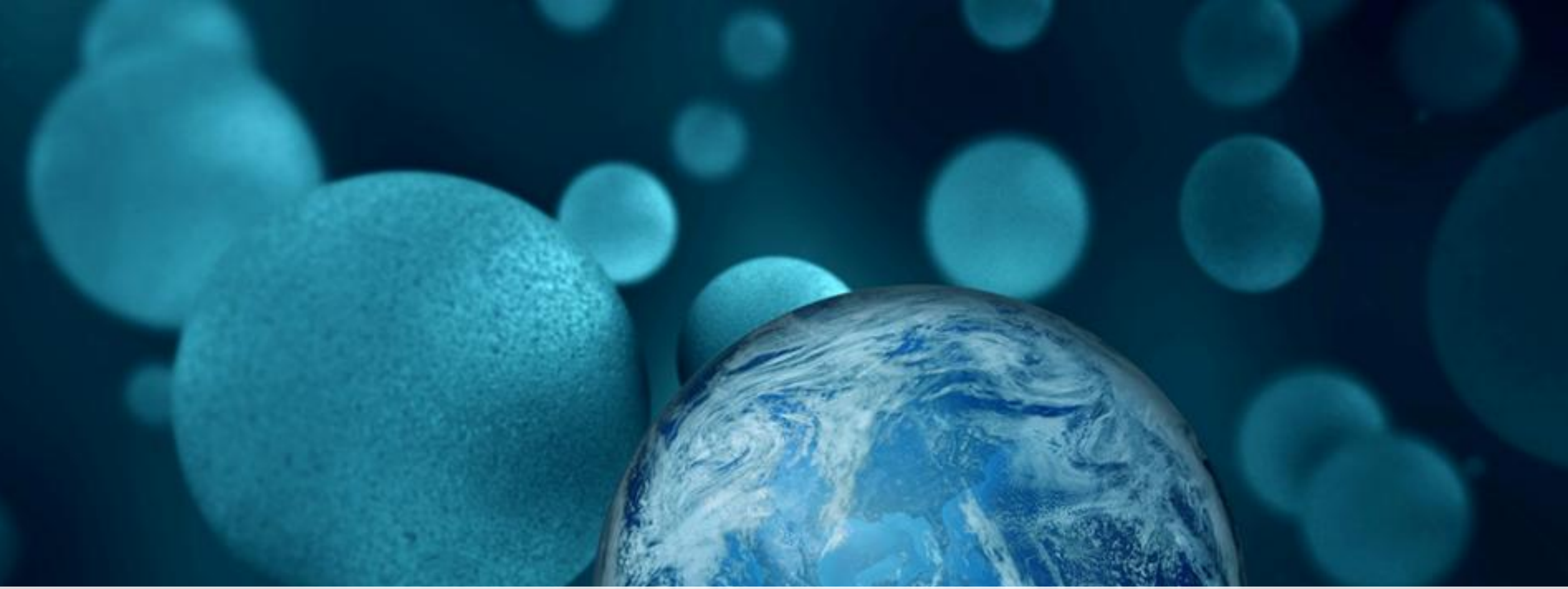


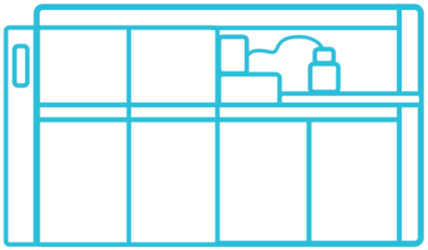
Thermo Fisher SCIENTIFIC

Discovering The Cutting Edge of Trace Elemental Analysis: Introducing the iCAP TQ ICP-MS

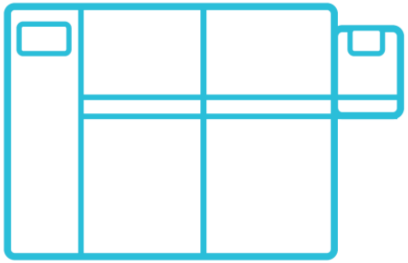
Elsamoul Hamdnalla
Thermo Fisher Scientific

ICP-MS Performance for All Applications

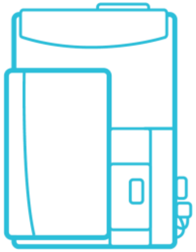
Technology for All Challenges



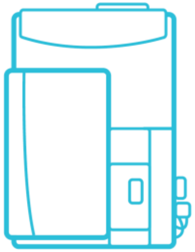
Thermo Scientific™ NEPTUNE Plus MC-ICP-MS
Multicollector ICP-MS



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

All the Power,
None of the Complexity

Triple quadrupole accuracy with single quadrupole ease of use

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

ThermoFisher SCIENTIFIC

Inside TQ Technology

- ✓ Quick-connect, self-aligning sample introduction system
- ✓ Drop-down door for easy interface access
- ✓ Range of interfaces provide full instrument flexibility, from maximum robustness to highest sensitivity
- ✓ Zero user maintenance beyond the interface
- ✓ Highest data accuracy in complex samples with the combined QCell and TQ functionality

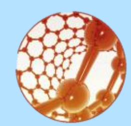
Applications we are going to focus on...

Environmental Analysis – As, Se

Clinical Research - Ti

Metallurgy

Advanced Applications



Speciation Analysis
Nanoparticles

Redefining Trace Element Analysis -Triple Quadrupole ICPMS

Meeting human health and environmental challenges

- Clinical Research and Toxicology
- Metallopharmaceuticals
- Environmental Analysis/Monitoring
- Food Safety

Advancing development in metals, materials and chemicals

- Material Analysis
- Nanoparticle Characterization
- Metallurgy
- Energy Production

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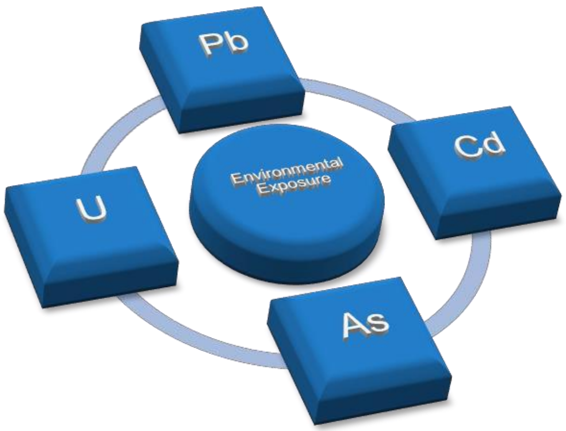
Determination of Trace Elements in Biological Samples

- Trace Elements play important roles for correct funtion of living beings

Intentional Exposure:
e.g. orthopedic surgery, drug treatment



Environmental Exposure:
e.g. occupational health, work safety

Matrix	Characteristics	Sample Preparation
Urine	**Easy and pain-free to obtain**	**Simple; Dilution**
	High salt content	
Blood	**Contains protein bound metals**	**Difficult; Microwave assisted digestion**
	Viscosity, Precipitation	
Serum	**Reveals metal distribution in the blood stream**	**Simple; Dilution**
	Salts, low molecular weight species	

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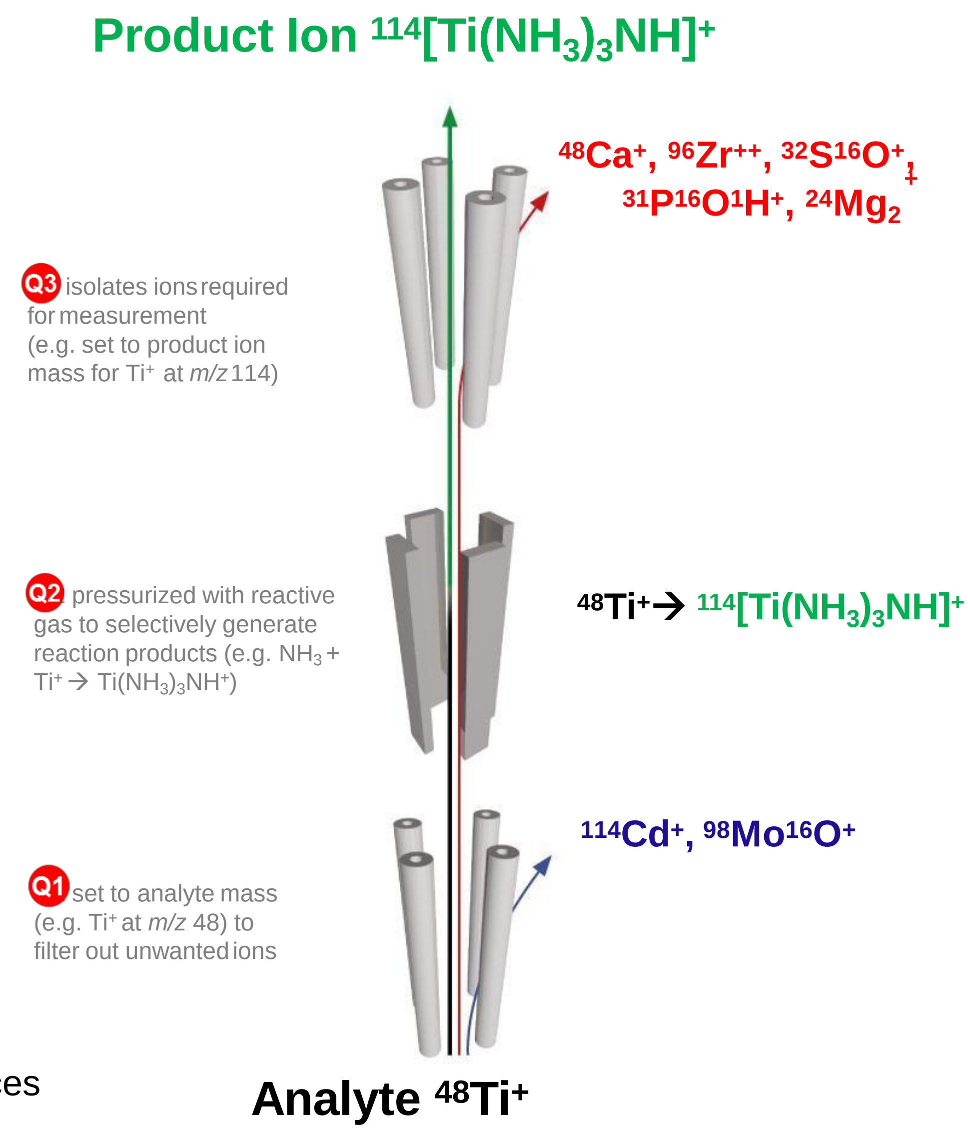
Problem: Analysis of Ti in Serum

- Ti may be released into the blood stream following degradation of orthopedic or dental implants
  - Low concentrations (less than 1 ng·mL^{-1})
  - Isobaric interference of main isotope ^{48}Ti through ^{48}Ca
  - PO^+, POH^+, CaH^+ interferences on other Ti isotopes



- Analytical methods need to be

→ ***Robust*** to cope with sample matrix

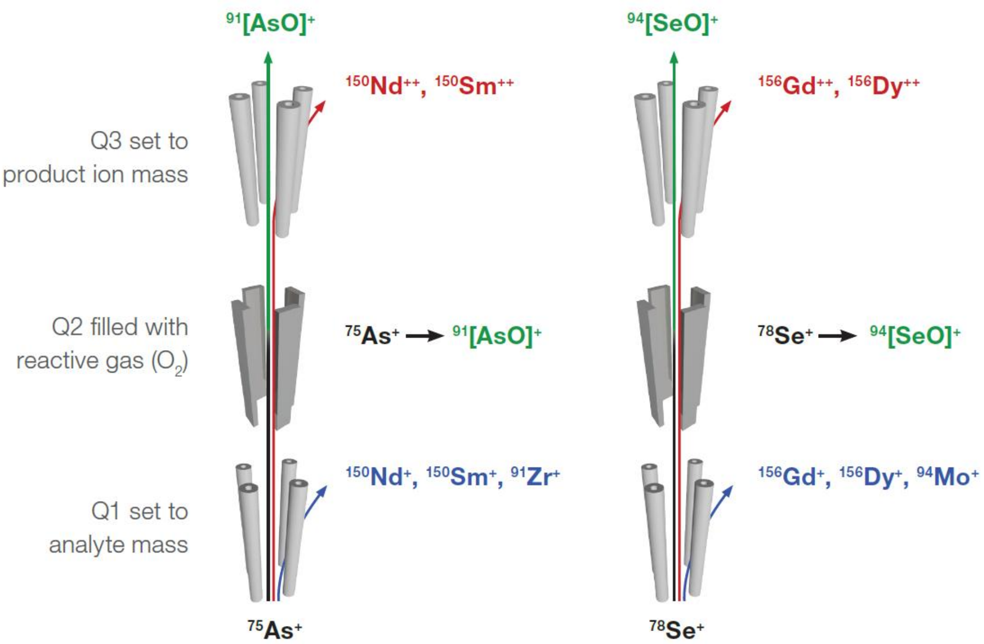
→ ***Sensitive*** to enable detection of low levels

→ ***Specific*** to address Ti accurately despite interferences

Solution – use the iCAP TQ

- Control ions entering the cell using Q1
- Use O_2 to efficiently convert As and Se to AsO and SeO in Q2 – the REE^{++} don‘t react
- Selectively detect AsO and SeO free from REE^{++} interference, using Q3

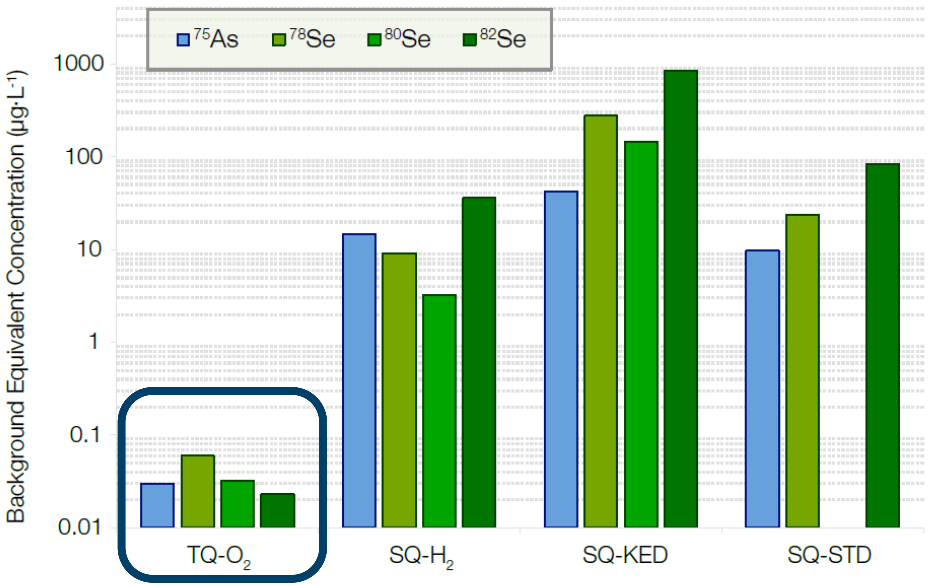
Type	^{75}As	Method to remove	^{78}Se	Method to remove
Polyatomic	$^{40}Ar^{35}Cl$	KED	$^{40}Ar^{38}Ar$	KED, H_2
	$^{40}Ca^{35}Cl$			
Isobaric	$^{150}Nd^{2+}$	O_2	$^{156}Gd^{2+}$	O_2
	$^{150}Sm^{2+}$		$^{156}Dy^{2+}$	



ICP-MS using triple quadrupole technology – Thermo Scientific iCAP TQ ICP-MS

As and Se with REE present - results in different modes

- Interference removal capability



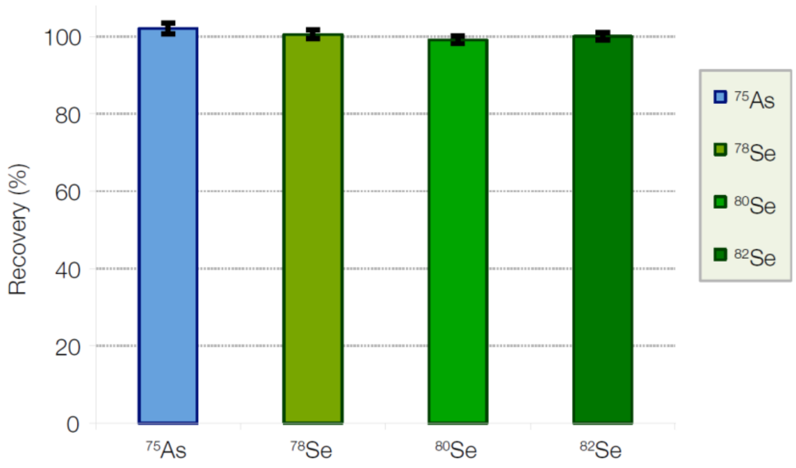
- 1ppm Dy, Gd, Nd, Sm and Tb added
- Increased BECs observed for all SQ-modes due to unresolved doubly charged REE interferences
- Hydrogen is suitable for removing Ar based polyatomics, but is not capable of fully removing REE^{2+} interferences
- TQ-O_2 mode shows dramatically lower BEC values for both As and Se

Proving the accuracy of the sample analysis

Sample analysis results

AGV-1	Content in original sample (µg·g⁻¹)	Certified content (µg·g⁻¹)
^{75}As	0.892	0.88
^{78}Se	< LOQ	-
Deep Sea Sediment		
^{75}As	1.303	-
^{78}Se	0.109	-

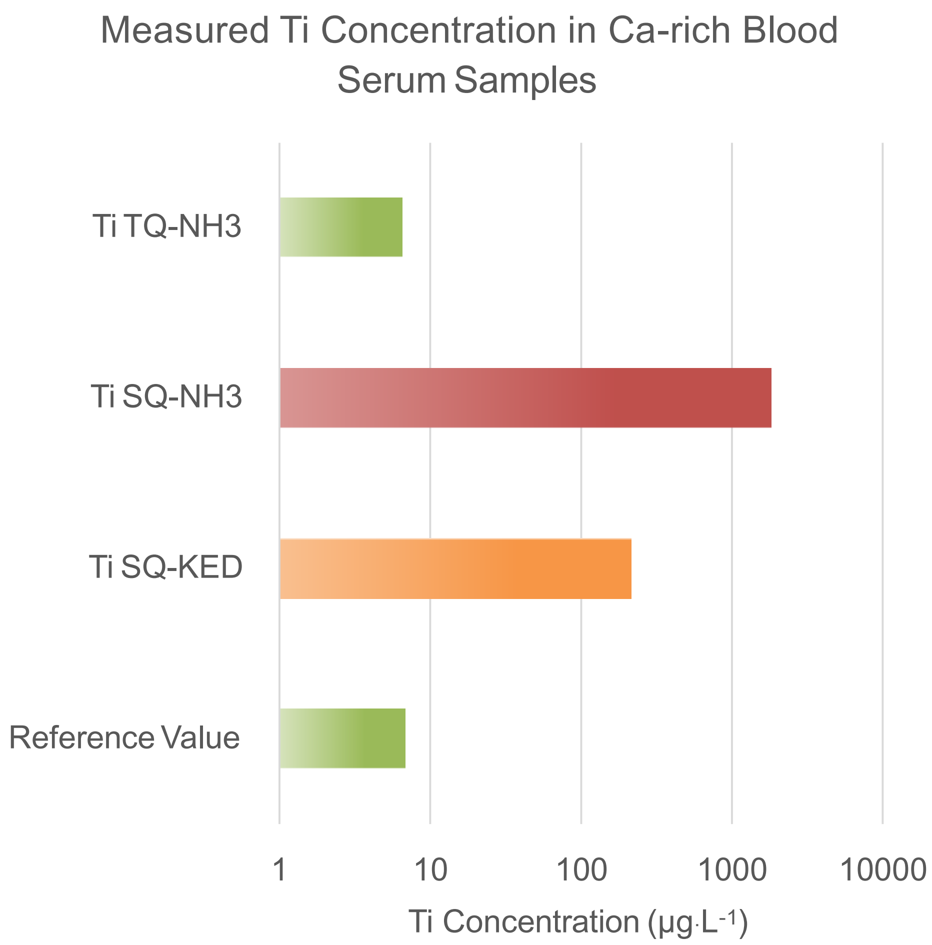
Spike recovery in REE matrix solution (1 ppb As and Se)



Spike recovery results in samples (1 ppb As and Se)

Analyte	AGV-1	Sediment
Arsenic	94.6 %	97.6 %
Selenium	93.4 %	97.6 %

Recovery of Ti in Serum using Different Measurement Modes



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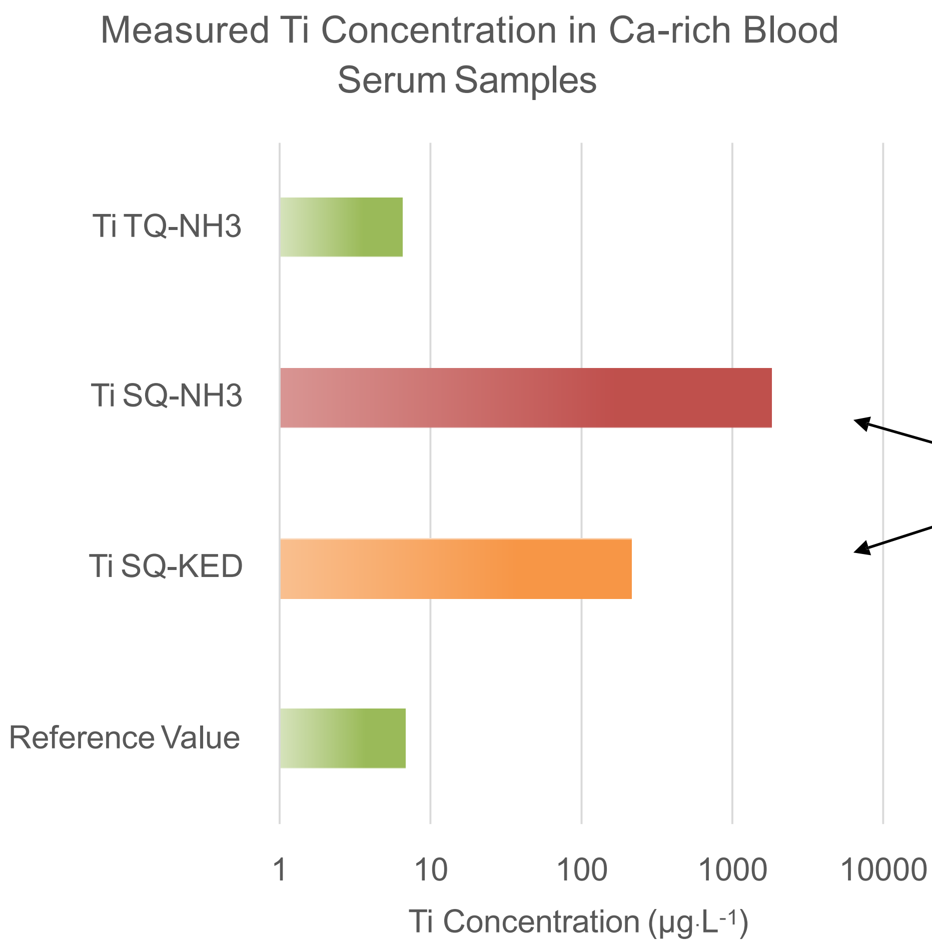
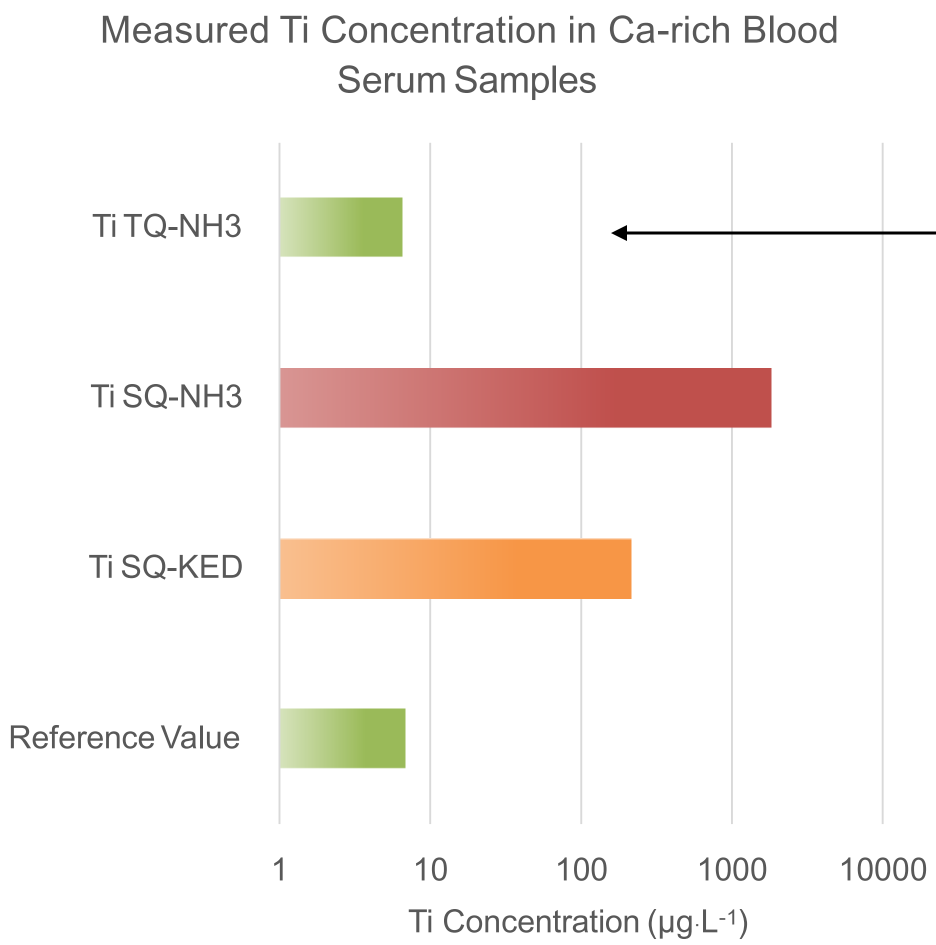
Recovery of Ti in Serum using Different Measurement Modes



Single Quadrupole ICP-MS
False positive results due to unresolved isobaric ^{48}Ca Interference!

Recovery of Ti in Serum using Different Measurement Modes



Triple Quadrupole ICP-MS
Only by using triple quad technology can accurate results for Ti be obtained!

Multielemental Analysis in Serum (and Urine)

	LOD	MDL	Serum L-1		Serum L-2		Urine
			Measured	Reference or reported value	Measured	Reference or reported value	Measured
Na*	0.0027	0.027	2743	2330-3504	3255	2820-4241	2977
Mg*	0.0001	0.0010	21.0	13.4-20.1	39.7	27.1-40.7	85.6
P*	0.0008	0.08	52.3	43.3-65.1	120	88-132	710
S*	0.145	1.3800	1100	1008	1495	1335	476
K*	0.0021	0.02	150	101-153	260	176-265	1946
Ca*	0.002	0.0200	90.1	69-104	124	95-143	99.8
Fe*	0.00002	0.00023	1.64	1.17-1.77	2.18	1.72-2.58	0.005
Li	1.13	11.2920	5778	4202-6320	10806	7739-11639	22.4
B	0.67	6.746	70.1	79.4	87	82.1	1548
Al	0.20	1.9670	54.2	25.2-75.7	122	96-144	13.7
V	0.002	0.022	1.04	1.10	1.26	1.10	0.229
Cr	0.008	0.0800	1.70	1.30-3.05	5.20	4.00-7.50	0.838
Mn	0.008	0.084	10.7	7.9-11.9	14.2	11.6-17.4	0.914
Co	0.0001	0.0010	1.38	0.67-1.57	2.16	2.13-3.97	0.027
Ni	0.006	0.055	6.26	3.38-7.9	9.41	7.9-11.9	1.45
Zn	0.051	0.5130	1052	844-1269	1527	1404-1831	359
As	0.002	0.018	0.383	0.400	0.374	0.380	1.31
Se	0.010	0.1000	80.8	51-120	124	95-176	7.31
Rb	0.004	0.035	4.20	4.40	8.70	8.70	812
Sr	0.006	0.0570	95.7	95.0	106	110	89.2
Mo	0.005	0.048	0.710	0.760	1.20	1.21	7.62
Cd	0.001	0.0100	0.130	0.130	0.140	0.140	0.229
Ti	0.002	0.02	6.64	6.80	6.38	6.80	0.151
Sb	0.006	0.0600	11.6	10.4	16.1	15.0	0.040
I	0.022	0.219	75.5	71.8	69.9	60.9	82.8
Ba	0.003	0.0300	172	190	133	139	2.09
Pb	0.0007	0.007	0.370	0.400	0.666	0.660	0.446
U	0.0001	0.0010	0.288	0.302	0.357	0.359	0.020

mg·L^{-1} (Na* – Fe*)

µg·L^{-1} (Li – U)

Elements accurately measured from 0.005 – 10,806 ppb

Determination of Trace Elements in Metals and Alloys

Nickel Alloys are used for industrial applications

e.g. turbine blades

Selenium content is critical, may lead to weakness

Solid Sampling

LA-ICP-MS

GD-MS

Liquid Sampling

ICP-MS

Analysis of Se impurities in a Ni matrix

- Se analysis using ICP-MS
  - Elevated 1st ionization potential → low ion yield
  - Main isotopes affected through Ar based polyatomics
  - Additional Ni interferences on all Se isotopes
  - Potential for additional interferences in case Br is present

Isotope m/z	Abundance (%)
74	0.90
76	9.00
77	7.60
78	23.60
80	49.70
82	9.20

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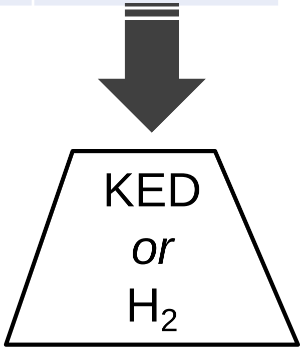
Isotope m/z	Abundance (%)	Normal Matrix Interference
74	0.90	
76	9.00	$^{40}Ar^{36}Ar^+$
77	7.60	$^{40}Ar^{37}Cl^+$
78	23.60	$^{40}Ar^{38}Ar^+$, $^{78}Kr^+$
80	49.70	$^{40}Ar^{40}Ar^+$, $^{80}Kr^+$
82	9.20	$^{82}Kr^+$



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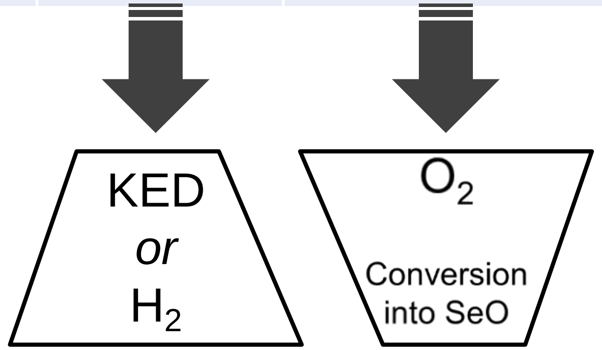
  - Potential for additional interferences in case Br is present

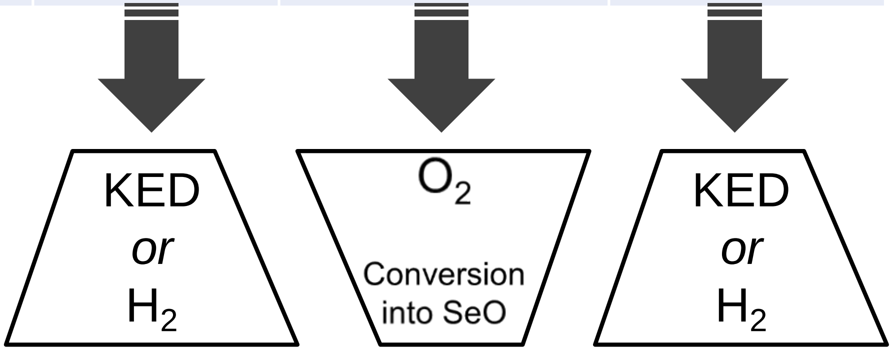
Isotope m/z	Abundance (%)	Normal Matrix Interference	Additonal Ni Matrix Interference
74	0.90		$^{58}Ni^{16}O^+$
76	9.00	$^{40}Ar^{36}Ar^+$	$^{60}Ni^{16}O^+$
77	7.60	$^{40}Ar^{37}Cl^+$	$^{60}Ni^{16}O^{1}H^+$
78	23.60	$^{40}Ar^{38}Ar^+$, $^{78}Kr^+$	$^{62}Ni^{16}O^+$
80	49.70	$^{40}Ar^{40}Ar^+$, $^{80}Kr^+$	$^{64}Ni^{16}O^+$
82	9.20	$^{82}Kr^+$	$^{64}Ni^{18}O^+$, $^{64}Ni^{16}O^{1}H^+$



Analysis of Se impurities in a Ni matrix

- Se analysis using ICP-MS
  - Elevated 1st ionization potential → low ion yield
  - Main isotopes affected through Ar based polyatomics
  - Additional Ni interferences on all Se isotopes
  - Potential for additional interferences in case Br is present

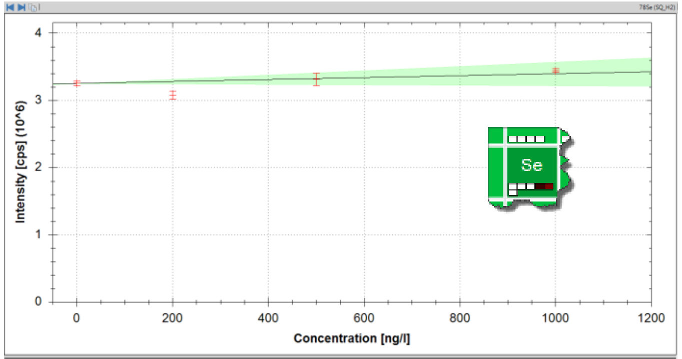
Isotope m/z	Abundance (%)	Normal Matrix Interference	Additonal Ni Matrix Interference	Additonal Bromine Interference
74	0.90		$^{58}Ni^{16}O^+$	
76	9.00	$^{40}Ar^{36}Ar^+$	$^{60}Ni^{16}O^+$	
77	7.60	$^{40}Ar^{37}Cl^+$	$^{60}Ni^{16}O^{1}H^+$	
78	23.60	$^{40}Ar^{38}Ar^+$, $^{78}Kr^+$	$^{62}Ni^{16}O^+$	
80	49.70	$^{40}Ar^{40}Ar^+$, $^{80}Kr^+$	$^{64}Ni^{16}O^+$	$^{79}Br^{1}H^+$
82	9.20	$^{82}Kr^+$	$^{64}Ni^{18}O^+$, $^{64}Ni^{16}O^{1}H^+$	$^{81}Br^{1}H^+$



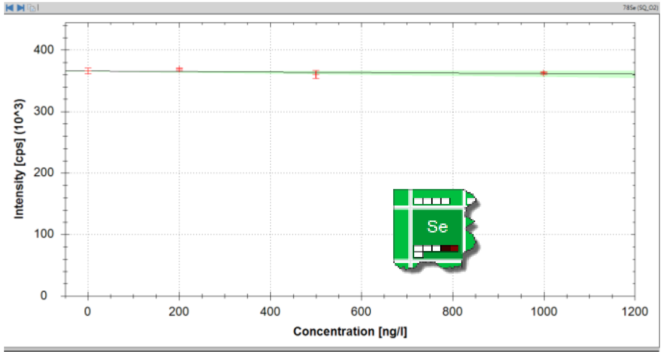
Interference Removal using SQ-ICP-MS

Sample: Se in 100 ppm Ni

H_2



f(x) = b*x + a				
Parameter	Value	Confidence Delta at 90%	StdErr	Rel. StdErr [%]
b	144.339	+/-174.393	106.486	73.775
a	3,250,861.921	+/-0.000	0.000	0.000
R^2	0.367			
BEC	22,522.419 ng/l			
IDL(LOD)	666.626 ng/l			



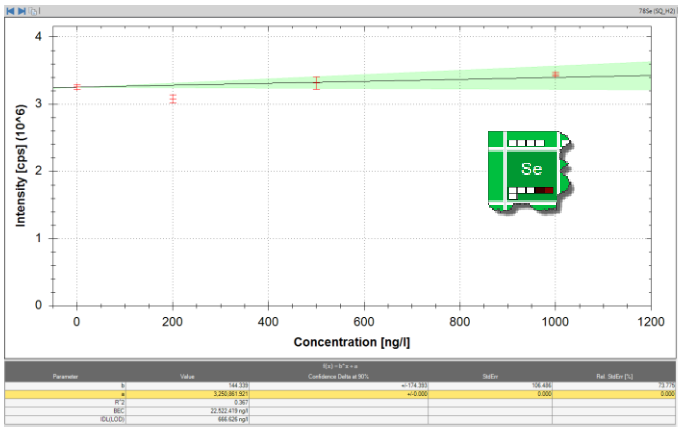
f(x) = b*x + a				
Parameter	Value	Confidence Delta at 90%	StdErr	Rel. StdErr [%]
b	-4.158	+/-4.486	2.739	65.885
a	365,710.443	+/-0.000	0.000	0.000
R^2	0.326			
BEC	-87,955.992 ng/l			
IDL(LOD)	N/A			

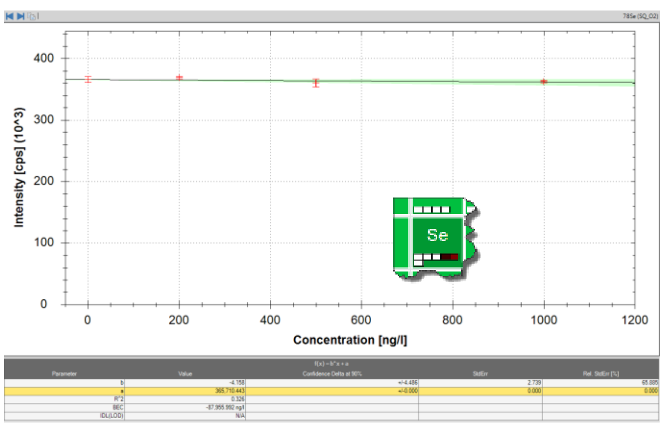
O_2

- Both reaction gases are not suitable to fully remove the interferences!

Sample: Se in 100 ppm Ni

H_2





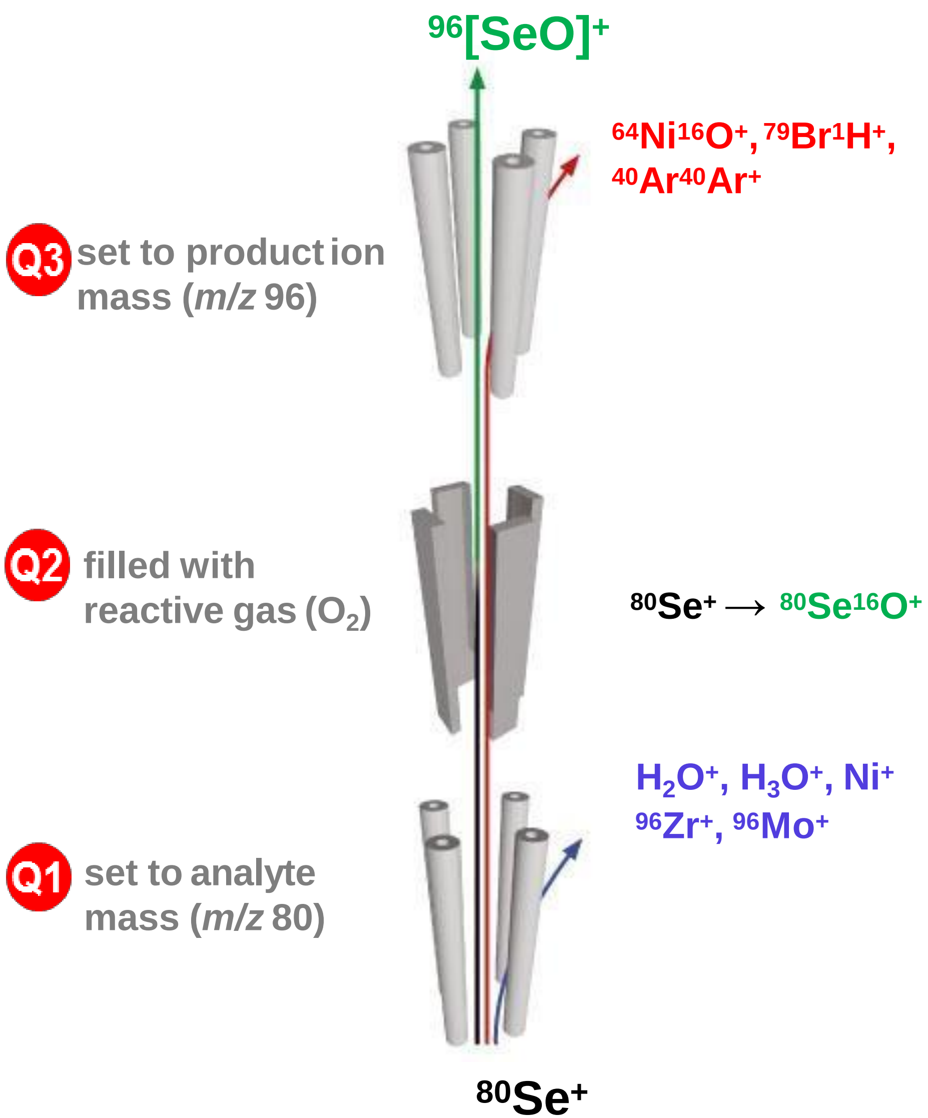
O_2

- Both reaction gases are not suitable to fully remove the interferences!
- As all primary ions enter the CRC, new interferences including water adducts are observed

Ion Mass	Identifier	Interference
92	$^{76}Se^{16}O^+$	$^{58}Ni^{16}O\ H_2O^+$
93	$^{77}Se^{16}O^+$	$^{58}Ni^{16}O\ H_3O^+$
94	$^{78}Se^{16}O^+$	$^{60}Ni^{16}O\ H_2O^+$, $^{58}Ni^{18}O\ H_2O^+$
96	$^{80}Se^{16}O^+$	$^{62}Ni^{16}O\ H_2O^+$, $^{60}Ni^{18}O\ H_2O^+$
98	$^{82}Se^{16}O^+$	$^{64}Ni^{16}O\ H_2O^+$, $^{62}Ni^{18}O\ H_2O^+$

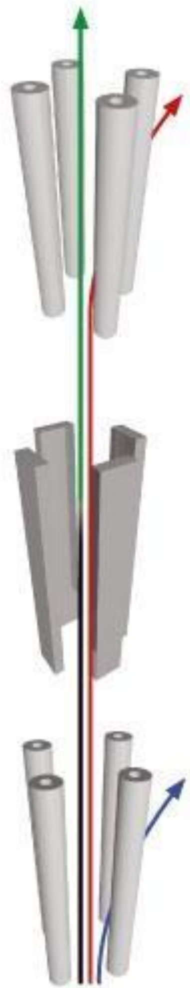
Need to control ions entering the CRC system !

Interference Removal using TQ-ICP-MS



Interference Removal using TQ-ICP-MS

$^{96}[SeO]^+$



$^{64}Ni^{16}O^+$, $^{79}Br^{1}H^+$, $^{40}Ar^{40}Ar^+$

Q3 set to product ion mass (*m/z* 96)

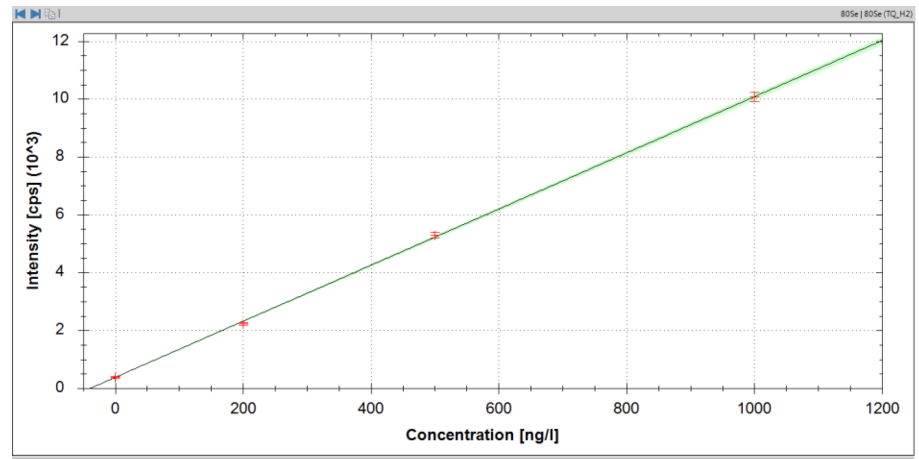
Q2 filled with reactive gas (O_2)

$^{80}Se^+ \longrightarrow {}^{80}Se^{16}O^+$

H_2O^+, H_3O^+, Ni^+, $^{96}Zr^+$, $^{96}Mo^+$

Q1 set to analyte mass (*m/z* 80)

$^{80}Se^+$



f(x) = b*x + a

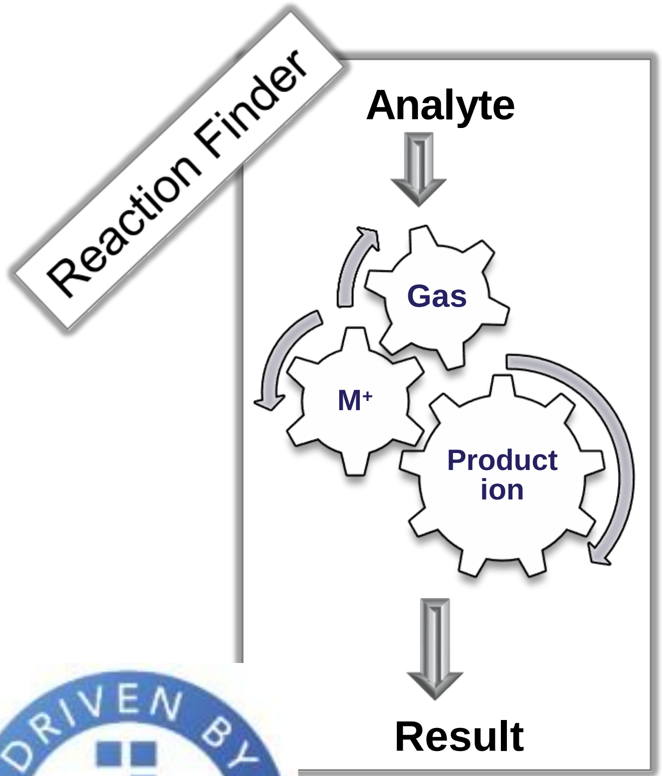
Parameter	Value	Confidence Delta at 90%	StdErr	Rel. StdErr [%]
b	9.701	+/-0.097	0.059	0.608
a	376.906	+/-0.000	0.000	0.000
R^2	1.000			
BEC	38.854 ng/l			
IDL(LOD)	10.548 ng/l			

Mode/ Isotope	Sensitivity [cps·L·µg⁻¹]	BEC [ng·L⁻¹]	IDL [ng·L⁻¹]
TQ-H_2			
^{78}Se	4,500	46.5	12.9
^{80}Se	9,700	38.9	10.5
TQ-O_2			
^{78}Se	1,000	47.8	18.8
^{80}Se	2,200	13.2	5.10

Accurate Multielemental Analysis with TQ-ICP-MS

- In Biological Samples
  - High detection sensitivity allows detection of low Titanium concentrations in complex biological samples
  - Excellent agreement with certified value obtained for Titanium in serum
- In Metals and Alloys
  - Q1 separation of lower mass ions enables effective and complete removal of all interferences on Selenium in Nickel alloys
  - Only triple quadrupole ICP-MS is capable of fully resolving all spectral interferences
- Full flexibility and usability of both single and triple quadrupole modes
  - Full multielemental analysis with dedicated interference removal for difficult analytes and comprehensive He KED in one run

Redefining TQ-ICP-MS with Unique Ease of Use



DRIVEN BY QTEGRA ISDS

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Redefining TQ-ICP-MS with Unique Ease of Use

Without Reaction Finder

- Select — Select the Analytes to be measured
- Select — For each analyte, select the isotopes to be measured
- Select — Select the internal standard element
- Select — Select the Q1 Analyte
- Select — Select the CRC gas (None, He, H_2, O_2, NH_3)
- Select — Select the mode (KED, Single Quad Mode, Triple Quad Mode)
- Select — Select the Q3 Mass (On-mass/mass shift product ion)
- Decide — Are the suggested settings ok? If not, update them
- Analyze — Enter sample names and positions or import from LIMS and start the LabBook

With Reaction Finder

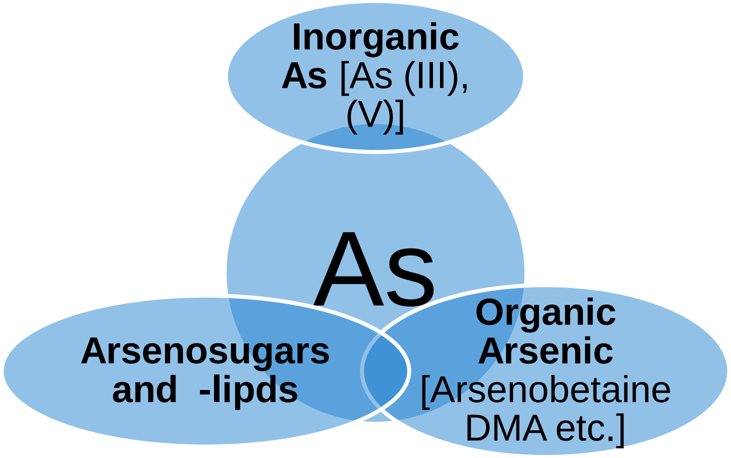
- Select — Select the Analytes to be measured
- Select — Select the internal standard element
- Decide — Are the suggested settings ok? If not, update them
- Analyze — Enter sample names and positions or import from LIMS and start the LabBook

< 20 min to set up method

Example Application: As and Se in Environmental Samples

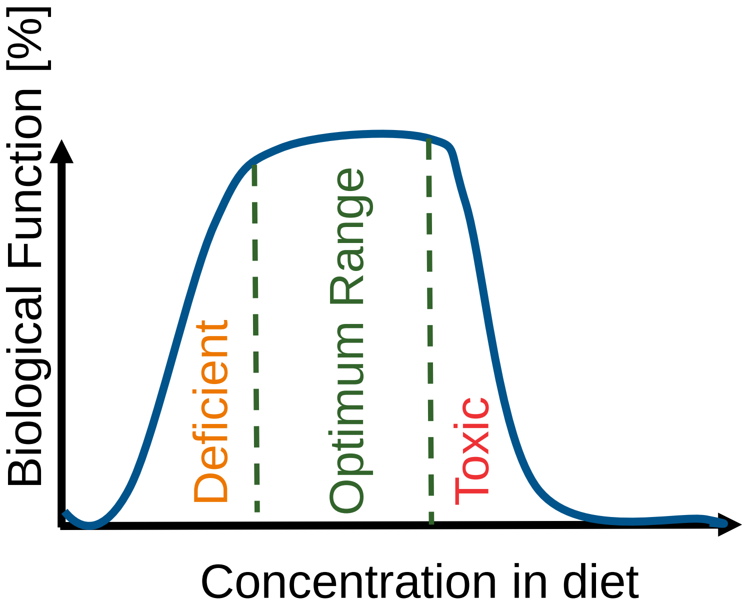
The role of As and Se in the environment

- Arsenic: A potential hazard in the food chain



- Many different chemical forms (species) are known that strongly differ in toxicity and bioavailability
- Plants such as rice are well known for high accumulation of As from soils

- Selenium: An essential nutrient

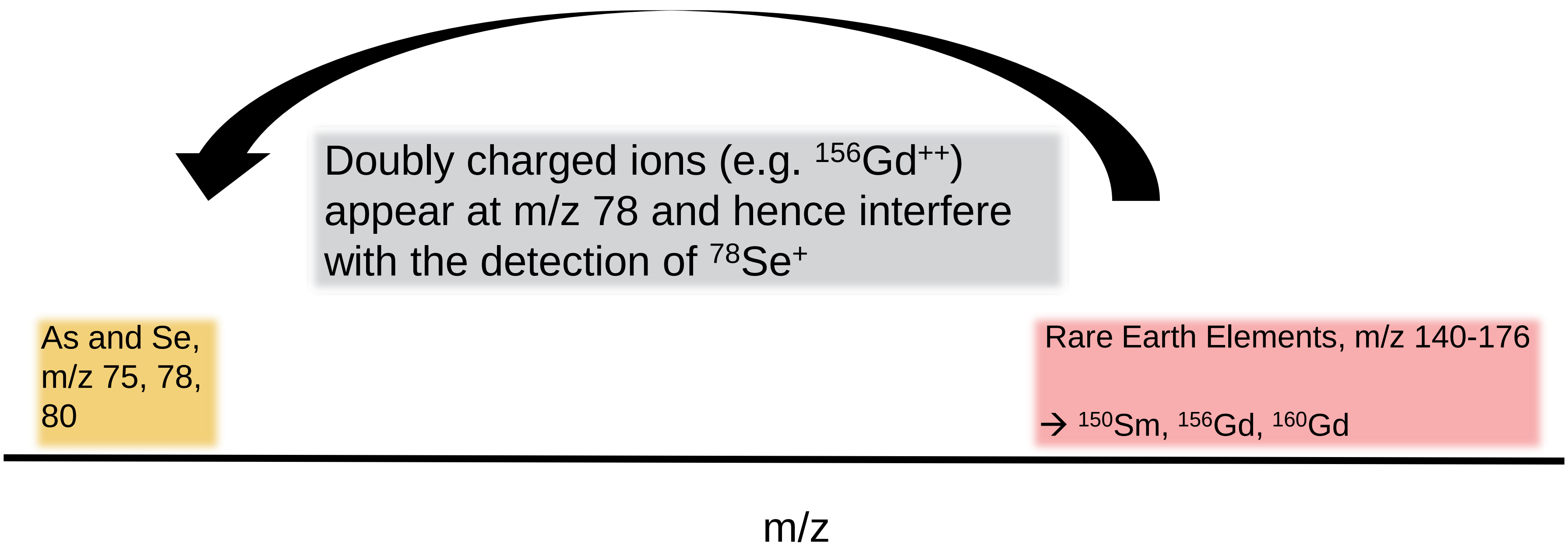


- Knowledge of Se content in soil may prevent Se deficiency in both human and animal populations

Example Application – As and Se in Environmental Samples

Interferences on As and Se: Ar_2, ArCl – easy to remove using He KED but if REE are present....

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



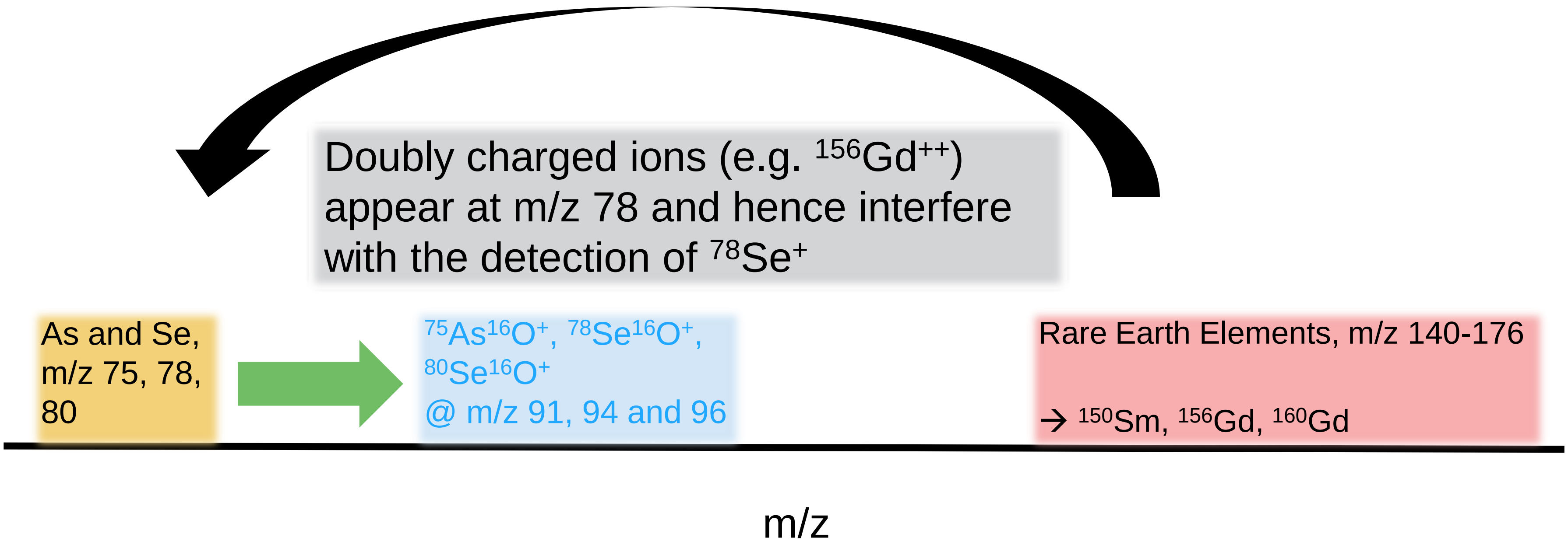
Example Application – As and Se in Environmental Samples

Single Quad ICP-MS: KED

Typically enhances M^{2+} Interferences

Solution:

Mass shift As and Se using O_2



Example Application – As and Se in Environmental Samples

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

Doubly charged ions (e.g. $^{156}Gd^{++}$) appear at m/z 78 and hence interfere with the detection of $^{78}Se^{+}$

As and Se, m/z 75, 78, 80

!

Rare Earth Elements, m/z 140-176

→ ^{150}Sm, ^{156}Gd, ^{160}Gd

m/z

Recovery of Ti in Serum using Different Modes

Single Quadrupole ICP-MS

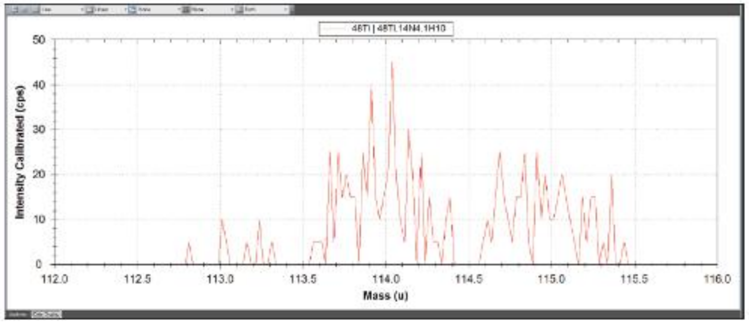
Triple Quadrupole ICP-MS

	Ti SQ-KED, µg·L^{-1}	Ti SQ-NH_3, µg·L^{-1}	Ti TQ-NH_3, µg·L^{-1}	Ti Reported Value, µg·L^{-1}
Serum L-1	167	1800	6.64	6.8
Serum L-2	262	1850	6.38	6.8

False positive results due to unresolved isobaric ^{48}Ca Interference

Only by using triple quad technology can accurate results for Ti be obtained!

Background signal on $^{48}Ti^{14}N_4H_{10}$ for a solution containing 10 mg·L^{-1} of Cd



- Excellent agreement with certified value obtained for Ti
- Attainable detection sensitivity allowed to detect low concentrations of Ti in the prepared sample solution

This would be a problem using single quadrupole instruments with NH_3!

Control Se Impurities in a Nickel Matrix

- Analysis of Se impurities is crucial e.g. for its use in turbine blades
  - Low concentrations
  - Polyatomic interferences

Thermo Scientific™ iCAP™ RQ ICP-MS

Thermo Scientific™ iCAP™ TQ ICP-MS

- Analytical methods need to be

→ ***Robust*** to cope with sample matrix

→ ***Sensitive*** to enable detection of low levels

→ ***Specific*** to address Se accurately despite interferences

Single quad or triple quad application?

Advanced Applications using the iCAP TQ ICP-MS

- Speciation Analysis

- ChromControl plug-in for hardware control
- Hyphenation of Gas Chromatography (GC) using GCI-100

Analyte	Advantage when using iCAP TQ	Application
S, P	Improved Interference Removal, lower background	Speciation of Biomolecules like proteins, peptides or DNA
As	Higher Sensitivity when analysing $^{75}As^{16}O$ instead of ^{75}As [KED]	All sample matrices: Food, Water, Urine etc.
Se	Higher Sensitivity when analysing SeO instead of Se [KED]; Reduced background on ^{80}Se	All sample matrices

Advanced Applications using the iCAP TQ ICP-MS

- Nanoparticle Analysis
- npQuant plug-in for Qtegra ISDS

Analyte	Advantage when using iCAP TQ	Application
Au, Ag	No apparent advantage → High Detection Sensitivity	Method Development, Validation
Ti	Increased specificity and lower background	Environmental samples, Cosmetic products
Others	Improved interference removal is advantageous to reduce background and address particles more efficiently	

Thermo Scientific iCAP TQ ICP-MS Delivering Quality ICP-MS Results

- Accurate trace elemental quantification enabled through improved interference removal
  - Simple method development – let Reaction Finder set up your method
  - Higher productivity – Get the right result first time
  - Lower LODs for challenging applications

- Reliable and consistent operation built on successful Platform
  - More uptime – less cleaning and longer periods of uninterrupted analysis
  - Less downtime for maintenance – easiest access to interface

Thermo Scientific iCAP TQ ICP-MS Delivering Quality ICP-MS Results

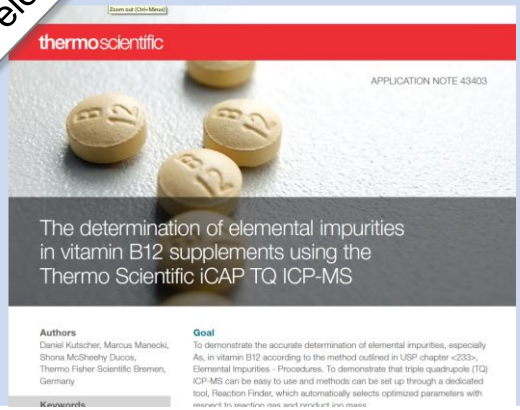
- Automated solutions, with integrated software features & control
  - Accelerate the TEQ workflow with intelligent dilution
  - Unattended operation - leave personnel free to do more important tasks in the lab

- Qtegra ISDS software providing intuitive workflows to streamline efficiency
  - Common platform to iCAP 7000 Series ICP-OES for reduced training and operator flexibility
  - Plug-in based platform enables easy implementation of advanced applications

iCAP TQ ICP-MS application and technical notes

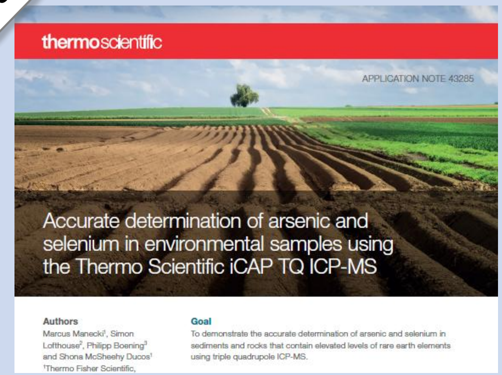
Pharma

Multi-elemental



Environment

Targeted

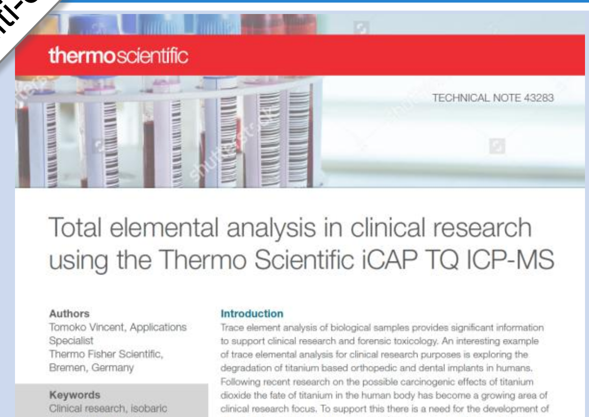


Metallurgy

Targeted

Clinical research

Multi-elemental



Proof data for you to use

ThermoFisher SCIENTIFIC