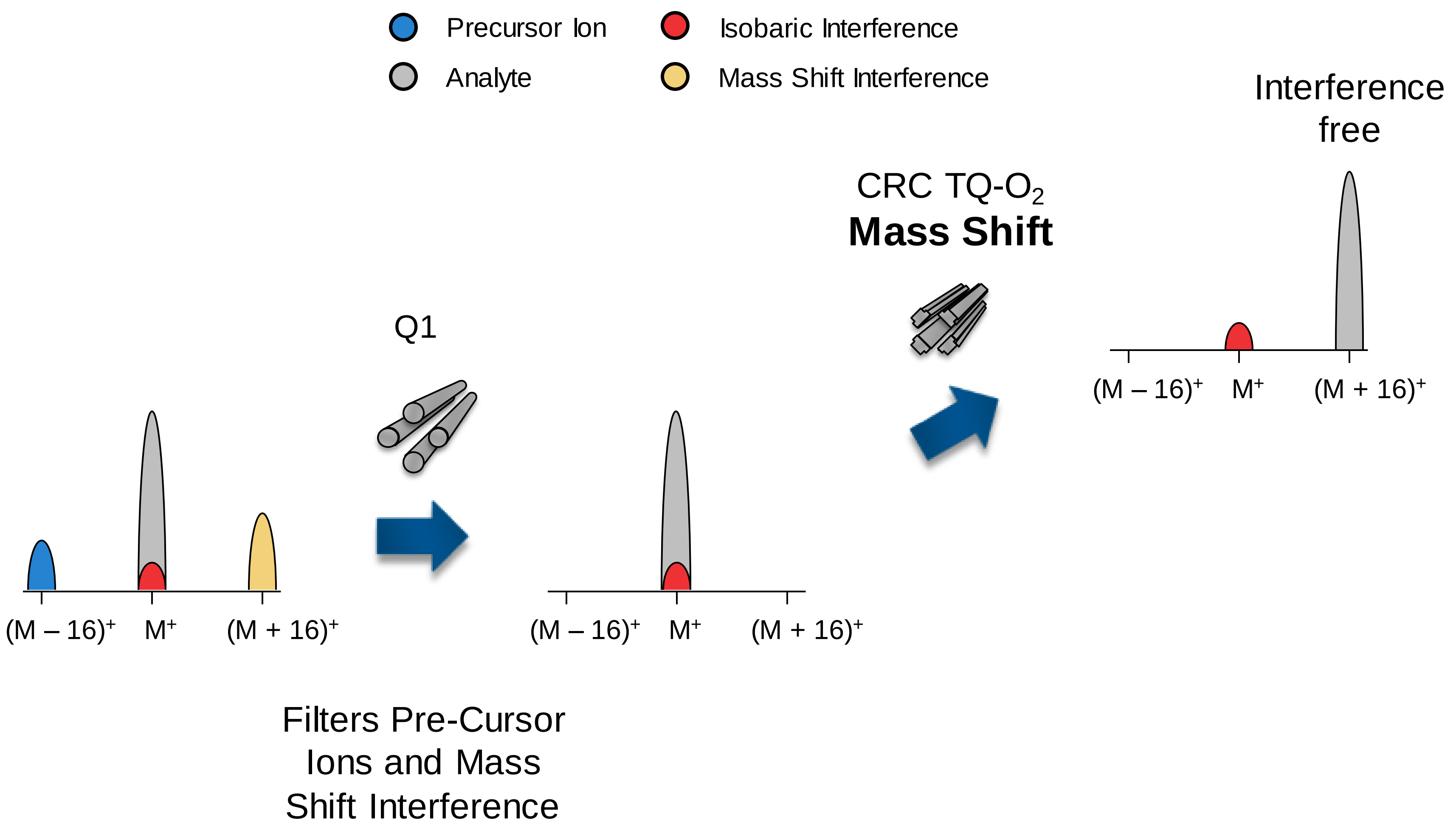
- More specific interference removal through reactive chemistry inside the CRC
- Removal of unwanted ions in Q1 eliminates interferences on product ion mass and unwanted side reactions

Result:

- Better detection limits, even in challenging sample matrices

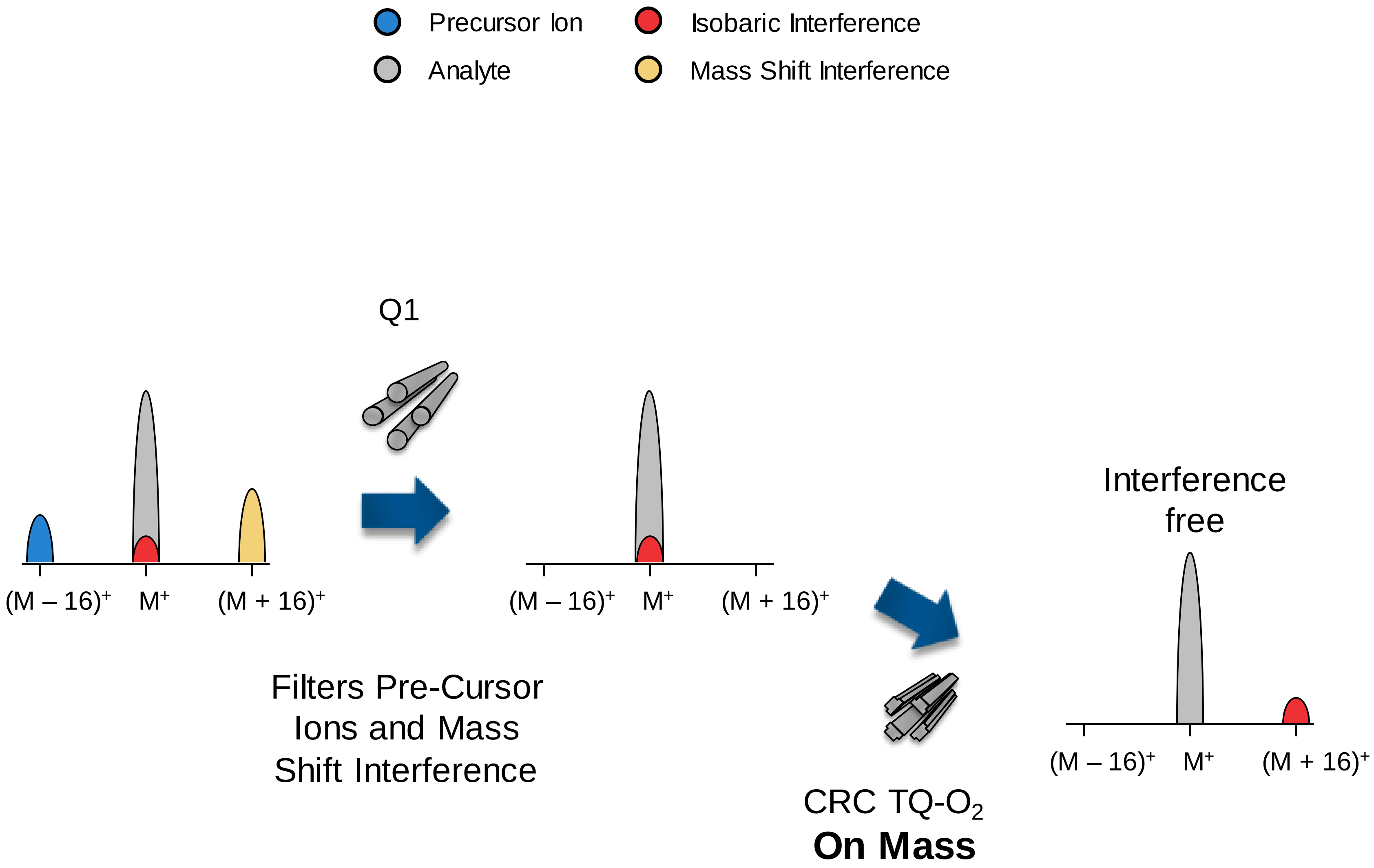
- Get more accuracy in unknown or varying sample matrices

Triple Quadrupole ICP-MS – Two ‘Styles’ of Operation



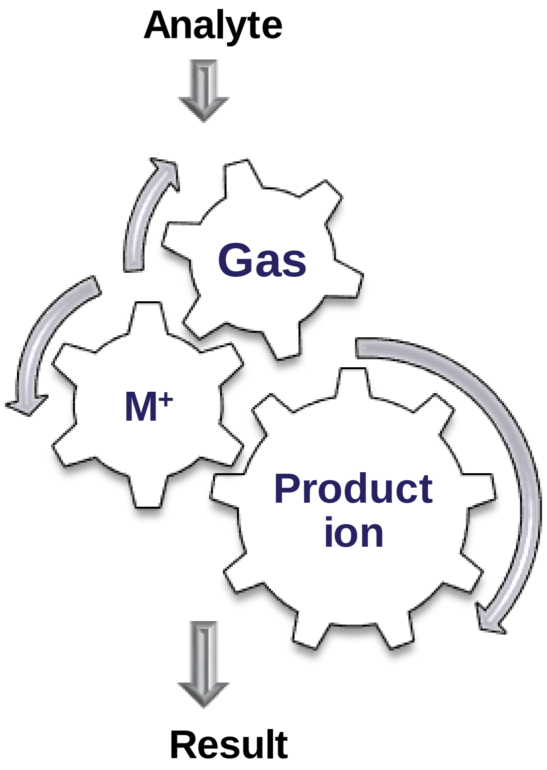
This mode of operation is ‘Mass Shift’

Triple Quadrupole ICP-MS – Two ‘Styles’ of Operation

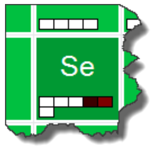


This mode of operation is ‘On Mass’

Reaction Finder Method Development Assistant



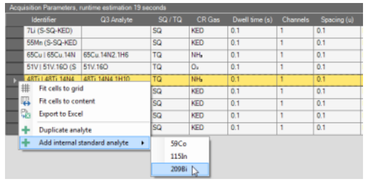
1. Select Element/Isotope of interest



2. Reaction Finder proposes most appropriate gas/scan setting combination

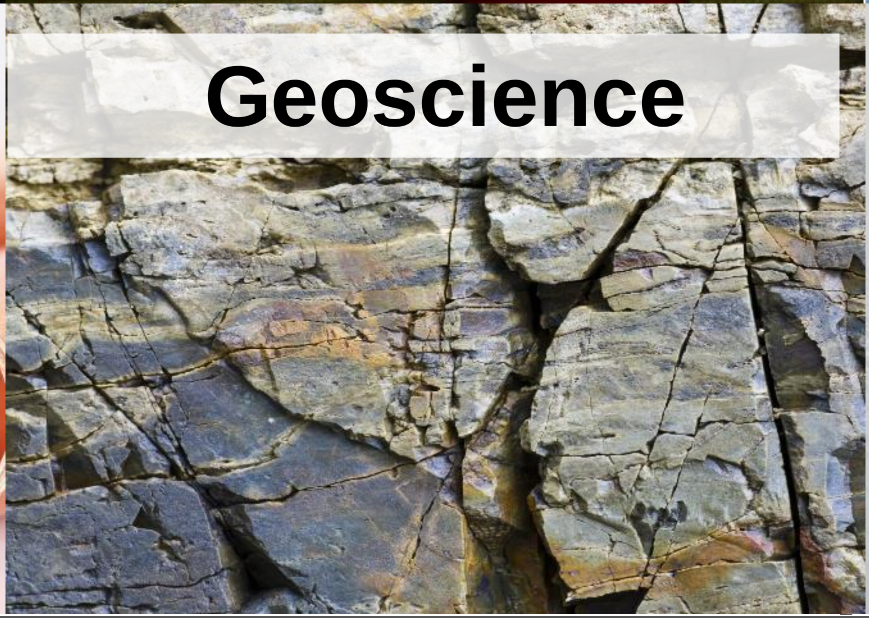
Identifier	Q3 Analyte	SQ / TQ	CR Gas	Dwell time (s)	Channels	Spacing (u)	
78Se | 78Se.16O	78Se.16O (93.912	TQ	O_2	0.1	1	0.1	Normal
80Se | 80Se.16O	80Se.16O	TQ	O_2	0.1	1	0.1	Normal

3. Choose from list of Internal Standards



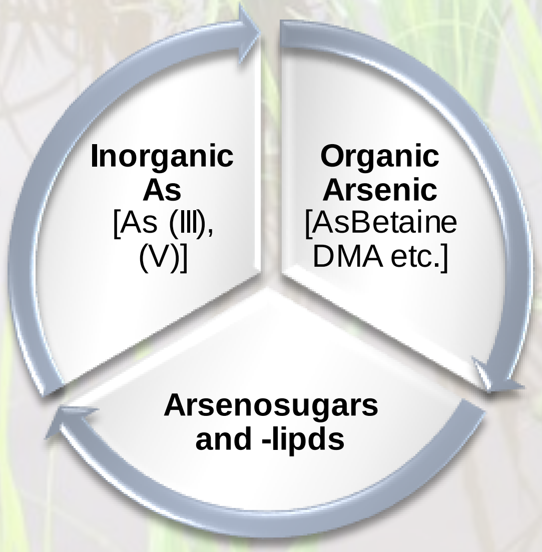
Redefining triple quadrupole ICP-MS with unique ease of use

The Power of Triple Quadrupole Technology

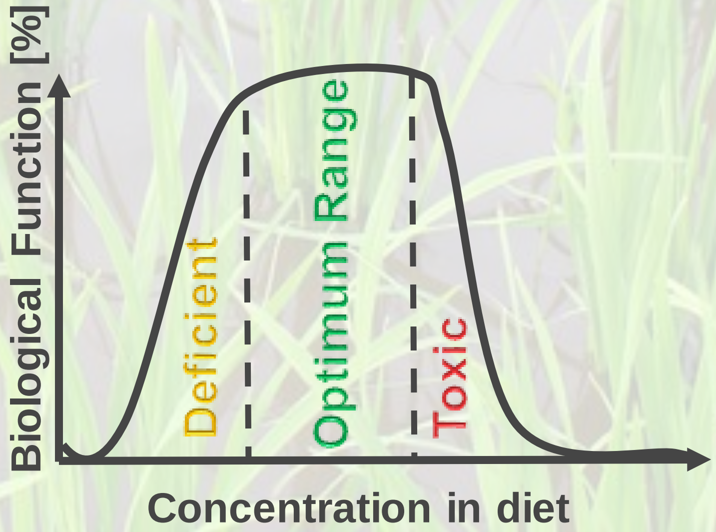


Accurate Arsenic and Selenium Quantification in Environmental and Food Samples

- Arsenic: A potential hazard in the food chain
- Many different chemical forms (species)
- Plants such as rice are well known for high accumulation of As from soils

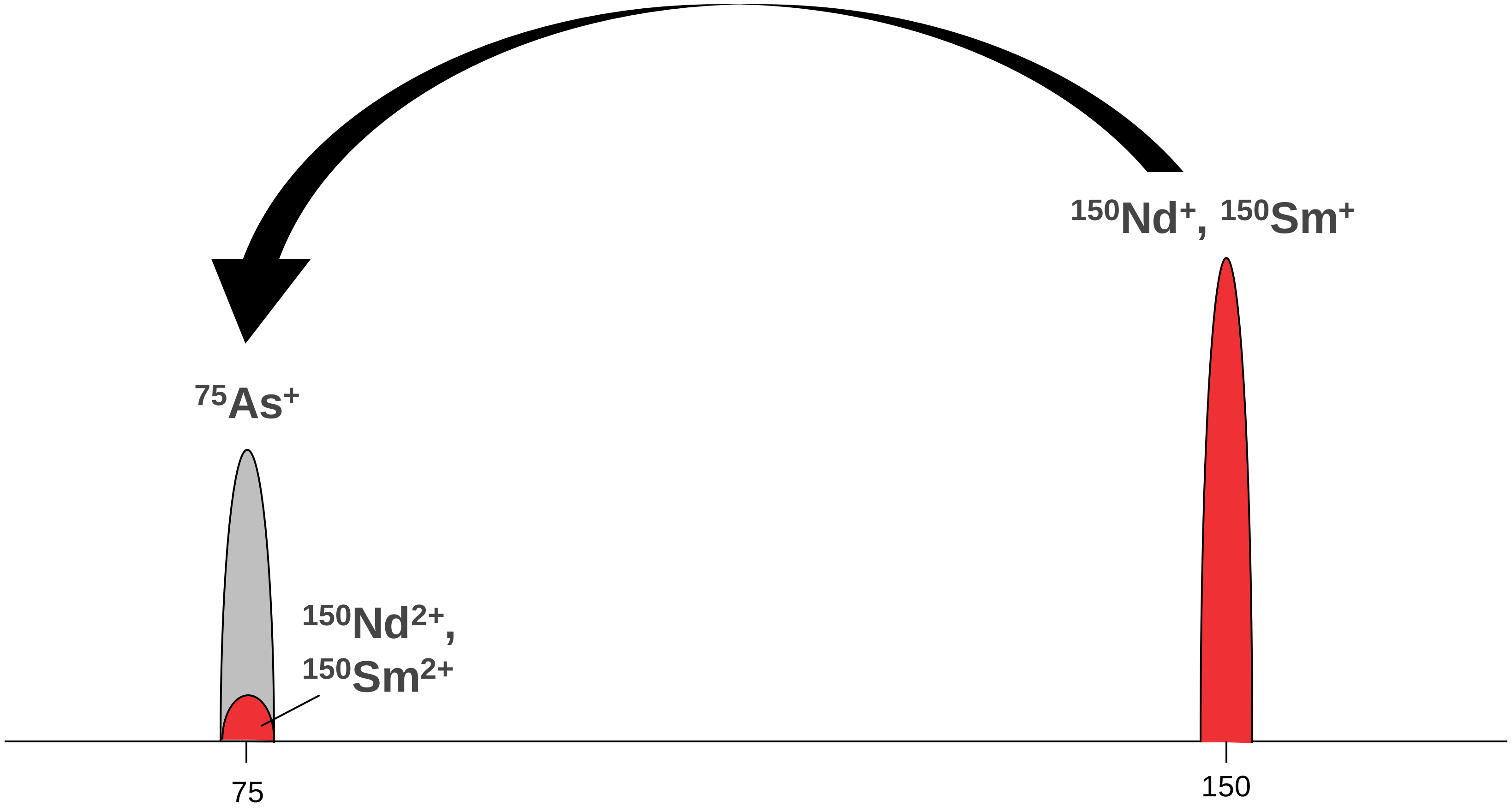


- Selenium: An essential nutrient
- Knowledge of Se content in soil may prevent Se deficiency in both human and animal populations



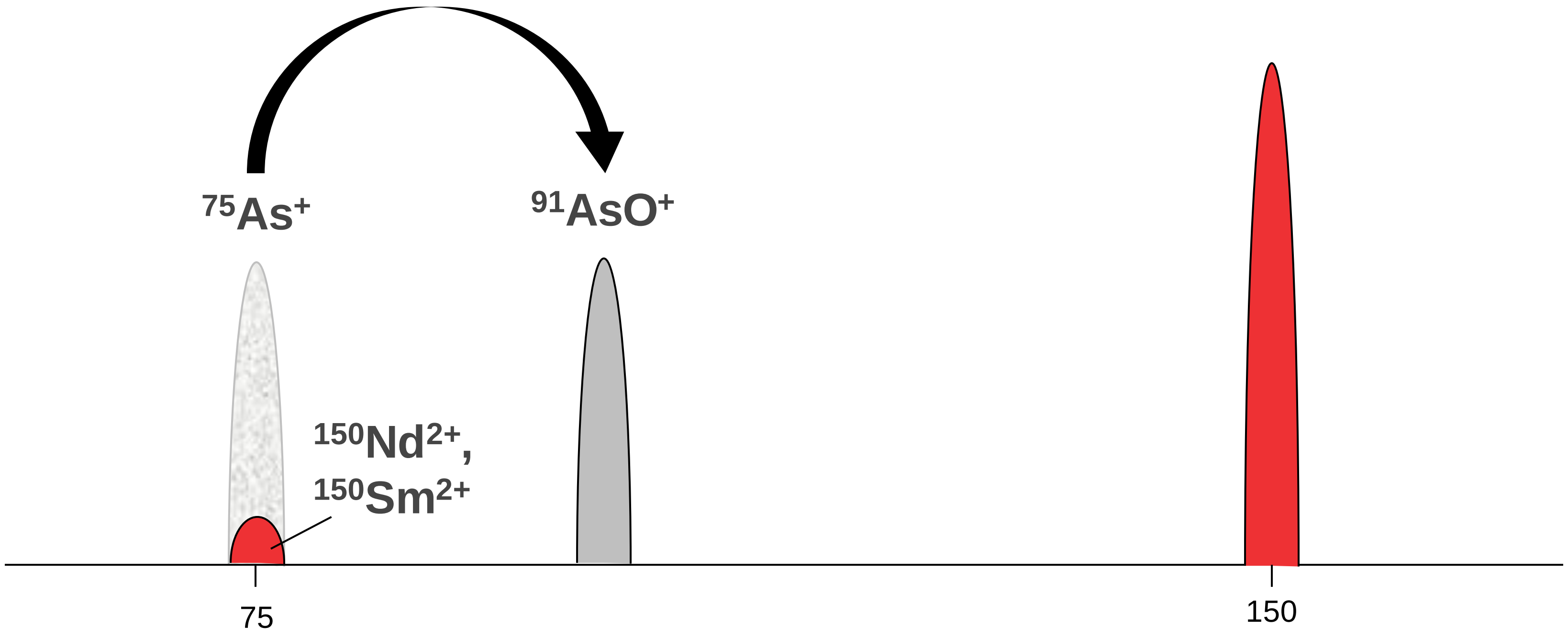
Accurate Analysis of Arsenic and Selenium in REE Matrix

Single Quad ICP-MS: KED
Typically enhances M^{2+} Interferences



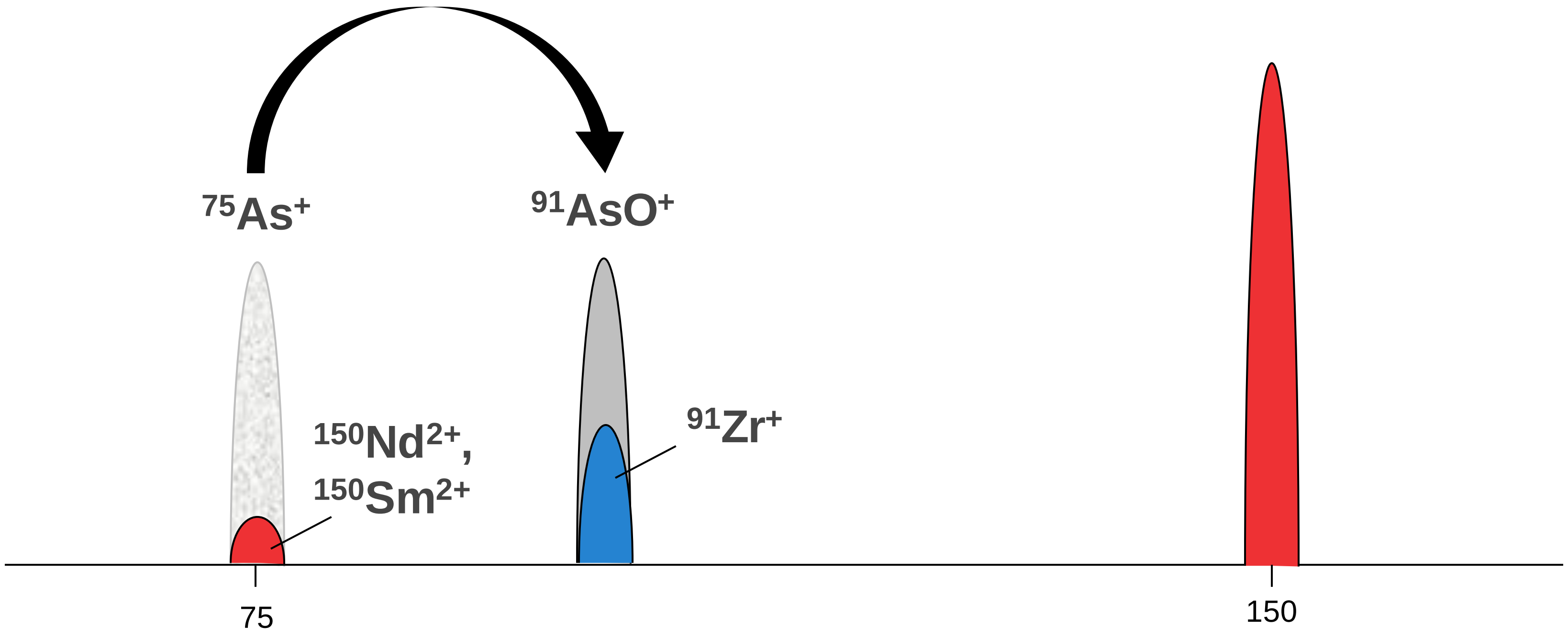
Accurate Analysis of Arsenic and Selenium in REE Matrix

Single Quad ICP-MS: KED
Typically enhances M^{2+} Interferences
Solution: Mass shift As and Se using O_2



Accurate Analysis of Arsenic and Selenium in REE Matrix

Single Quad ICP-MS: KED
Typically enhances M^{2+} Interferences
Solution: Mass shift As and Se using O_2
Other interferences: $^{91}Zr^{+}$, $^{94,96}Mo^{+}$, if present in the sample

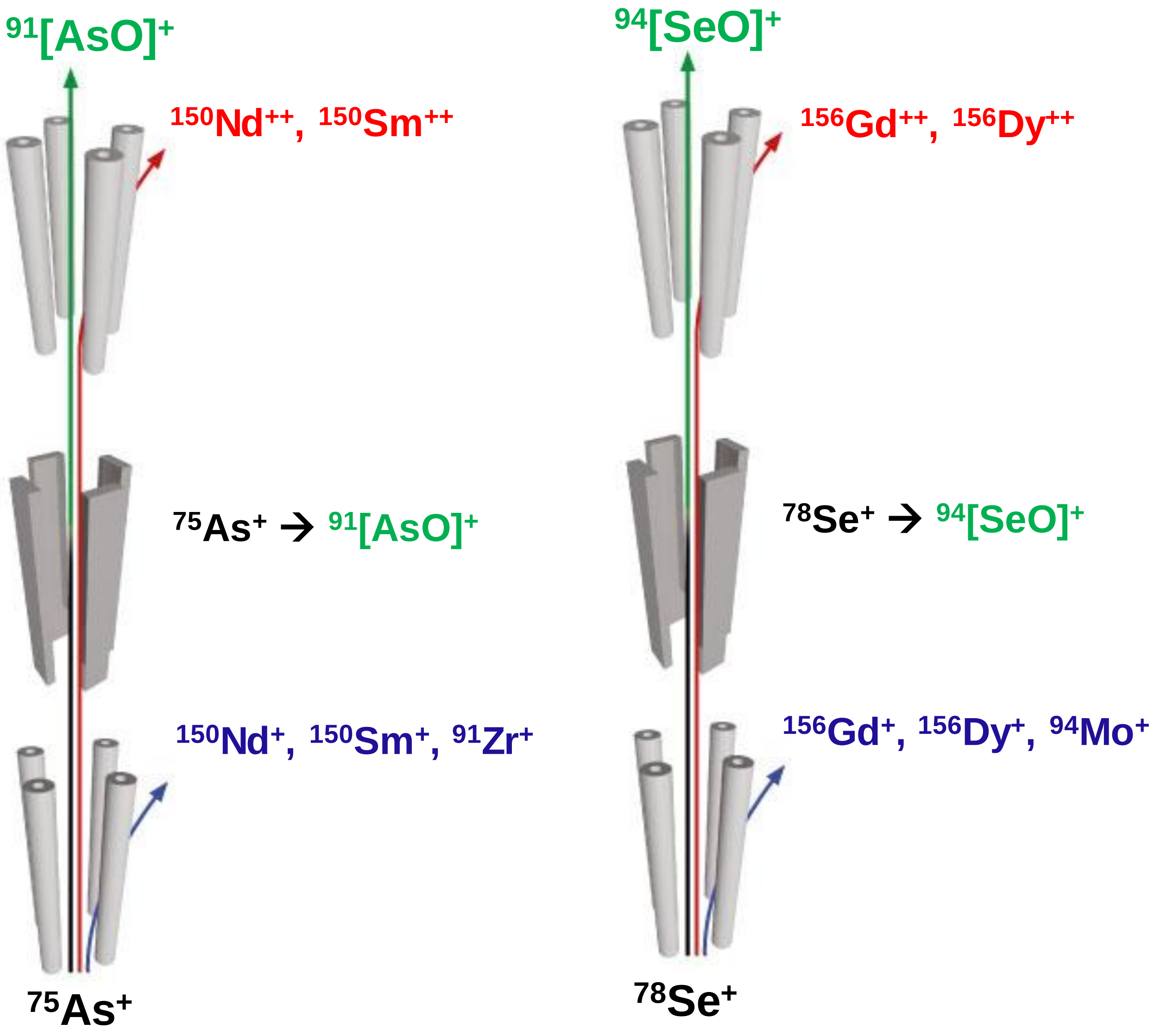


Accurate Analysis of Arsenic and Selenium in REE Matrix

Q3 set to product ion mass

Q2 filled with reactive gas (O_2)

Q1 set to analyte mass

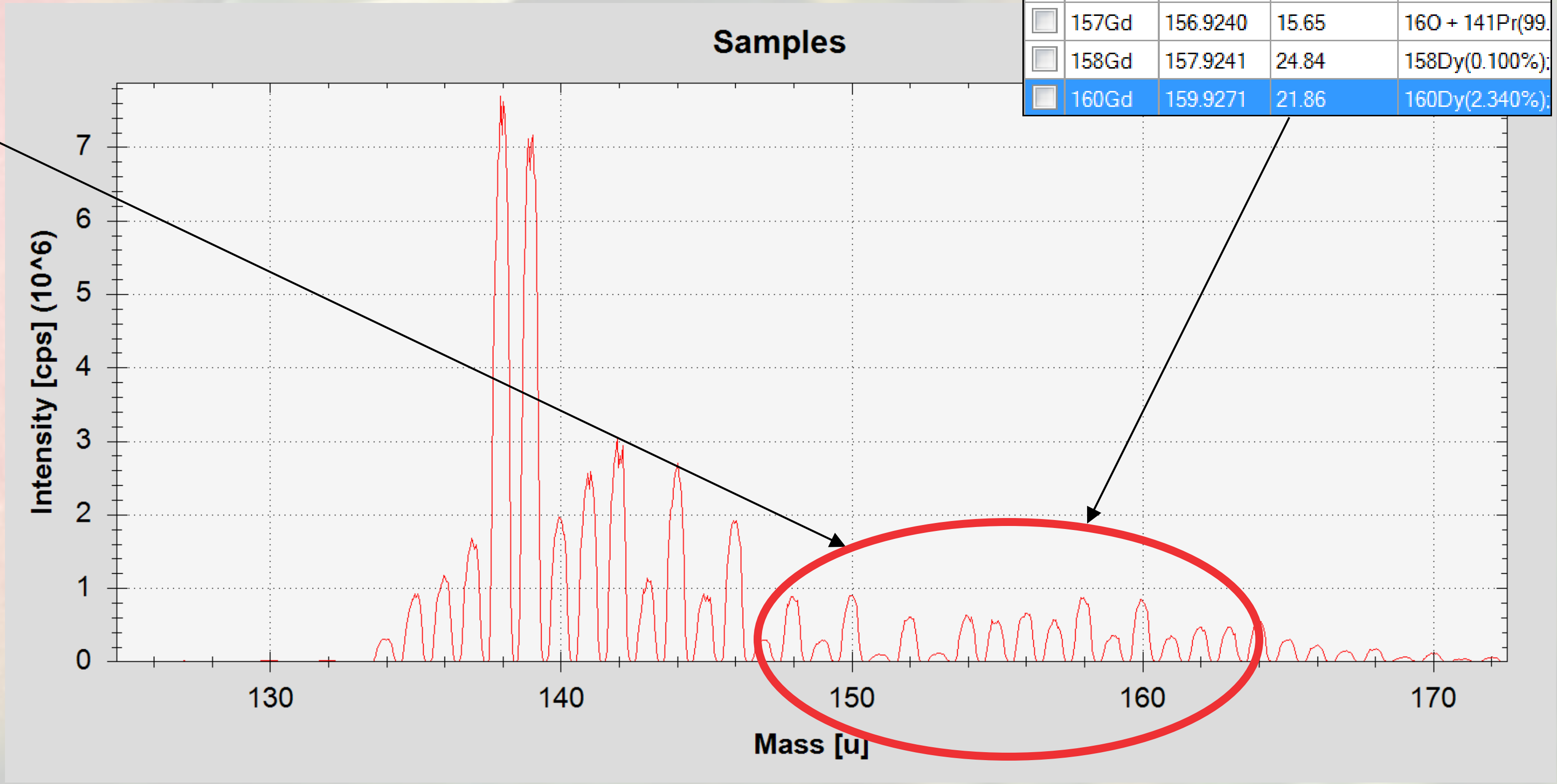


Rare Earth Elements in Apple Leaves (NIST 1515)

Nd and Gd isotopic profile in NIST 1515 Apple Leaves

	Symbol	Mass	Abundance	Interferences
☐	142Nd	141.9077	27.13	142Ce(11.080%
☐	143Nd	142.9098	12.18	16O + 127I(99.76
☐	144Nd	143.9101	23.80	144Sm(3.100%)
☐	145Nd	144.9126	8.30	12C + 133Cs(98
☐	146Nd	145.9131	17.19	130Ba + 16O(0.1
☐	148Nd	147.9169	5.76	148Sm(11.300%
☑	150Nd	149.9209	5.64	150Sm(7.400%)

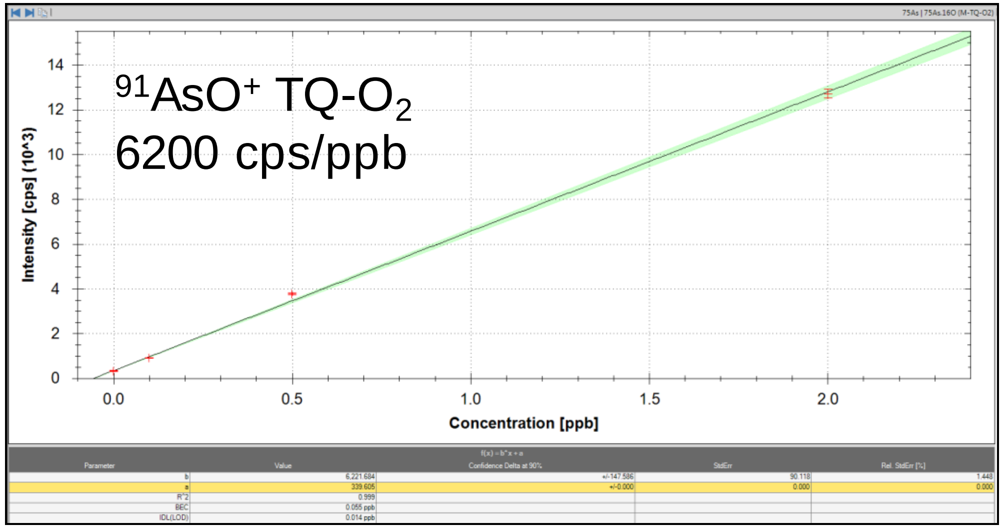
	Symbol	Mass	Abundance	Interferences
☐	152Gd	151.9198	0.20	152Sm(26.700%
☐	154Gd	153.9209	2.18	154Sm(22.700%
☐	155Gd	154.9226	14.80	16O + 139La(99
☐	156Gd	155.9221	20.47	138Ce + 18O(0.0
☐	157Gd	156.9240	15.65	16O + 141Pr(99.
☐	158Gd	157.9241	24.84	158Dy(0.100%);
☐	160Gd	159.9271	21.86	160Dy(2.340%);

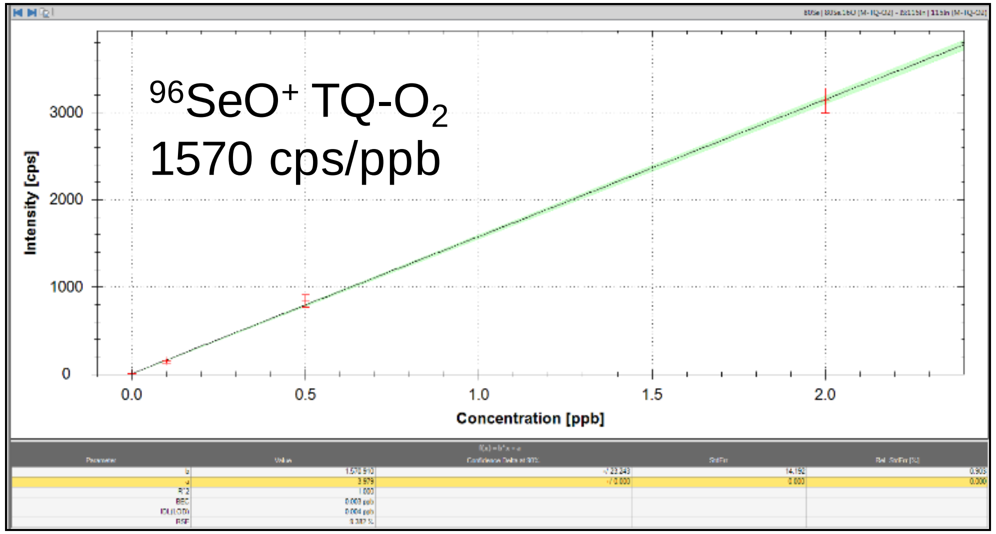


REE will lead to false positive results for As and Se – only triple quadrupole based ICP-MS can overcome these

Comparison of SQ KED vs TQ-O_2 in NIST 1515

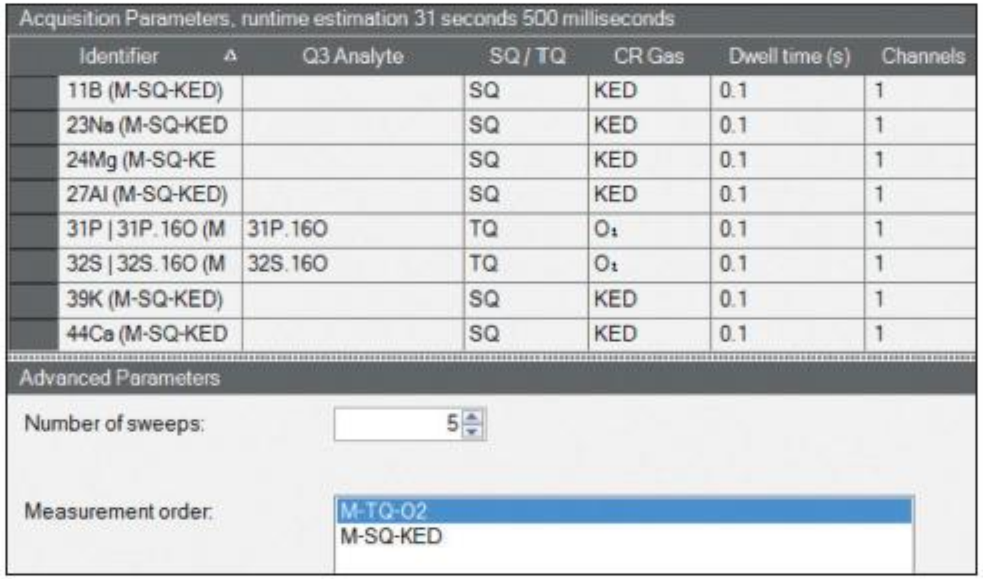
Analyte	Reference value mg/kg	SQ KED mode mg/kg	TQ-O_2 mode mg/kg
^{75}As	0.038	1.730	0.036
^{80}Se	0.050	4.800	0.052





Setup for analysis

- Multielement Analysis – one run fits all!
  - For most elements KED is still the best choice
  - For others, TQ modes can offer better detection limits through advanced interference removal
  - Reaction Finder automatically determines the appropriate settings and fully automizes scanning during a sample run



Acquisition Parameters, runtime estimation 31 seconds 500 milliseconds

Identifier	Q3 Analyte	SQ / TQ	CR Gas	Dwell time (s)	Channels
11B (M-SQ-KED)		SQ	KED	0.1	1
23Na (M-SQ-KED		SQ	KED	0.1	1
24Mg (M-SQ-KE		SQ	KED	0.1	1
27Al (M-SQ-KED)		SQ	KED	0.1	1
31P | 31P.16O (M	31P.16O	TQ	O_2	0.1	1
32S | 32S.16O (M	32S.16O	TQ	O_2	0.1	1
39K (M-SQ-KED)		SQ	KED	0.1	1
44Ca (M-SQ-KED		SQ	KED	0.1	1

Advanced Parameters

Number of sweeps: 5

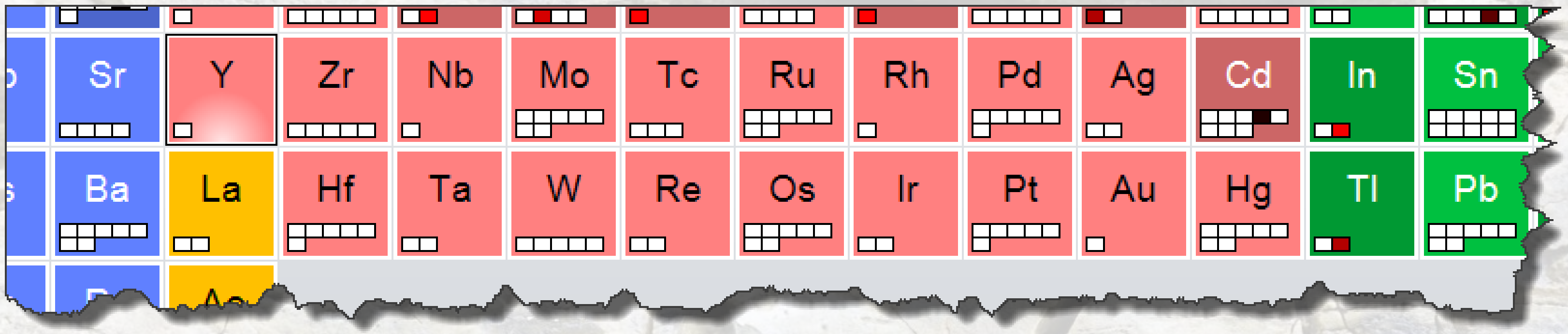
Measurement order: M-TQ-O2
M-SQ-KED

Analyte	Measurement mode	Internal standard
^{11}B	SQ-KED	^{45}Sc
^{23}Na	SQ-KED	^{45}Sc
^{24}Mg	SQ-KED	^{45}Sc
^{27}Al	SQ-KED	^{45}Sc
^{31}P as ^{31}P^{16}O at *47 m/z*	TQ-O_2	^{115}In
^{32}S as ^{32}S^{16}O at *48 m/z*	TQ-O_2	^{115}In
^{39}K	SQ-KED	^{45}Sc
^{44}Ca	SQ-KED	^{45}Sc
^{51}V	SQ-KED	^{71}Ga
^{52}Cr	SQ-KED	^{71}Ga
^{55}Mn	SQ-KED	^{71}Ga
^{57}Fe	SQ-KED	^{71}Ga
^{59}Co	SQ-KED	^{71}Ga
^{60}Ni	SQ-KED	^{71}Ga
^{63}Cu	SQ-KED	^{71}Ga
^{66}Zn	SQ-KED	^{71}Ga
^{75}As as ^{75}As^{16}O at *91 m/z*	TQ-O_2	^{115}In
^{75}As	SQ-KED	^{115}In
^{78}Se as ^{78}Se^{16}O at *94 m/z*	TQ-O_2	^{115}In
^{78}Se	SQ-KED	^{115}In
^{85}Rb	SQ-KED	^{115}In
^{88}Sr	SQ-KED	^{115}In
^{98}Mo	SQ-KED	^{115}In
^{111}Cd	SQ-KED	^{115}In
^{121}Sb	SQ-KED	^{115}In
^{138}Ba	SQ-KED	^{159}Tb
^{208}Pb	SQ-KED	^{159}Tb
^{232}Th	SQ-KED	^{159}Tb
^{238}U	SQ-KED	^{159}Tb

Multielemental Analysis in Apple and Tomato leaves with TQ-ICP-MS

- Excellent agreement to certified and reported values achieved for all elements
- Accurate analysis for As and Se despite the REE in matrix
- Full multi-elemental analysis, with a mix of comprehensive and dedicated interference removal in one sample run
- Option to quantify S and P at low levels with TQ-O_2 mode

Analyte	IDL (μg·L^{-1})	MDL (μg·kg^{-1})	NIST 1515 apple leaves N=3		NIST 1573A tomato leaves N=3	
			Measured (mg·kg^{-1})	Certified (mg·kg^{-1})	Measured (mg·kg^{-1})	Certified (mg·kg^{-1})
^{11}B	0.4	52.4	25.2±0.6	27±2	30.0±0.2	33.3±0.7
^{23}Na	4	524	29.3±0.8	24.4±1.2	123±1.5	136±4
^{24}Mg	1	131	2686±84	2710±80	10556±210	12000*
^{27}Al	0.1	13.1	258±8.4	286±9	532±13	598±12
^{31}P as ^{31}P^{16}O at *m/z* 47	0.05	6.0	1530±10	1590*	2040±33	2160±40
^{32}S as ^{32}S^{16}O at *m/z* 48	0.02	2.6	1819±15	1800*	9779±0.2	9600*
^{39}K	3	393	16106±75	16100±200	27299±198	27000±500
^{44}Ca	1	131	15218±2300	15260±1500	49339±235	50500±900
^{51}V	0.001	0.13	0.24±0.01	0.26±0.03	0.80±0.01	0.835±0.010
^{52}Cr	0.005	0.66	0.29±0.01	0.3*	1.97±0.02	1.99±0.06
^{55}Mn	0.003	0.39	52.6±0.6	54±3	242±1.9	246±8
^{57}Fe	1	131	81.5±1.6	80*	366±4.8	368±7
^{59}Co	0.011	1.44	0.08±0.003	0.09*	0.57±0.01	0.57±0.02
^{60}Ni	0.023	3.01	0.85±0.13	0.91±0.12	1.57±0.02	1.59±0.07
^{63}Cu	0.088	11	5.59±0.05	5.64±0.24	4.7±0.1	4.7±0.14
^{66}Zn	0.026	3.41	11.3±0.16	12.5±0.3	28.2±0.37	30.9±0.7
^{75}As as ^{75}As^{16}O at *m/z* 91	0.006	0.79	0.036±0.003	0.038±0.007	0.117±0.03	0.112±0.004
^{75}As	0.004	0.52	0.469±0.012	0.038±0.007	0.143±0.01	0.112±0.004
^{78}Se as ^{78}Se^{16}O at *m/z* 94	0.003	0.39	0.052±0.006	0.050±0.009	0.053±0.01	0.054±0.003
^{78}Se	0.046	6.03	1272±187	0.050±0.009	0.11±0.01	0.054±0.003
^{85}Rb	0.004	0.52	9.1±0.17	9*	13.97±0.03	14.89±0.27
^{88}Sr	0.002	0.26	25.5±0.34	25±2	84.8±0.49	85*
^{98}Mo	0.003	0.39	0.094±0.01	0.094±0.013	0.47±0.06	0.46*
^{111}Cd	0.001	0.13	0.013±0.001	0.014*	1.45±0.03	1.52±0.04
^{121}Sb	0.001	0.13	0.012±0.005	0.013*	0.057±0.003	0.063±0.006
^{138}Ba	0.002	0.262	48.8±0.1	49±2	60.2±0.7	63*
^{208}Pb	0.001	0.131	0.422±0.002	0.470±0.024	-	N.D.
^{232}Th	0.001	0.131	0.03±0.002	0.03	0.107±0.002	0.12*
^{238}U	0.001	0.131	0.008±0.002	0.006*	0.033±0.001	0.035*



- Main interest due to:
  - Mining/Exploration of Ores/Recycling
  - Geological Research
  - Environmental & Food Safety Analysis

- “Normally“ interference free

Recommended / Information Values for GSP 2

United States Geological Survey
Certificate of Analysis
Granodiorite, Silver Plume, Colorado, GSP-2

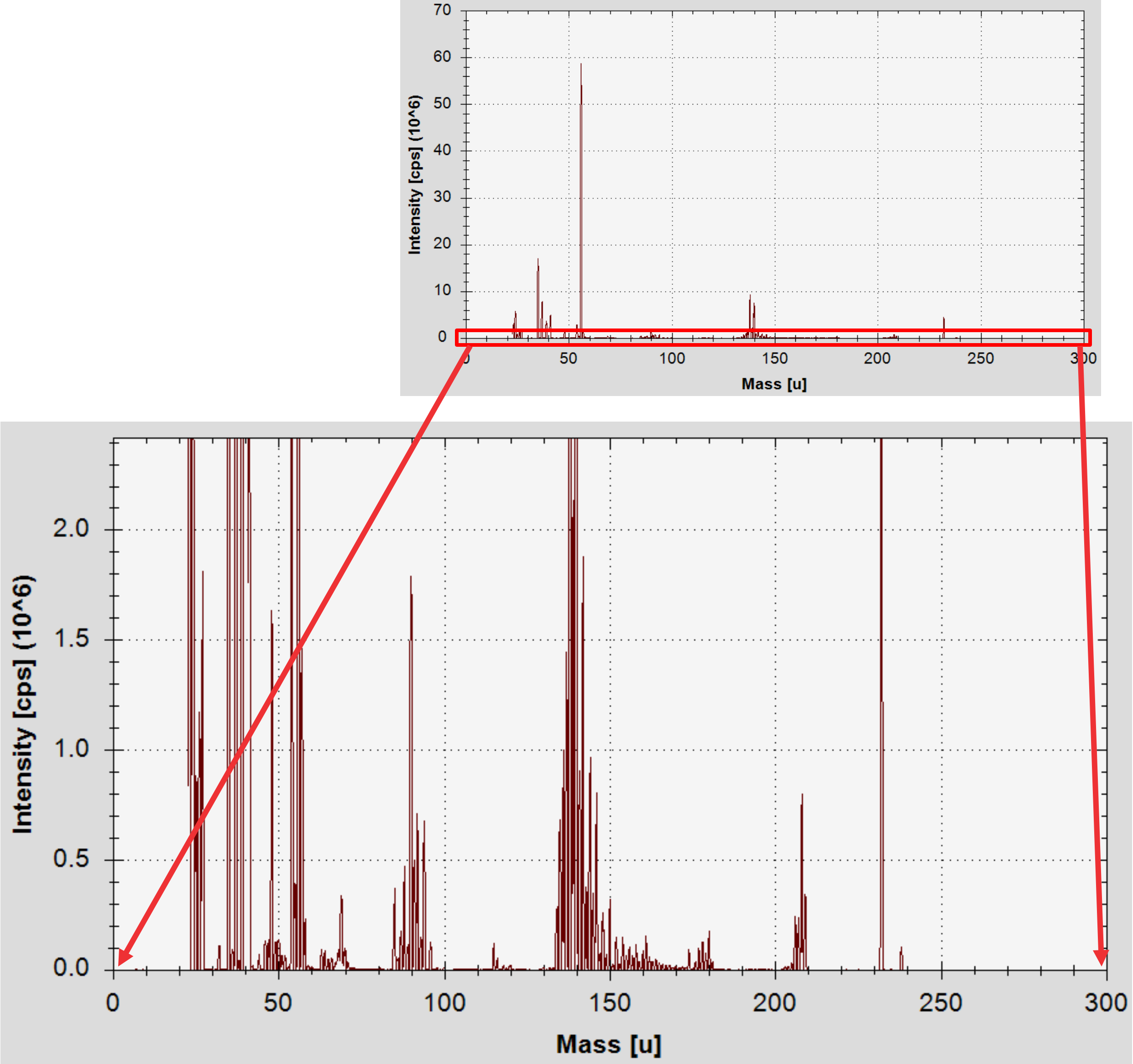
Recommended values

Element	Wt %	±	Oxide	Wt %	±
Al	7.88	0.11	Al_2O_3	14.9	0.2
Ca	1.50	0.04	CaO	2.10	0.06
Fe_{tot}	3.43	0.11	$Fe_2O_{3\ tot}$	4.90	0.16
K	4.48	0.12	K_2O	5.38	0.14
Mg	0.58	0.02	MgO	0.96	0.03
Na	2.06	0.07	Na_2O	2.78	0.09
P	0.13	0.01	P_2O_5	0.29	0.02
Si	31.1	0.4	SiO_2	66.6	0.8
Ti	0.40	0.01	TiO_2	0.66	0.02

Element	µg/g	±	Element	µg/g	±
Ba	1340	44	Pb	42	3
Ce	410	30	Rb	245	7
Co	7.3	0.8	Sc	6.3	0.7
Cr	20	6	Sm	27	1
Cu	43	4	Sr	240	10
Eu	2.3	0.1	Th	105	8
Ga	22	2	U	2.40	0.19
La	180	12	V	52	4
Mn	320	20	Y	28	2
Nb	27	2	Yb	1.6	0.2
Nd	200	12	Zn	120	10
Ni	17	2	Zr	550	30

Information values

Element	µg/g	±	Element	µg/g	±
Be	1.5	0.2	Ho	1.0	0.1
Cs	1.2	0.1	Li	36	1
Dy	6.1		Lu	0.23	0.03
Er	2.2		Mo	2.1	0.6
F	3000		Pr	51	5
Gd	12	2	Tl	1.1	
Hf	14	1	Tm	0.29	0.02



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Interferences on Platium Group Metals

Hf

	Symbol	Mass	Abundance
☐	174Hf	173.9401	0.16
☐	176Hf	175.9414	5.21
☐	177Hf	176.9432	18.61
☑	178Hf	177.9437	27.30
☑	179Hf	178.9458	13.63
☐	180Hf	179.9466	35.10

$^{178}Hf^{16}O^{+}$, $^{179}Hf^{16}O^{+}$
$^{177}Hf^{16}O^{1}H^{+}$, $^{178}Hf^{16}O^{1}H^{+}$

	Symbol	Mass	Abundance
☐	190Pt	189.9599	0.01
☐	192Pt	191.9610	0.79
☑	194Pt	193.9627	32.90
☑	195Pt	194.9648	33.80
☐	196Pt	195.9649	25.30
☐	198Pt	197.9679	7.20

Pt

Interferences on Platinum Group Metals

Hf

	Symbol	Mass	Abundance
☐	174Hf	173.9401	0.16
☐	176Hf	175.9414	5.21
☐	177Hf	176.9432	18.61
☑	178Hf	177.9437	27.30
☑	179Hf	178.9458	13.63
☐	180Hf	179.9466	35.10

$^{178}Hf^{16}O^{+}$, $^{179}Hf^{16}O^{+}$
$^{177}Hf^{16}O^{1}H^{+}$, $^{178}Hf^{16}O^{1}H^{+}$ →

	Symbol	Mass	Abundance
☐	190Pt	189.9599	0.01
☐	192Pt	191.9610	0.79
☑	194Pt	193.9627	32.90
☑	195Pt	194.9648	33.80
☐	196Pt	195.9649	25.30
☐	198Pt	197.9679	7.20

Pt

Sr → $^{87}Sr^{16}O^{+}$

Cu → $^{63}Cu^{40}Ar^{+}$

Y → $^{89}Y^{14}N^{+}$

Rh

	Symbol	Mass	Abundance
☑	103Rh	102.9055	100.00

$^{206}Pb^{2+}$ ← Pb

Interferences on Platium Group Metals

Hf

	Symbol	Mass	Abundance
☐	174Hf	173.9401	0.16
☐	176Hf	175.9414	5.21
☐	177Hf	176.9432	18.61
☑	178Hf	177.9437	27.30
☑	179Hf	178.9458	13.63
☐	180Hf	179.9466	35.10

$^{178}Hf^{16}O^{+}$, $^{179}Hf^{16}O^{+}$
$^{177}Hf^{16}O^{1}H^{+}$, $^{178}Hf^{16}O^{1}H^{+}$

	Symbol	Mass	Abundance
☐	190Pt	189.9599	0.01
☐	192Pt	191.9610	0.79
☑	194Pt	193.9627	32.90
☑	195Pt	194.9648	33.80
☐	196Pt	195.9649	25.30
☐	198Pt	197.9679	7.20

Pt

Polyatomic

Sr → $^{87}Sr^{16}O^{+}$

Cu → $^{63}Cu^{40}Ar^{+}$

Y → $^{89}Y^{14}N^{+}$

Rh

	Symbol	Mass	Abundance
☑	103Rh	102.9055	100.00

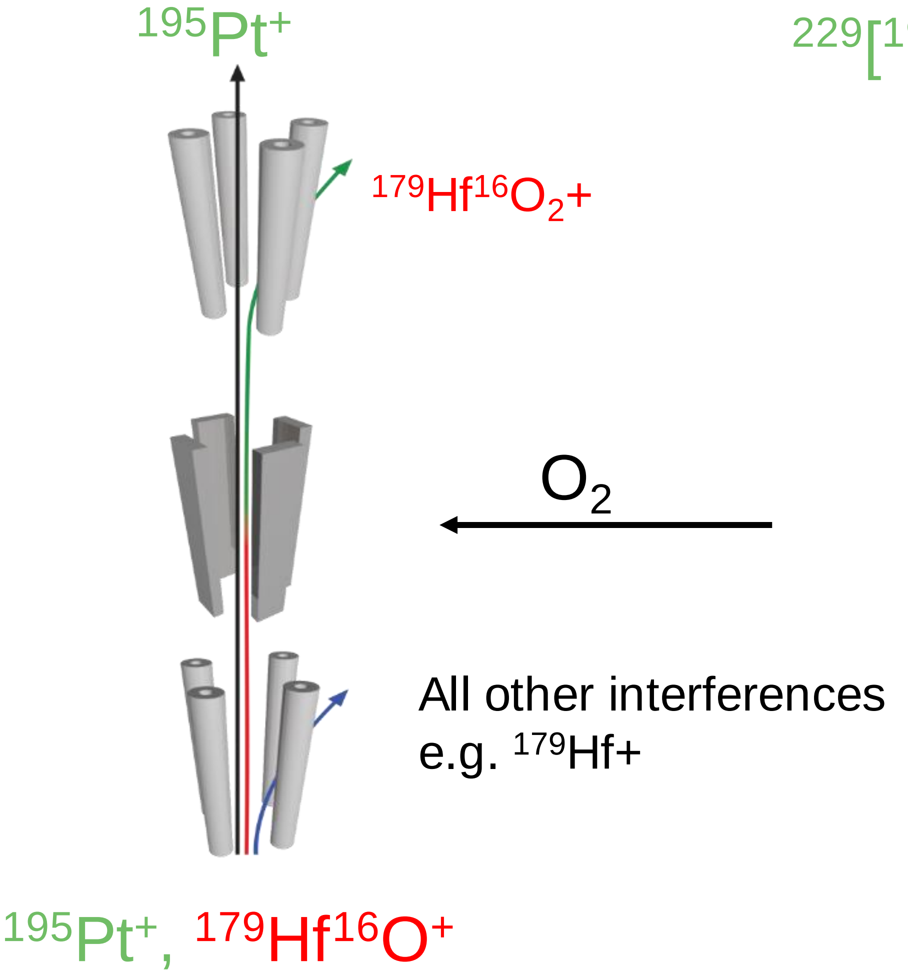
Doubly charged

$^{206}Pb^{2+}$ ← Pb

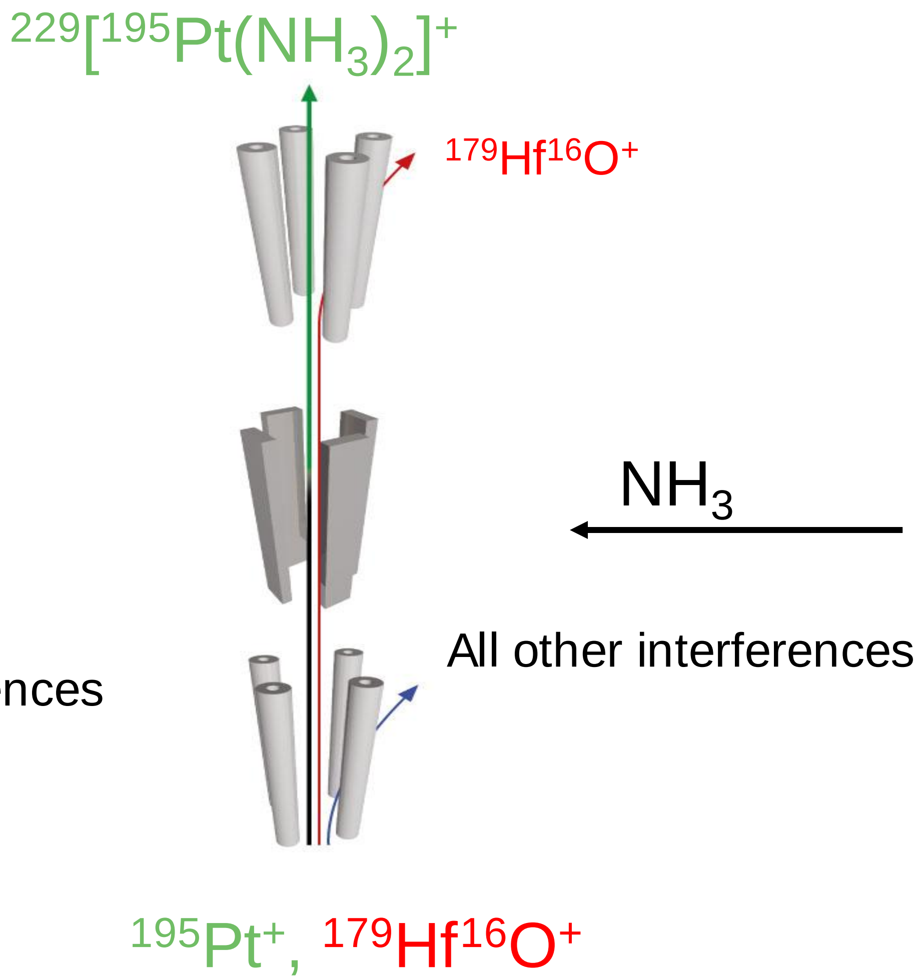
Interferences on Platinum Group Metals

Analyte	Interfering Elements	Type
Zr	Y, Ge	Polyatomic
Nb	Br	Polyatomic
Rh	Sr, Y, Cu	Polyatomic
Ru	Mo, Pd	Isobaric
Pd	Y, Ru, Cd	Isobaric
Hf	Yb, Ho	Both
Ta	Hf, Ho	Both
Ir	Lu, Ta	Polyatomic
Pt	Hf, Hg	Both
Au	Ta	Polyatomic

Interference Removal using TQ-ICP-MS



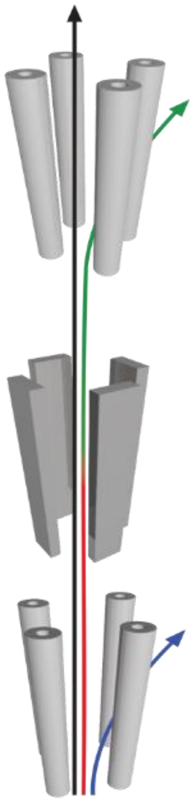
“On Mass“



“Mass Shift“

Interference Removal using TQ-ICP-MS

$^{195}Pt^+$



$^{179}Hf^{16}O_2+$

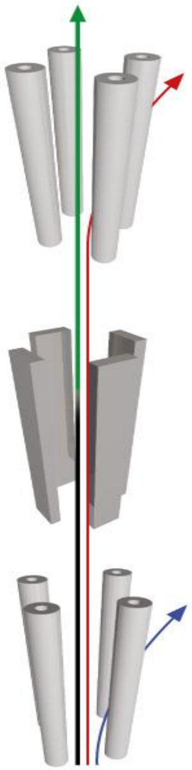
O_2

All other interferences
e.g. $^{179}Hf+$

$^{195}Pt^+$, $^{179}Hf^{16}O^+$

“On Mass“

$^{229}[^{195}Pt(NH_3)_2]^+$



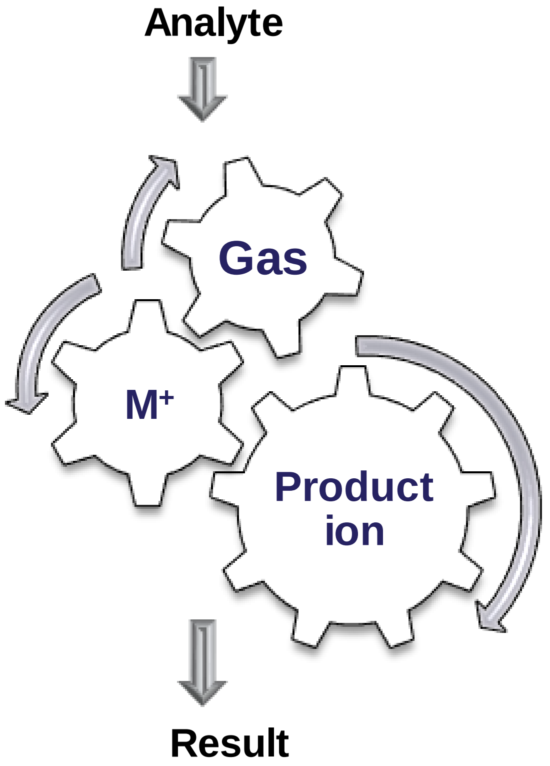
$^{179}Hf^{16}O^+$

NH_3

All other interferences

$^{195}Pt^+$, $^{179}Hf^{16}O^+$

“Mass Shift“



Reaction Finder helps to set up for analysis!

Sample Preparation

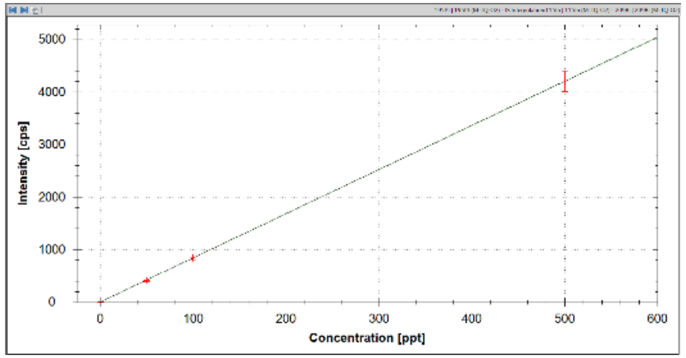
- 50mg of sample
- Addition of 2mL of HF and 0.5mL of HNO_3, cover and remain at room temperature for 6-8h
- Evaporate to dryness
- Addition of 2mL of HF, 0.5mL of $HCLO_4$ and 0.2mL of HNO_3, autoclave digstion at up to 220°C for 3.5 h
- Evaporate to dryness
- Addition of 1mL HCl and 1 mL HNO_3
- Evaporate to dryness
- Addition of 0.8mL HCl and 0.8mL of HNO_3
- Make up to 10mL final volume

- Additional 20-fold dilution before analysis
- All dilutions were made in 3% HCl, 1% HNO_3 to increase assure stability of PGM in solutions
- Rinse solution used contained same acid matrix

Digestion procedure described in Karandashev et al., Zavodskaya Laborarorija, 2016, p. 6-15

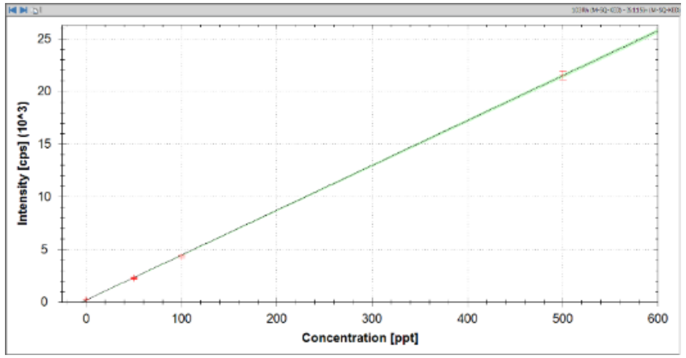
Results

- ^{195}Pt: Analysed on mass (O_2) and using mass shift (NH_3, $^{195}Pt^+ \rightarrow [^{195}Pt(NH_3)_2]^+$)



Sample	Result [ppt]		Recovery [%]	
	TQ-O_2	TQ-NH_3	TQ-O_2	TQ-NH_3
ICV (50ppt)	45.2	53.0	106	90
CCV (average, N=7)	-	-	95.2 ± 3.6	95.0 ± 6.4
GSP unspiked (N = 6)	4.64 ± 0.5	5.60 ± 1.3	-	-
GSP spiked 10ppt (N = 6)	16.01 ± 2.4	16.59 ± 2.4	114	110
GSP spiked 25ppt (N = 6)	29.88 ± 2.8	29.24 ± 4.7	101	95
Sensitivity [cts·(µg·L^{-1})$^{-1}$]	8,406	2,144		
Detection Limit [ng·L^{-1}]	0.1 ppt	0.7 ppt		

- ^{103}Rh: Analyzed in SQ-KED and TQ-O_2



Sample	Result [ppt]		Recovery [%]	
	SQ-KED	TQ-O_2	SQ-KED	TQ-O_2
ICV (50ppt)	50.8	51.5	102	103
CCV (average, N=7)	-	-	97.6 ± 3.1	92.0 ± 8.3
GSP unspiked (N = 6)	Not detected	Not detected	-	-
GSP spiked 10ppt (N = 3)*	10.86 ± 0.6	10.56 ± 0.2	108	106
GSP spiked 25ppt (N = 3)*	26.06 ± 1.8	23.27 ± 0.7	104	93
Sensitivity [cts·(µg·L^{-1})$^{-1}$]	42,568	23,208		
Detection Limit [ng·L^{-1}]	0.4 ppt	2.0 ppt		

Mineral Sample Results

African Mineral Standards Certified Reference Material AMIS0416

Element	Value [g·t^{-1}]	Result [g·t^{-1}]	Mode	Recovery [%]
Rh	0.29 ± 0.04	0.27 ± 0.04	^{103}Rh, TQ-O_2	93.1 ± 13
Pd	0.80 ± 0.06 (Cert., NiS Collection)	0.73 ± 0.05	^{108}Pd, TQ-O_2	91.3 ± 6.3
	0.75 ± 0.04 (Prov., Pb Collection)			97.3 ± 6.7
Ir	0.13 ± 0.02	0.14 ± 0.02	^{191}Ir, TQ-O_2	107.6 ± 15.3
Pt	1.46 ± 0.18 (Pb Collection)	1.27 ± 0.14	^{194}Pt, TQ-NH_3	86.9 ± 10.3
		1.33 ± 0.14	^{194}Pt, TQ-O_2	91.1 ± 9.6
		1.30 ± 0.15	^{195}Pt, TQ-NH_3	89.0 ± 10.3
		1.31 ± 0.19	^{195}Pt, TQ-O_2	89.7 ± 13.0
Au	0.14 ± 0.04 (Pb Collection)	0.073 ± 0.02	^{197}Au, TQ-NH_3	52.1 ± 14.3*
	0.13 ± 0.02 (NiS Collection)	0.086 ± 0.03	^{197}Au, TQ-O_2	61.4 ± 21.4*

* Potentially stability issues in sample preparation and storage

Trace Metals in Pharmaceutical Products and Cell Culture Media

- Inorganic impurities in pharmaceutical products are of great concern due to their inherent toxicity and adverse effects on drug efficacy, stability and shelf-life.

- The United States Pharmacopeia (USP) General Chapters <232> and <233> are ICP specific replacements for an outdated and non-specific colorimetric test prescribed by USP <231>.

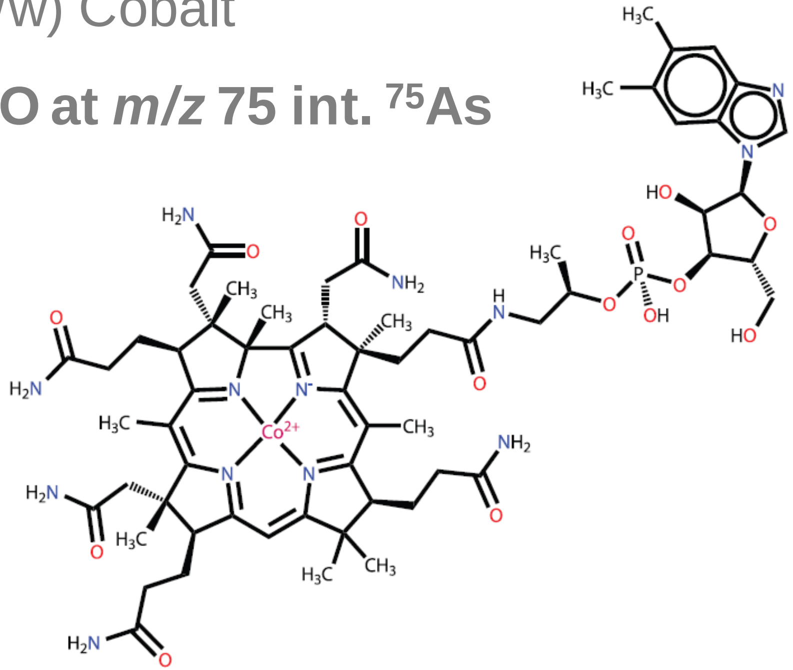
Chapter <232>	**Chapter <233>**
Sets out permissible daily exposure limits from well characterized toxicity studies of 15 elements	Sets out sample preparation and analysis methods by ICP-OES and ICP-MS

Other methods can be used as long as they have been validated and shown to provide appropriate limits of detection, precision and specificity for the metal impurities.

Determination of elemental impurities in Vitamin B12

Vitamin B12 contains approx. 4% (w/w) Cobalt

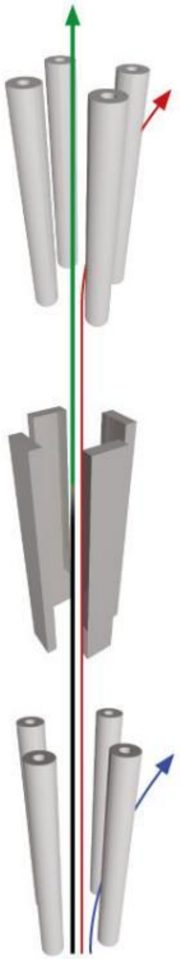
$^{59}Co^{16}O$ at *m/z* 75 int. ^{75}As



Q3 isolates ions required for measurement (e.g. set to product ion mass for As^+ at *m/z* 91)

Q2 pressurized with reactive gas to selectively generate reaction products (e.g. O_2 + As^+ → AsO^+)

Q1 set to analyte mass (e.g. As^+ at *m/z* 75) to filter out unwanted ions



Determination of Arsenic in Vitamin B12 in SQ and TQ Modes

Concentration Vitamin B12	Signal at *m/z*=59 (SQ-KED) [CPS]	Signal at *m/z*=75 (SQ-KED) [CPS]	BEC in SQ-KED mode [$ng \cdot g^{-1}$]	Signal at *m/z*=75 ($TQ-O_2$)	BEC in $TQ-O_2$ mode [$ng \cdot g^{-1}$]
BLK	73	2	0.0008	4	0.0007
0.0001 $mg \cdot mL^{-1}$	202,455	13	0.003	9	0.001
0.001 $mg \cdot mL^{-1}$	2,174,144	88	0.02	10	0.001
0.01 $mg \cdot mL^{-1}$	24,003,087	852	0.21	8	0.001
0.1 $mg \cdot mL^{-1}$	243,093,619	8744	2.47	18	0.002

- SQ-KED mode elevated BEC due to CoO contribution that cannot be suppressed with He KED
- $TQ-O_2$ mode - measuring AsO at *m/z* 91 is free from CoO interference and shows single digit $ng \cdot g^{-1}$ BEC regardless of Vitamin B12 concentration

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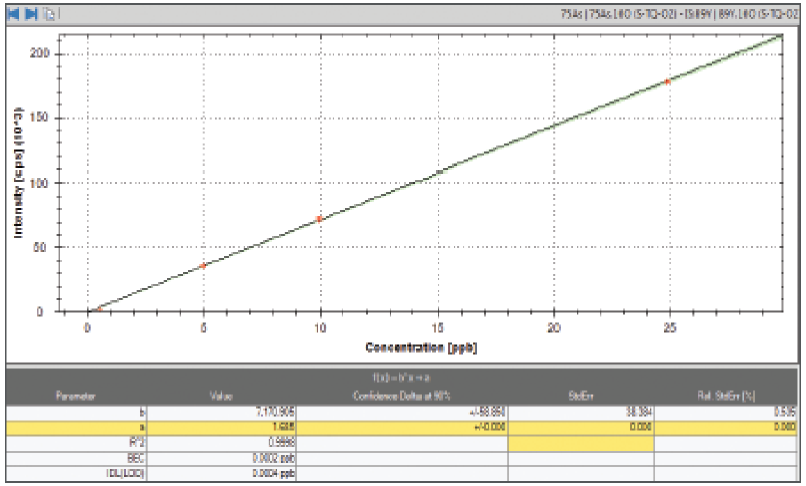
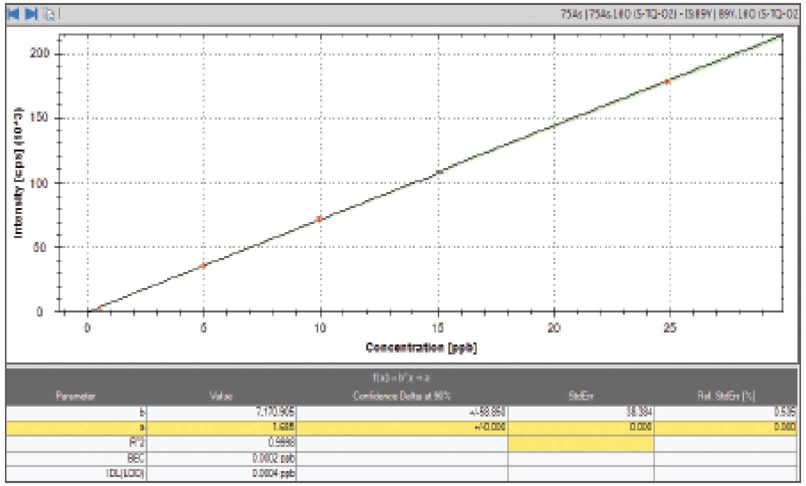
Calibration and Results for a Vitamin B12 Supplement



- Sample prepared according to manufacturers instructions, then diluted 1:100 for analysis
- As Sensitivity – 7000 cps·ppb^{-1}
- BEC – 0.0002 ng·g^{-1}
- Spike recovery of As, Cd, Hg, Pb determined

Element	Mode	Result [ng·g^{-1}]	Spiked concentration [ng·g^{-1}]	Recovery [%]	Recovery with butanol [%]
As	TQ-O_2	0.019	9.87	179	99
As	SQ-KED	0.150	9.87	171	100
Cd	SQ-KED	Below IDL	3.29	97	98
Hg	SQ-KED	0.05	1.32	98	98
Pb	SQ-KED	0.1	6.58	105	107

Calibration and Results for a Vitamin B12 Supplement



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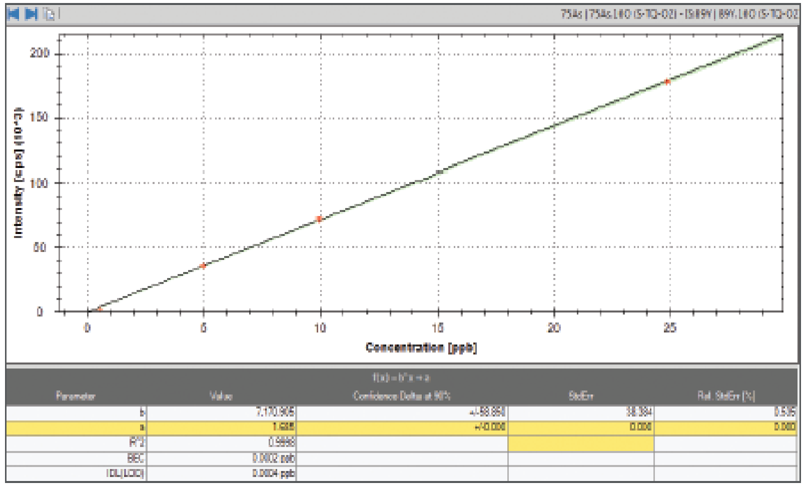
Pb	SQ-KED	0.1	6.58	105	107

Increased recovery for As due to ionization effects in the sample solution! Addition of butanol to homogenize carbon content

Calibration and Results for a Vitamin B12 Supplement



- Sample prepared according to manufacturers instructions, then diluted 1:100 for analysis
- As Sensitivity – 7000 cps·ppb^{-1}
- BEC – 0.0002 ng·g^{-1}
- Spike recovery of As, Cd, Hg, Pb determined

Element	Mode	Result [ng·g^{-1}]	Spiked concentration [ng·g^{-1}]	Recovery [%]	Recovery with butanol [%]
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Cd	SQ-KED	Below IDL	3.29	97	98
Hg	SQ-KED	0.05	1.32	98	98
Pb	SQ-KED	0.1	6.58	105	107

10 times higher reported concentration in SQ KED mode for As than TQ mode due to the CoO interference

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Performance and Simplicity for Ultraclean Applications

- ✓ **Improving wafer yields by ensuring ultra clean chemicals and materials**
- ✓ **Automated method development for reliable QA/QC**
- ✓ **On-line chemical analysis solution as well as laboratory QA/QC**
- ✓ **Performance to sub-ppt levels**

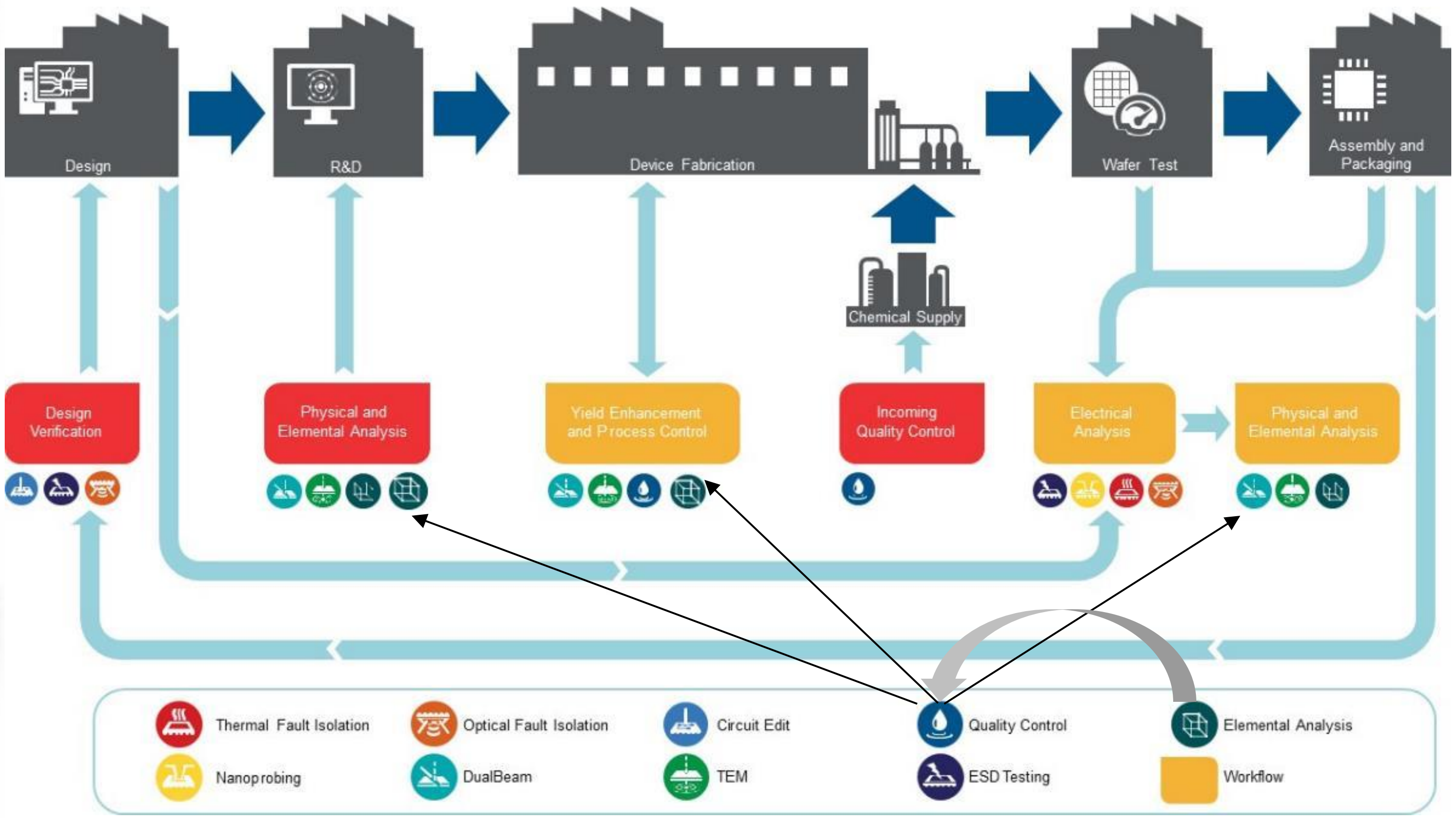
Semiconductor Analysis Applications

- ✓ Wafer VPD and high Si matrix
- ✓ Photoresist/Organic solvents
- ✓ Ultra clean chemicals
- ✓ Aggressive Acids – HF, HNO3 etc
- ✓ On-line chemical analysis close to wafer production

Ultra-high Chemical and Material QA/QC

- ✓ Chemicals – acids, organics
- ✓ Metals – Zn, Cu, W, Ag, Pt
- ✓ Materials - Silicon
- ✓ Ultra high purity water
- ✓ Gas purity

Performance and Simplicity for Ultraclean Applications



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Thermo Scientific iCAP Qnova Series ICP-MS

The iCAP RQ ICP-MS and the iCAP TQs ICP-MS, built on the same robust platform, have shared capabilities for ease of use and powerful ultralow detection. Ideal for the laboratory or for real time process quality control in the semiconductor manufacturing line.

- Ergonomic, compact design, ideal for integration with on-line monitoring installations and vapor phase decomposition (VPD) solutions
- Chemically inert and quick fit sample introduction system ensure minimal contamination and reproducible, consistent data
- Robust performance with a combination of hot and cold plasma operation in one run ensures data quality and productivity
- Streamlined and automated daily tasks with intuitive software workflows
- Easy installation and minimal bench space requirements in clean room and production facilities

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Thermo Scientific iCAP TQs ICP-MS

Push the boundaries of detection with triple quadrupole ICP-MS performance and ease of use for QA/QC analysis in the wafer fabrication process with the iCAP TQs ICP-MS

- Powerful triple quadrupole technology for improved interference removal and right first time analysis
- Ultralow detection, even in challenging matrices
- Easy and fast set-up using the unique **Reaction Finder** method development assistant
- Reliable instrument operation through integrated reaction gas handling features
- Dry fore-vacuum pump for clean room environments

ThermoFisher SCIENTIFIC

Integration into Lab and Fab Workflows

The intelligent design of the iCAP Qnova Series ICP-MS is ideal for integration with automated sample handling systems and specialized equipment

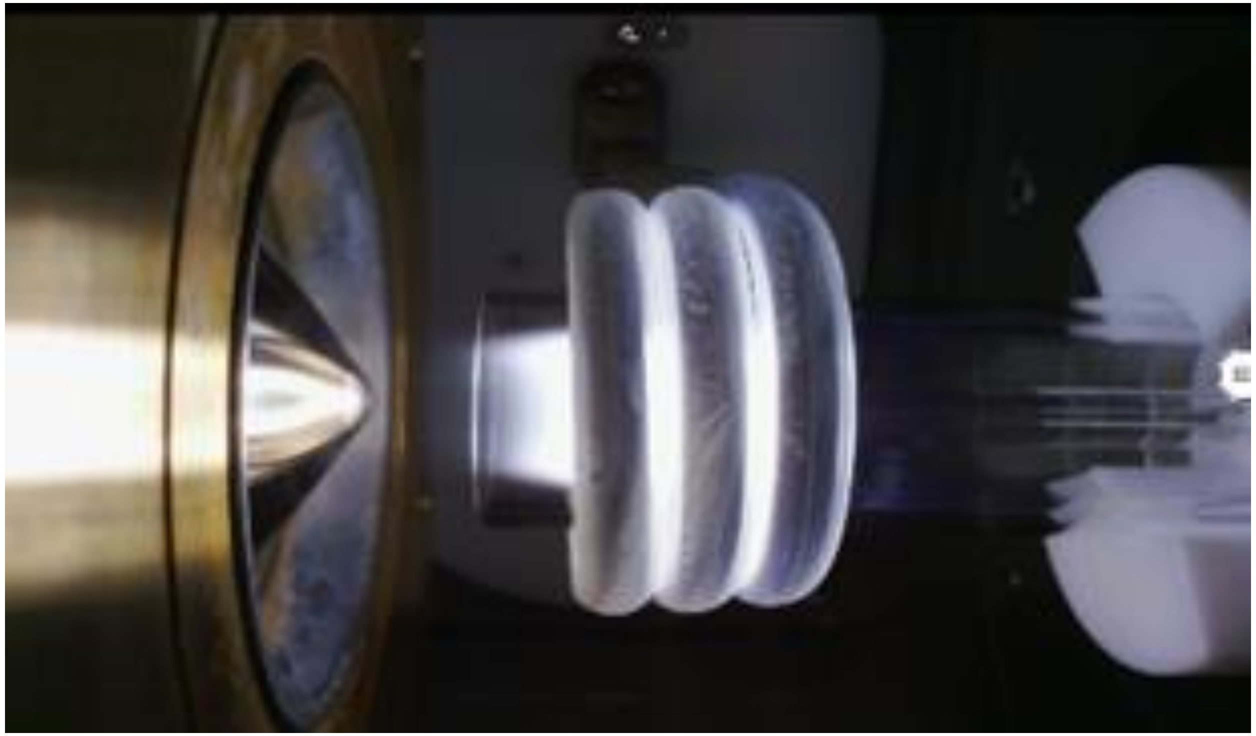
Automation Compatibility with a range of autosampler and autodilution accessories for direct analysis of high purity chemicals.

On-line monitoring Real-time statistical control of semiconductor process chemicals is enabled through integration with in-line automated process monitoring tools.

Vapor Phase Decomposition Integration with VPD solutions delivers an efficient and reproducible solution for wafer contamination control at ultralow levels.

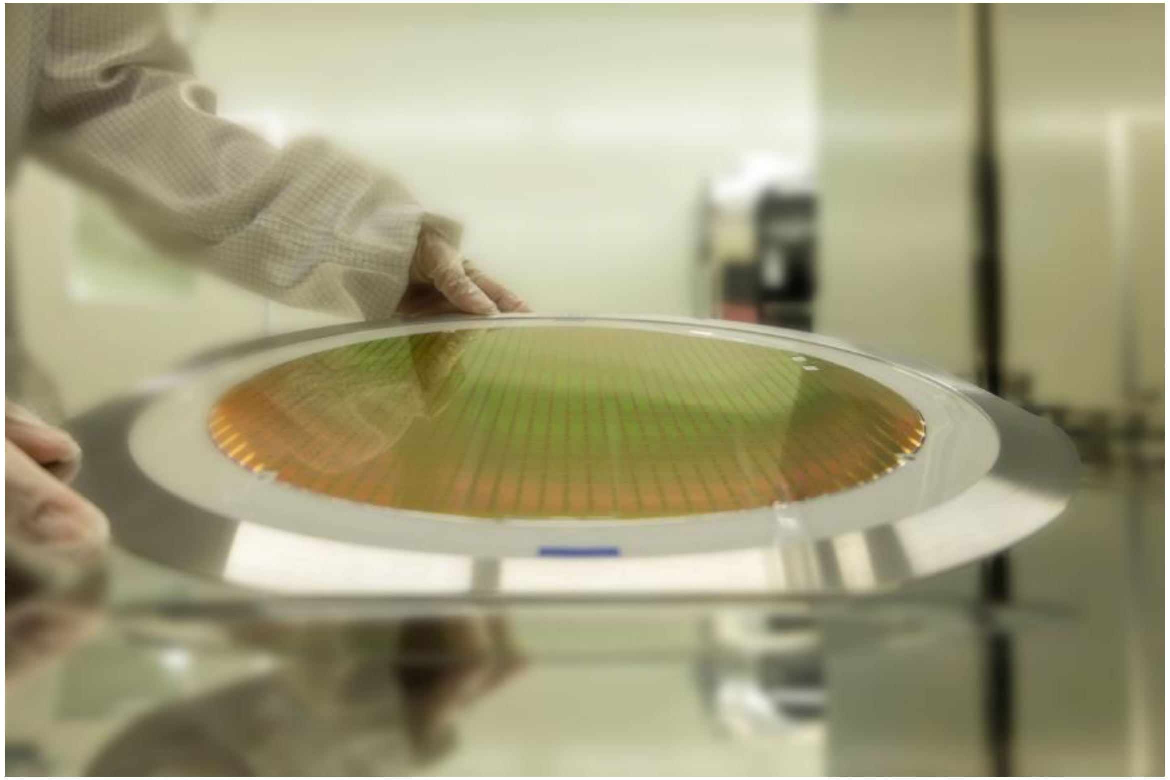
Typical Methodology for Semiconductor Applications

- Cold Plasma (CP) Operation
  - Lower forward power (< 600 W)
  - Reduced argon and carbon ionization – fewer polyatomic interferences
- CP, Kinetic Energy Discrimination (KED) and Triple Quadrupole (TQ) modes combined in one method
  - Remove all polyatomic interferences at once
  - Some analytes are more sensitive under hot plasma conditions



Determination of Ultratrace Elements in Photoresist Solvents

- Propylene glycol methyl ether acetate (PGMEA) and N-methyl-2-pyrrolidone (NMP):
  - Base organic solvents of semiconductor photoresists
  - Direct contact with wafer surfaces, impurities must be tightly controlled
- Simple ICP-MS analytical method should:
  - Detect ultratrace (ng·L^{-1}) levels of analytes
  - Avoid contamination caused by sample preparation
- PGMEA and NMP considered difficult matrices due to:
  - High volatility
  - High carbon content: significant polyatomic interferences!



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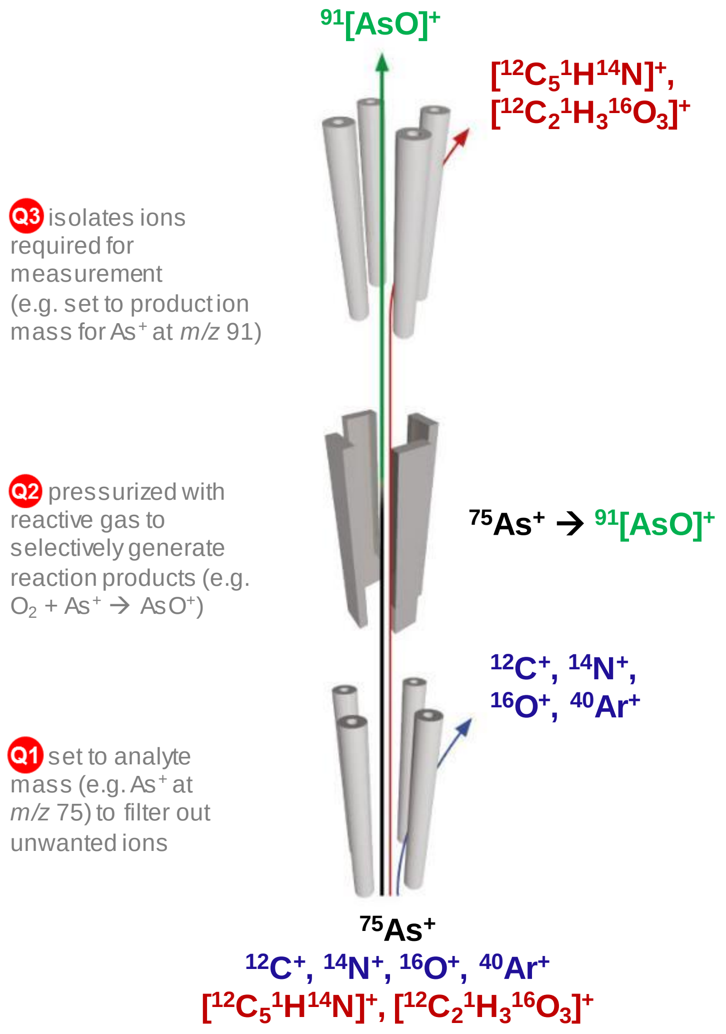
Hardware Setup for Ultratrace Elements in Photoresist Solvents

- Dedicated sample introduction system
  - 100 µL·min^{-1} self-aspirating PFA micro flow concentric nebulizer
  - Peltier cooled quartz spraychamber (at -10 ˚C).
  - High purity oxygen added to the aerosol to prevent carbon matrix build up on the interface
  - 1.0 mm diameter quartz injector to minimize carbon loading of the plasma
- Identical instrument parameters for both NMP and PGMEA
  - Improved sample throughput without any sacrifice in performance

TQ mass shift reaction



Results of Ultratrace Elements in Photoresist Solvents

Analyte	Analysis Mode	LoD ($ng \cdot L^{-1}$)		Spike Recovery (% of 100 $ng \cdot L^{-1}$)	
		PGMEA	NMP	PGMEA	NMP
^{7}Li	SQ-CP-NH_3	0.2	0.9	97%	98%
^{23}Na	SQ-CP-NH_3	1.3	10.2	98%	97%
^{24}Mg	SQ-CP-NH_3	0.4	5.0	97%	91%
^{27}Al	SQ-CP-NH_3	1.1	3.8	97%	100%
^{39}K	SQ-CP-NH_3	1.3	6.4	102%	106%
^{40}Ca	SQ-CP-NH_3	1.2	8.1	96%	94%
^{52}Cr	SQ-CP-NH_3	2.0	3.3	97%	92%
^{55}Mn	SQ-CP-NH_3	0.3	8.4	93%	99%
^{56}Fe	SQ-CP-NH_3	3.1	2.0	104%	103%
^{59}Co	SQ-CP-NH_3	0.4	0.7	95%	100%
^{63}Cu	SQ-CP-NH_3	1.2	3.4	98%	96%
^{51}V	SQ-KED	2.6	2.8	105%	96%
^{66}Zn	SQ-KED	14.3	18.8	94%	100%
^{98}Mo	SQ-KED	1.6	2.1	97%	111%
^{107}Ag	SQ-KED	1.5	1.8	97%	111%
^{111}Cd	SQ-KED	3.7	2.6	85%	107%
^{138}Ba	SQ-KED	0.1	0.0	94%	101%
^{48}Ti | $^{64}Ti.O$	TQ-O_2	0.8	1.4	97%	108%
^{75}As | $^{91}As.O$	TQ-O_2	1.9	3.7	88%	106%
^{80}Se | $^{96}Se.O$	TQ-O_2	2.0	2.4	99%	106%

- Detection limits calculated from 3σ of 10 replicate measurements of the calibration blank.
- Spike recoveries of between 88% and 111% were obtained for all twenty-one elements in NMP and PGMEA.
- Low detection limits achieved
  - ***Powerful interference removal capabilities from the combination of cold plasma and triple quadrupole technologies on the iCAP TQs ICP-MS.***

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Determination of Ultratrace Elements on Silicon Wafer Surfaces

- Maximum allowable trace metallic impurity levels in process materials are moving to lower levels.
- Most widely used semiconductor material is silicon wafer
  - > 99.9999999% Si (9N purity).
- Monitoring trace element contamination is a routine requirement in the semiconductor industry
  - Surface contamination sampled by vapor phase decomposition (VPD)
  - VPD samples contain high acid and silicon concentrations, low target analyte concentration
  - Sample matrix generates significant polyatomic interferences on target elements

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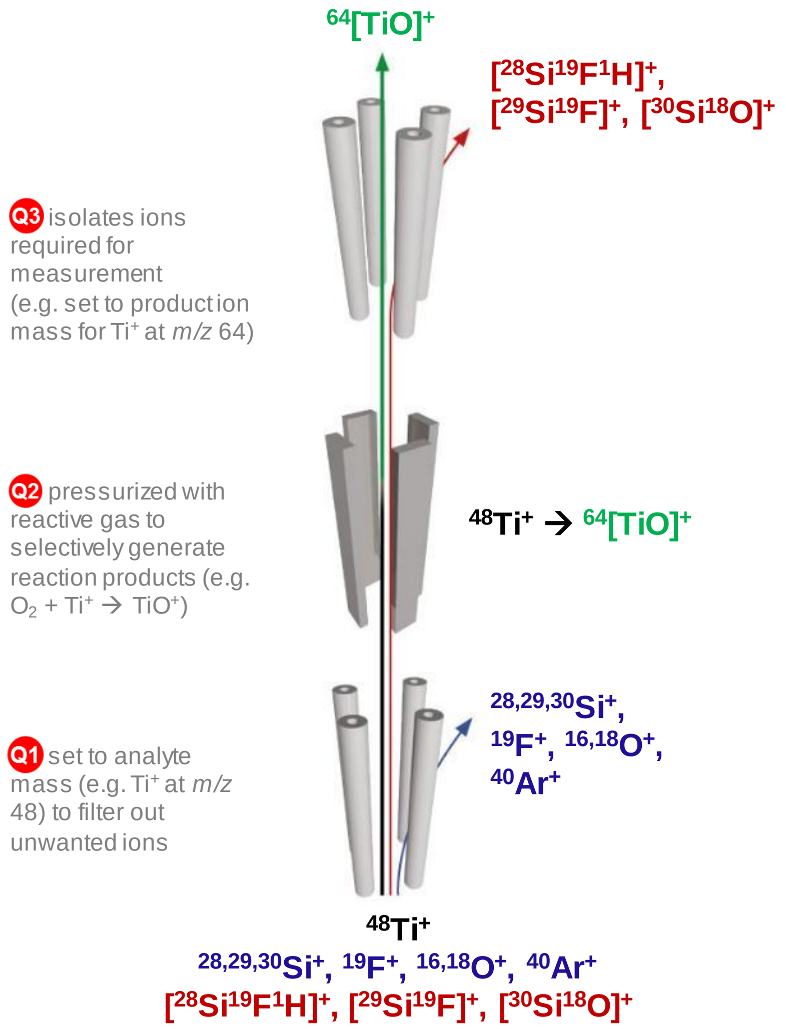
Hardware Setup for Ultratrace Elements on Silicon Wafer Surfaces

- Dedicated, HF resistant sample introduction system
  - 100 µL·min^{-1} self-aspirating PFA micro flow concentric nebulizer
  - PFA double pass spray chamber
  - 2.0 mm diameter quartz injector
- Routine, direct analysis of VPD samples is possible with these components

TQ mass shift reaction



Results of Ultratrace Elements on Silicon Wafer Surfaces

Analyte	Analysis Mode	LoD ($ng \cdot L^{-1}$)	Measurement Results ($ng \cdot L^{-1}$)	Spike Recovery (% of 50 $ng \cdot L^{-1}$)	Reproducibility (n=5) (%)
^{7}Li	SQ-CP-NH_3	0.1	0.02	98	95.2 ± 3.7
^{23}Na	SQ-CP-NH_3	0.6	2.3	100	100.2 ± 1.5
^{24}Mg	SQ-CP-NH_3	0.3	0.4	99	98.3 ± 2.2
^{27}Al	SQ-CP-NH_3	1.3	6	104	105.2 ± 3.1
^{39}K	SQ-CP-NH_3	0.6	1	98	101.0 ± 1.5
^{40}Ca	SQ-CP-NH_3	1	4.1	103	101.4 ± 4.1
^{48}Ti | ^{64}TiO	TQ-O_2	1.2	2.8	99	105.7 ± 1.5
^{51}V | ^{67}VO	TQ-O_2	0.2	0.8	98	103.1 ± 1.6
^{55}Mn	SQ-CP-NH_3	2.3	3.5	97	103.8 ± 1.9
^{56}Fe	SQ-CP-NH_3	1.6	3.4	98	102.2 ± 3.3
^{59}Co	SQ-CP-NH_3	0.9	2.2	97	99.0 ± 2.3
^{63}Cu	SQ-CP-NH_3	0.9	1.7	98	99.5 ± 1.2
^{66}Zn	SQ-KED	1.9	7.7	90	111.1 ± 3.4
^{71}Ga	SQ-KED	1.2	2.3	91	106.9 ± 4.4
^{74}Ge | ^{74}Ge	TQ-O_2	1	2.7	100	104.1 ± 1.2
^{75}As | ^{91}AsO	TQ-O_2	0.4	0.7	101	106.0 ± 2.5
^{88}Sr	SQ-KED	0.2	0.1	98	105.2 ± 2.8
^{90}Zr	SQ-KED	0.1	0.1	96	105.7 ± 2.6
^{98}Mo	SQ-KED	0.5	1.1	93	107.0 ± 3.8
^{107}Ag	SQ-KED	0.3	1	95	107.0 ± 4.0
^{111}Cd	SQ-KED	0.4	0.6	95	111.1 ± 4.5
^{118}Sn	SQ-KED	0.3	0.6	93	107.9 ± 4.0
^{121}Sb	SQ-KED	0.1	0.1	93	105.3 ± 2.0
^{138}Ba	SQ-KED	0.1	0.1	93	105.7 ± 4.5
^{184}W	SQ-KED	0.1	0.2	93	103.6 ± 3.8
^{208}Pb	SQ-KED	0.3	1.2	91	107.2 ± 4.1

- Detection limits calculated from 3σ of 10 replicate measurements of the calibration blank.
- Accurate spike recoveries from 90% to 104% were obtained for all 26 elements at 50 $ng \cdot L^{-1}$
- Excellent reproducibility and reliability was achieved, despite 200 $mg \cdot L^{-1}$ silicon matrix,
  - ***Powerful interference removal and robustness of the iCAP TQs ICP-MS in mixed hot and cold plasma analyses.***

Thermo Scientific iCAP TQs ICP-MS

- Accurate Results

  Rely on improved interference removal capabilities to get right results first time!

- Simple Method Development

  Let Reaction Finder in the Qtegra ISDS Software select the isotope, gas and scan settings for you!

  Run all elements using optimized settings in a single run!

- Productivity

  Use multiple analysis modes in one method for simplicity and productivity, without wasting sample or time!

Thermo Scientific Qtegra Intelligent Scientific Data Solution Software

Thermo Scientific iCAP TQs ICP-MS

All the Power, All the Performance None of the Complexity

- ✓ **Robust design for routine and research**
- ✓ **Advanced interference removal in challenging matrices**
- ✓ **Ultralow detection capabilities**
- ✓ **Unique ease of use**
- ✓ **Flexible automation options**
- ✓ **Ideal for advanced applications**

Join the Fun! *Cache a Chromeleon* Game

- Use your mobile device to complete challenges and earn a Charlie Chromeleon plush toy!
- If you are playing, you have earned points for attending this seminar. Be sure to scan the barcode on the desk outside the door.
- Ask booth staff for more details on how to play.

Thank You

Please join me in the
Trace Elemental Analysis
section of our booth where I'll
address additional comments and questions.

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