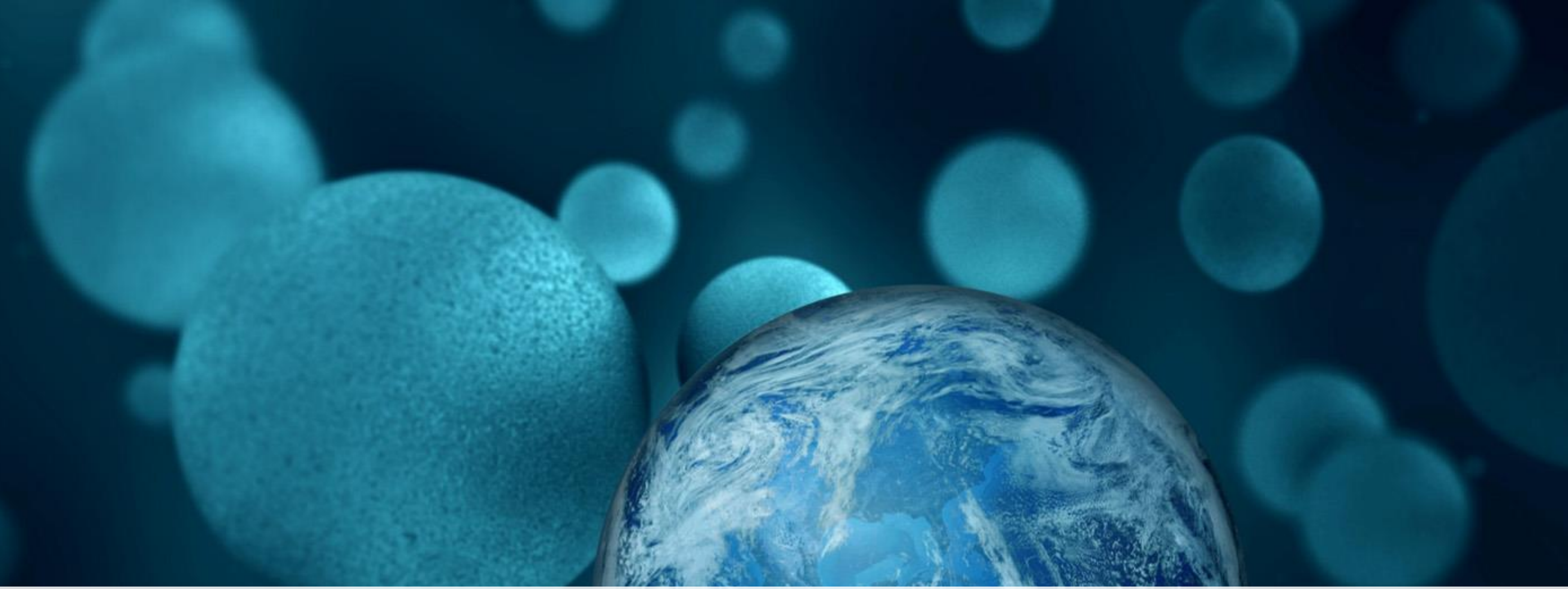


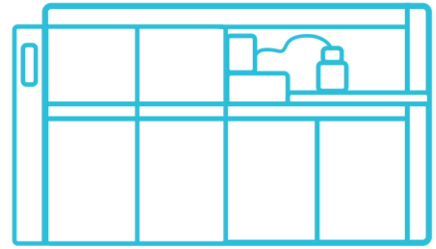
Thermo Fisher SCIENTIFIC

Latest developments in elemental analysis - Introducing the Thermo Scientific iCAP TQ ICP-MS

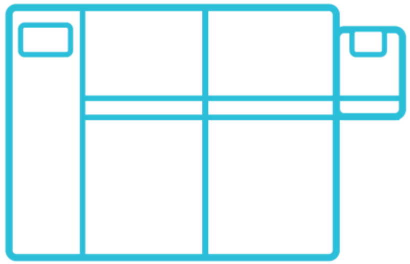
Simon Lofthouse, Sales Support Expert, TEA, EMEA
(simon.lofthouse@thermofisher.com)

Introducing our ICP-MS portfolio

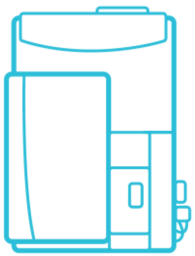
Technology for all challenges



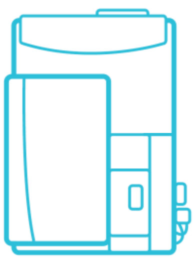
Thermo Scientific™ NEPTUNE Plus™ MC-ICP-MS
Multicollector ICP-MS



Thermo Scientific™ ELEMENT2/XR™ HR-ICP-MS
High Resolution ICP-MS



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

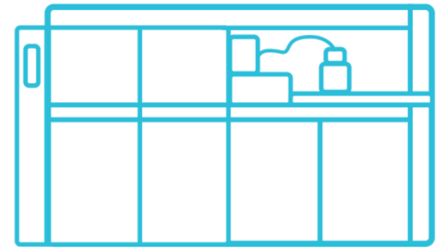


Thermo Scientific™ iCAP™ RQ ICP-MS
Single quadrupole ICP-MS

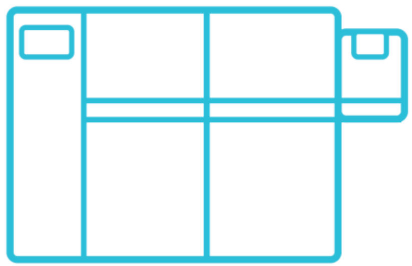
ThermoFisher SCIENTIFIC

Introducing our ICP-MS portfolio

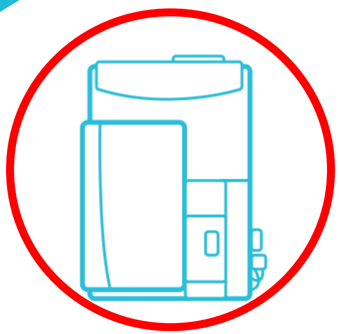
Technology for all challenges



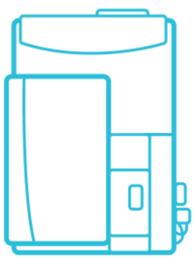
Thermo Scientific™ NEPTUNE Plus™ MC-ICP-MS
Multicollector ICP-MS



Thermo Scientific™ ELEMENT2/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

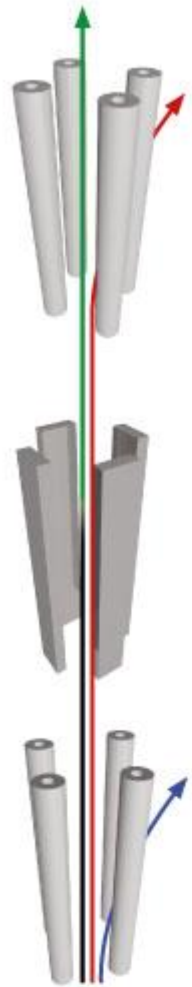
- ✓ **Advanced interference removal**
- ✓ **Robust design for routine analysis**
- ✓ **Integrated automation options**
- ✓ **Flexible for advanced applications**
- ✓ **Unique ease of use – Reaction Finder**

Triple quadrupole accuracy with single quadrupole ease of use

iCAP TQ ICP-MS: How it works - on mass reaction mode

$^{111}Cd^{+}$

ZrO_2, ZrO_2H, etc.



Q3 set to analyte mass (m/z 111)

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

Q2 filled with reactive gas (O_2)

$^{94}Zr^{16}O^{1}H^{+}$ **forms higher oxide products such as** ZrO_2, ZrO_2H **etc...**

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

Q1 set to analyte mass (*m/z* 111)

$^{91}Zr^{+}$

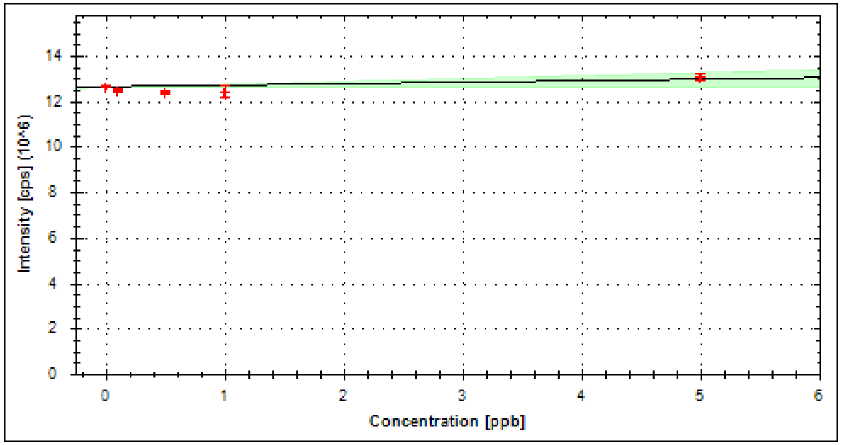
Q1 rejects unwanted ions and preselects the analyte. This first stage of mass filtration rejects precursors and ions with the same m/z ratio as the product ion.

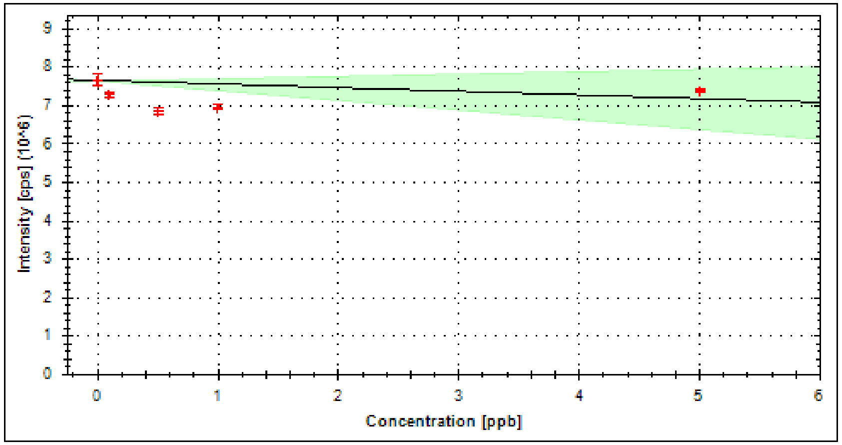
$^{111}Cd^{+}$

Yb in Gd matrix

- Calibration 0-5ppb Yb in 10ppm Gd – no gas
- Calibration 0-5ppb Yb in 10ppm Gd – KED

- NH_3 reacts with many of the polyatomic ions that interfere with the REE however NH_3 also reacts quickly with some REE.
- Pr, Eu, Dy, Ho, Er, Tm and Yb are less reactive with NH_3





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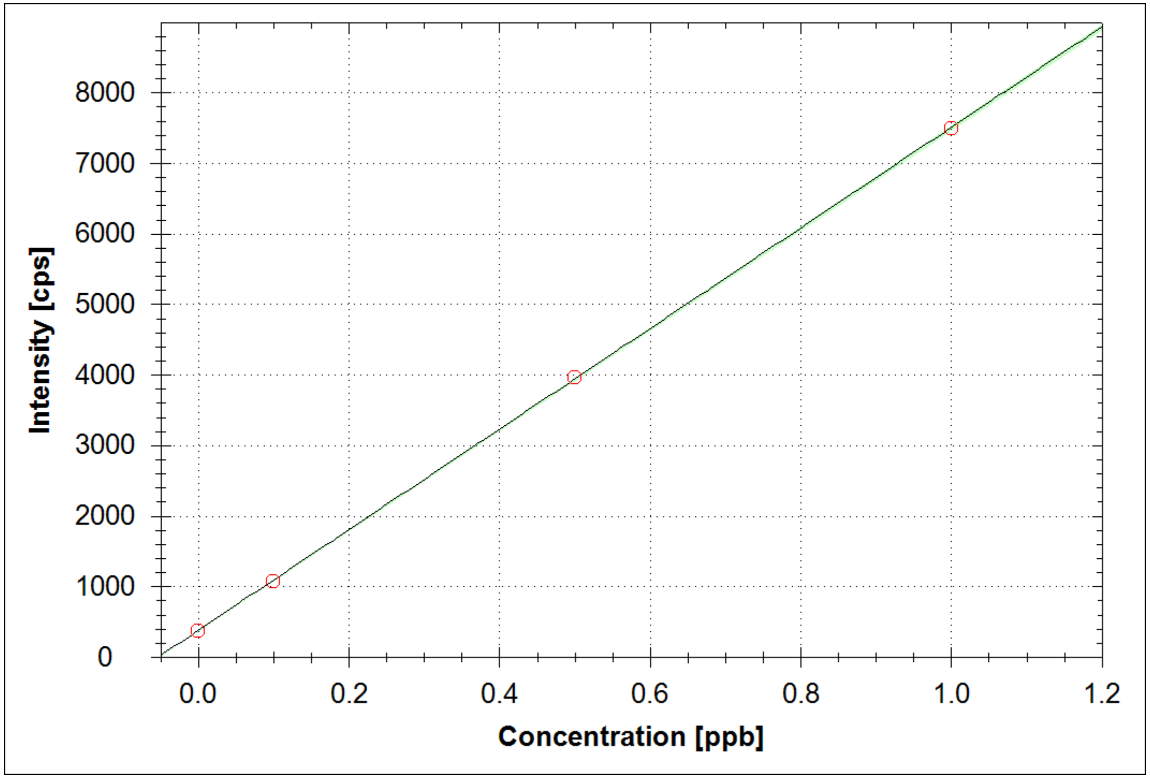
Yb measurement in 10pm Gd – TQ NH_3



- Sensitivity – 7100 cts/ppb
- BEC – 0.05 ppb
- IDL – 0.0001ppb

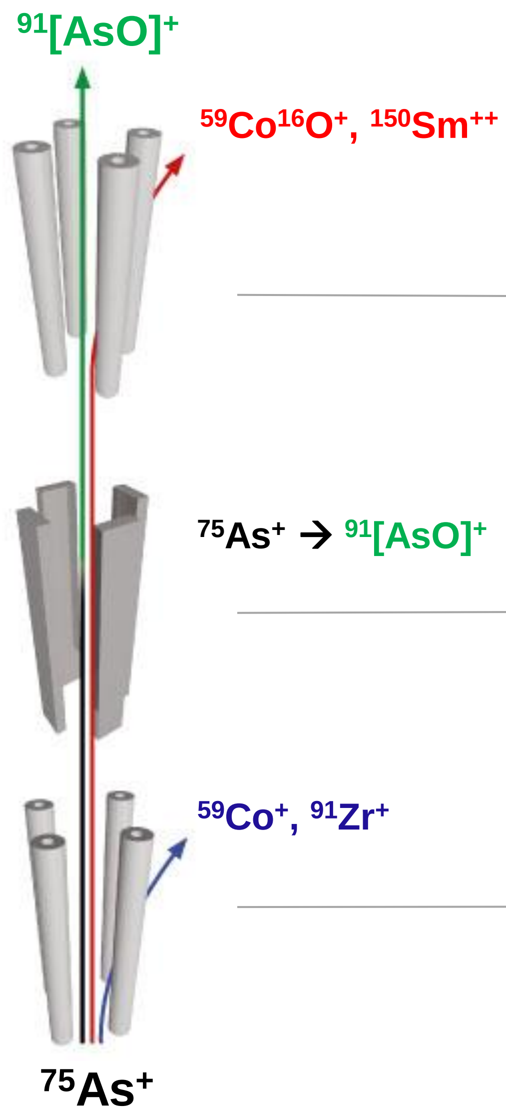
- Measure Yb on mass at 172
- NH_3 flow – 0.9ml/min

iCAP TQ ICP-MS: How it works - product ion reaction mode

Q3 set to product ion mass (*m/z* 91)

Q2 filled with reactive gas (O_2)

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

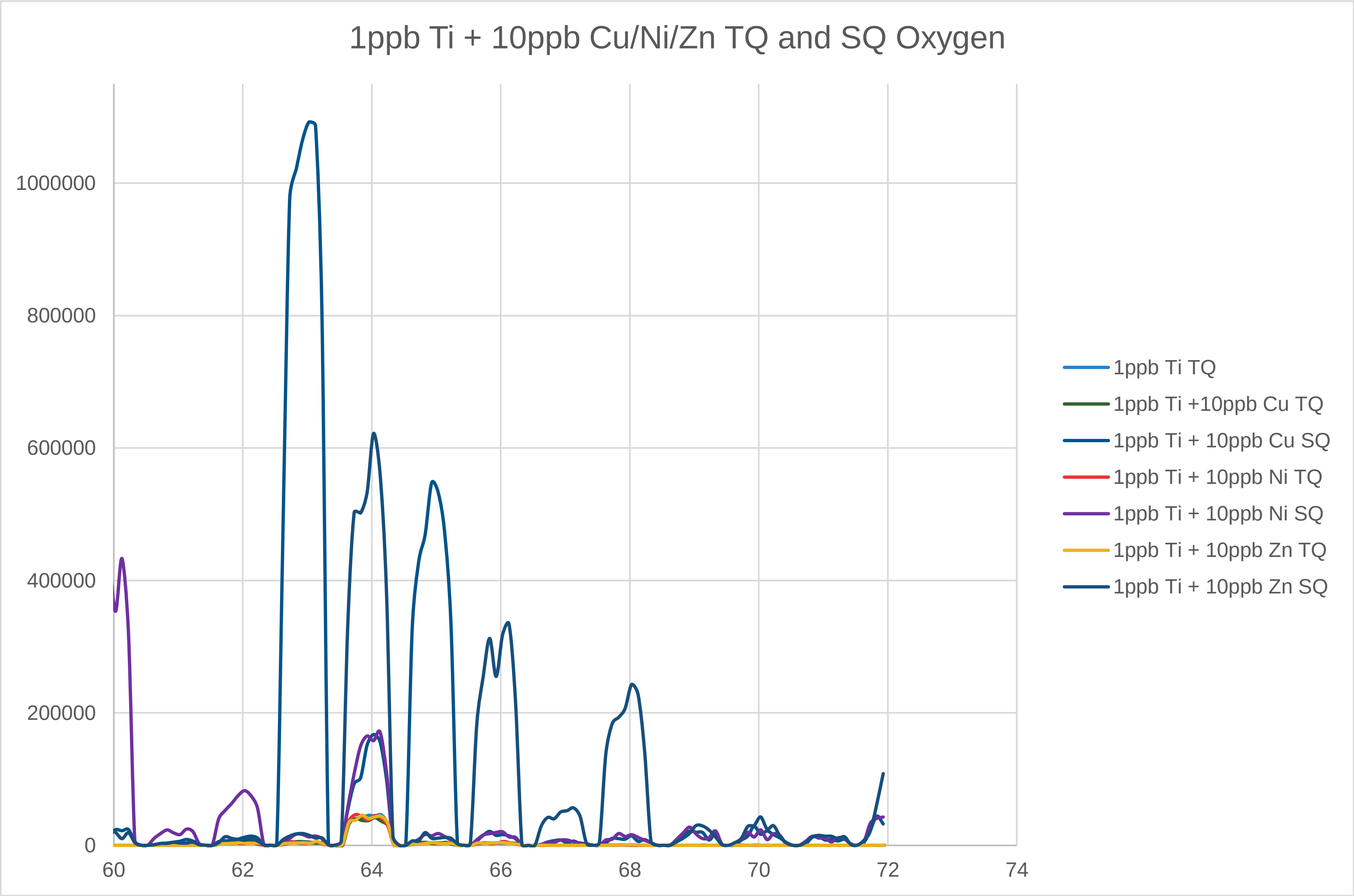


Q3 isolates the product ion of the analyte and removes any remaining interferences through a second stage of mass filtration

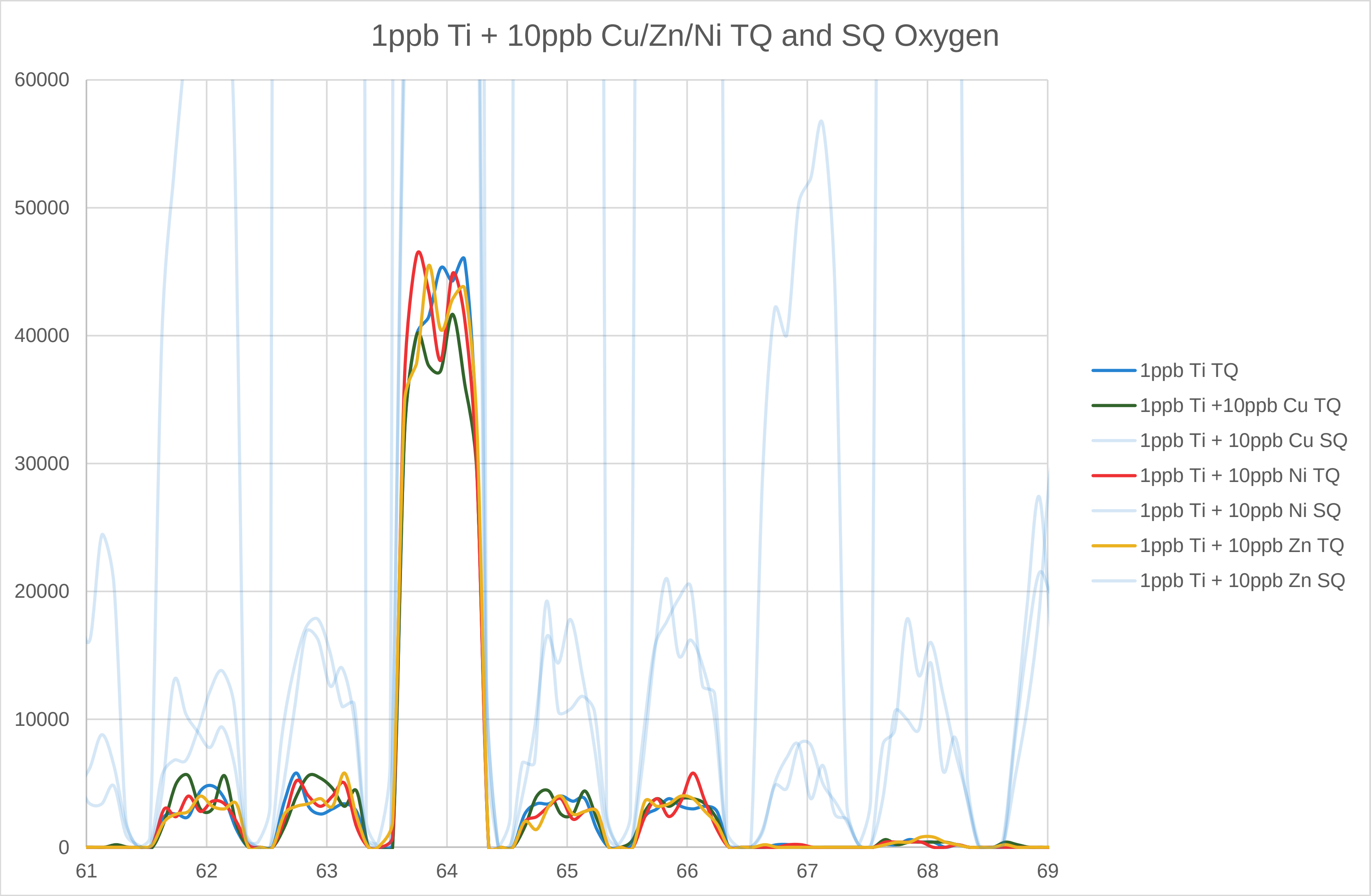
Optimal reaction conditions in Q2 are achieved through the selection of the appropriate measurement mode in Reaction Finder

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 product ion.

1ppb Ti – TQ and SQ modes (Oxygen)

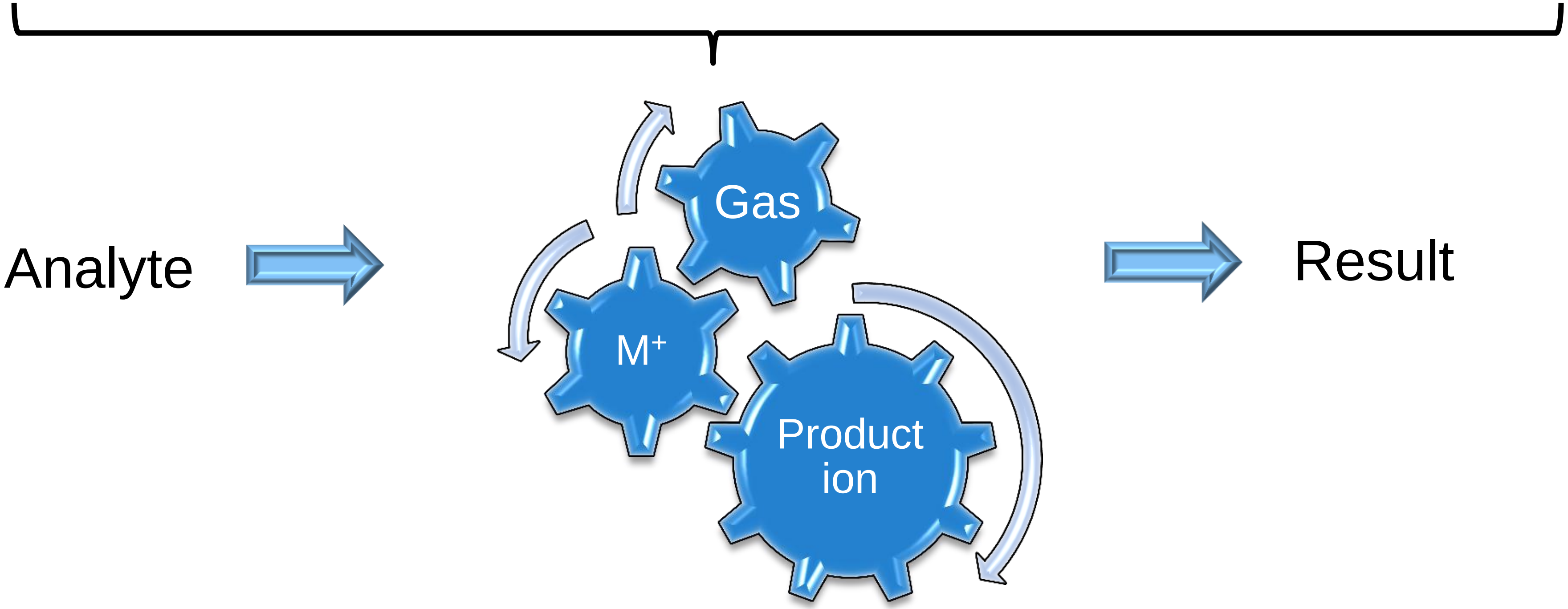


1ppb Ti – TQ – mass shift oxygen



All the **Power**, None of the **Complexity**

- Problem: when faced with measurement of a sample where interferences are expected, which is the best measurement mode?
- Solution: method development assistant – Reaction Finder
  - Software concept for intelligent selection of all 3 parameters
  - Just select the element for analysis and the software does the rest



Reaction Finder method development assistant

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

Reaction Finder in Thermo Scientific™ Qtegra™ ISDS Software

Reaction Finder is a supplied applet that preselects optimised conditions for each target isotope in each available mode

For example for ^{31}P, the Reaction Finder database defines the following method parameters:

Analyte type	Analyte	Is default isotope	Reaction gas	Q1 mass (u)	Q3 analyte	Is default Q3 Analyte	Is default reaction
Isotope	31P	✓	O_2 (Oxygen)	30.9737634	31P	☐	☐
Isotope	31P	✓	O_2 (Oxygen)	30.9737634	31P.16O	✓	✓
Isotope	31P	✓	O_2 (Oxygen)	30.9737634	31P.17O	☐	☐
Isotope	31P	✓	O_2 (Oxygen)	30.9737634	31P.18O	☐	☐
Isotope	31P	✓	O_2 (Oxygen)	30.9737634	31P.16O2	☐	☐
Isotope	31P	✓	O_2 (Oxygen)	30.9737634	31P.17O.16O	☐	☐
Isotope	31P	✓	O_2 (Oxygen)	30.9737634	31P.18O.16O	☐	☐
Isotope	31P	✓	O_2 (Oxygen)	30.9737634	31P.17O2	☐	☐
Isotope	31P	✓	O_2 (Oxygen)	30.9737634	31P.18O.17O	☐	☐
Isotope	31P	✓	O_2 (Oxygen)	30.9737634	31P.18O2	☐	☐
Isotope	31P	✓	H_2 (Hydrogen)	30.9737634	31P	☐	☐
Isotope	31P	✓	H_2 (Hydrogen)	30.9737634	31P.1H4	✓	☐
Isotope	31P	✓	None (No reaction gas)	30.9737634	31P	✓	☐
Isotope	31P	✓	He (Helium)	30.9737634	31P	✓	☐

None of the complexity, all of the flexibility:

- **Default reactions for all modes of iCAP TQ ICP-MS operation including collision/ reaction gases such as O_2, H_2, NH_3 and He**
- **Dedicated mass flow controller for each cell gas**

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iCAP TQ ICP-MS – Feature summary

Robust and reliable

Completely re-designed electronics

New RF-generator

QCell CRC - Safe use of reactive, pure gases incl. H_2 and NH_3

4 MFCs as standard for max. flexibility

Integrated automation, advanced applications and qualification

Easier to install and service

Qtegra ISDS software 2.8 with Reaction Finder

Ease of Use and Simplicity

Best LODs in challenging matrices

Same footprint as the iCAP RQ SQ-ICP-MS

Same robust sample introduction, interface and ion lens system

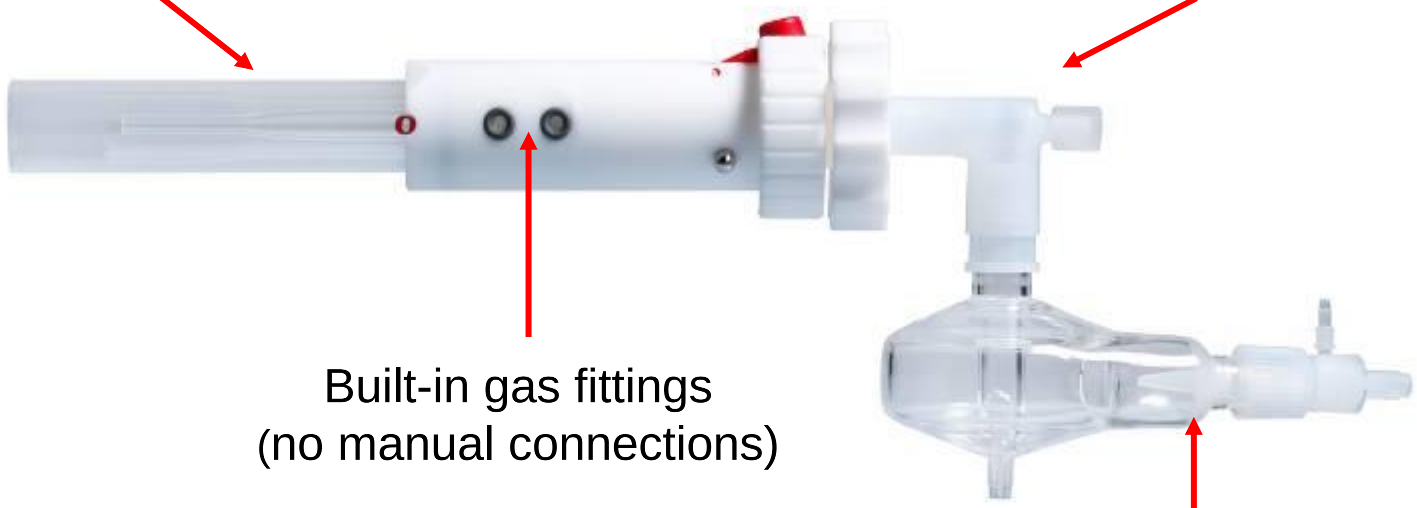
Future proof

Intuitive quick-connect sample introduction components

O-ring free self aligning injector

O-ring free demountable torch

Gas addition port (organics, aerosol gas diliution)



Built-in gas fittings (no manual connections)

O-ring free spray chamber

Interface design

Bench level pop-out interface for easy ambidextrous access to the cones

and

the extraction lens for simplest possible routine maintenance

…without needing to break the vacuum

Ion focusing: the *RAPID* lens

Right

Angle

Positive

Ion

Deflection

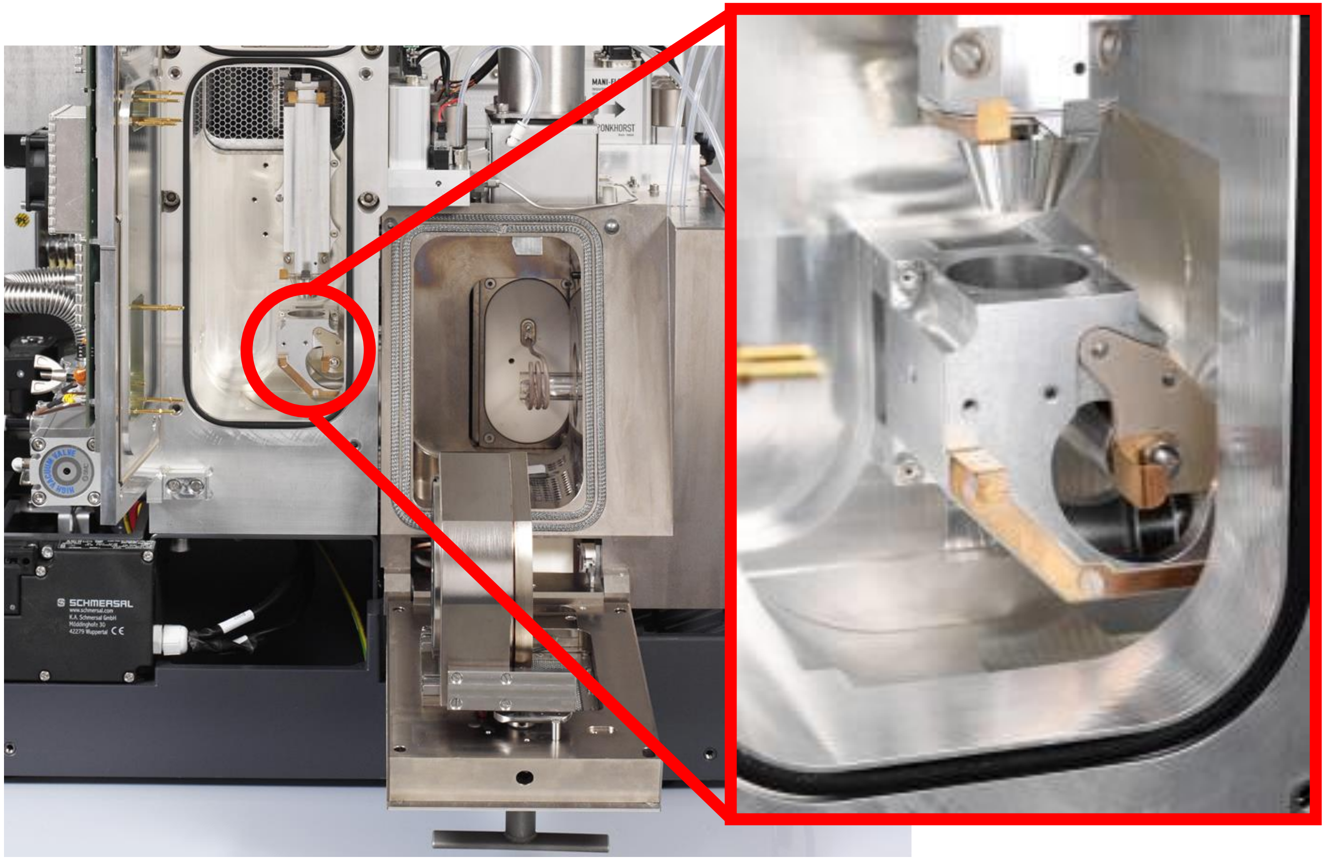
90º ion focusing with total ion deflection in 3 dimensions

and

Elimination of neutral species

for

Highest signal to noise ratio of any ICP-MS



ThermoFisher SCIENTIFIC

Redefining trace element analysis – application areas

Meeting human health
and environmental challenges

Advancing development in
metals, materials and chemicals

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

- **Material Analysis**
- **Nanoparticle Characterization**
- **Metallurgy**
- **Energy Production**

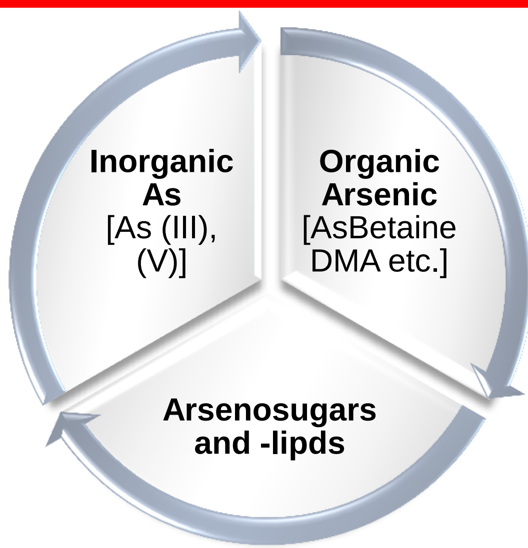
Arsenic and selenium in environmental samples

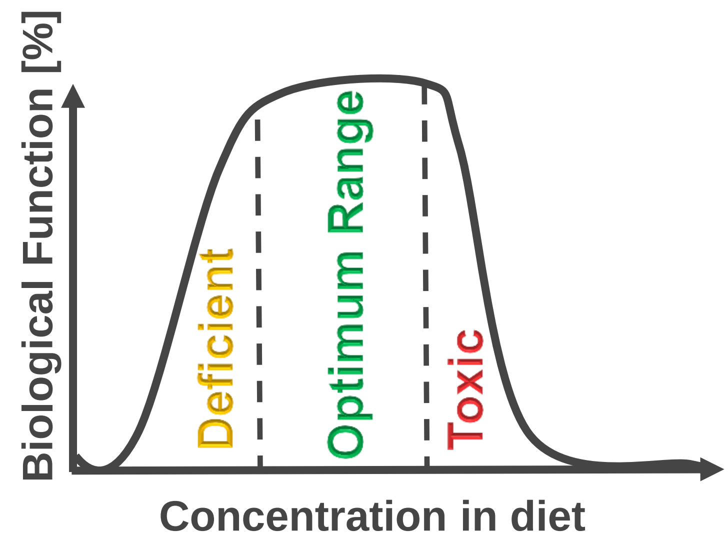
Low levels in samples

Multiple interferences: $ArCl^+$, Ar_2^+ and REE^{++}

33
As
Arsenic
74.921595

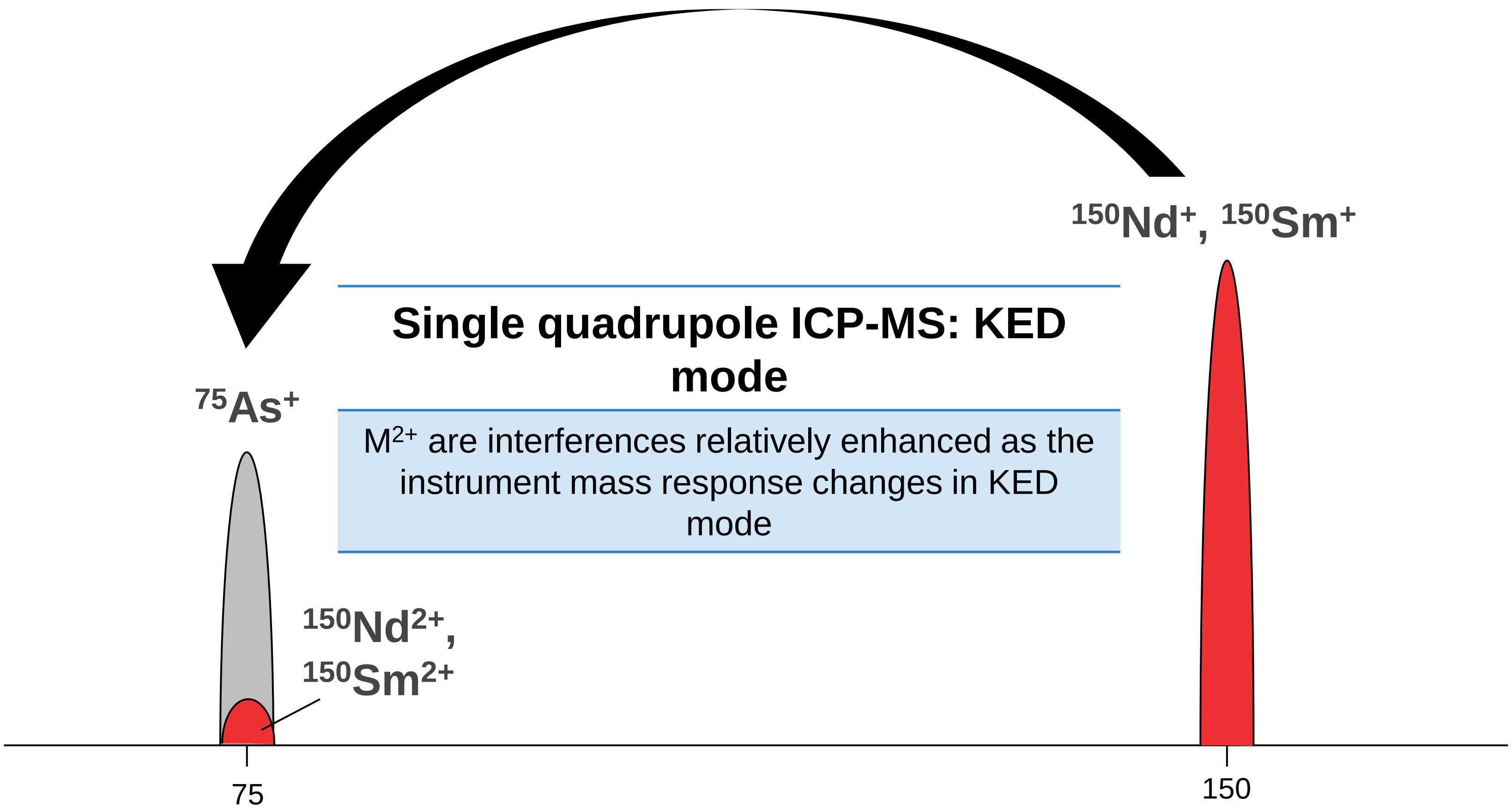
34
Se
Selenium
78.971





As and Se analysis in the presence of REE's – the problem

Usual interferences on As and Se - Ar_2, ArCl - easy to remove using He KED, but if REE are present...

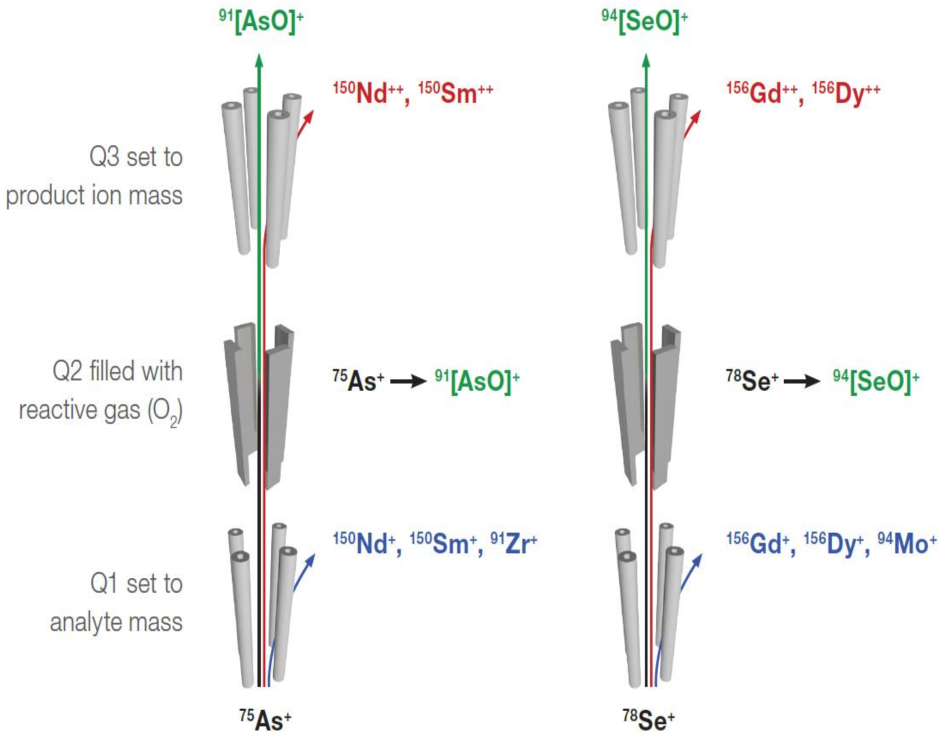


Single quadrupole ICP-MS: KED mode

M^{2+} are interferences relatively enhanced as the instrument mass response changes in KED mode

As and Se analysis in the presence of REE's: the iCAP TQ solution

- Control ions entering the collision cell using **Q1**
- Use O_2 to efficiently convert As and Se to AsO^+ and SeO^+ in **Q2** (i.e. the collision cell)
- REE^{++} species don't react
- Selectively detect AsO^+ (at mass 91) and SeO^+ (at mass 94) 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
	$^{150}Sm^{2+}$		$^{156}Dy^{2+}$	

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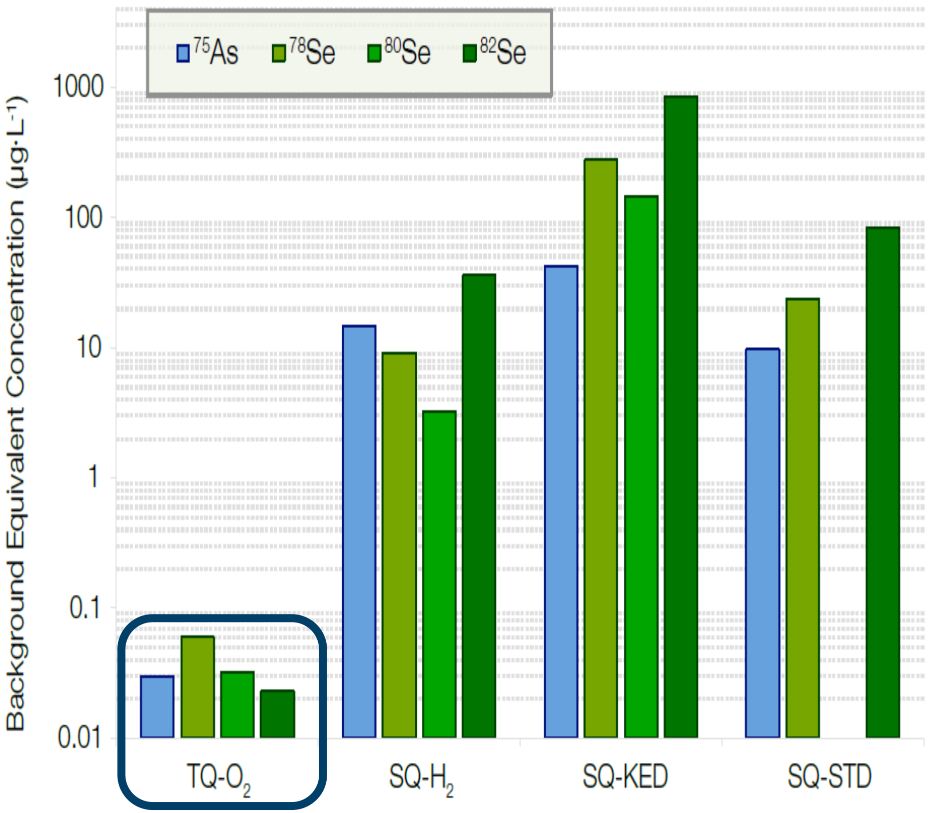
As and Se with REE present - results in different modes

Interference removal capability in each mode



- 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
- Accuracy assessed by analysis of AGV andesite reference material and a deep sea sediment
- Spike recovery tests also performed

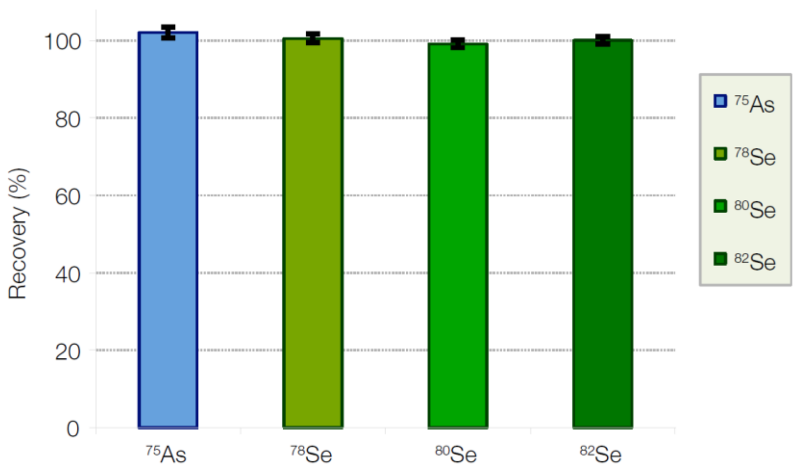
Proving the accuracy of the analysis

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	-

101% recovery for As in AGV reference material

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 %

Multi-element results: River water reference material (NRC SLRS-5)

Analyte	Measurement Mode	Measured µg/L	Certified Concentration	Recovery %
^{23}Na	KED	5085	5380	95
^{24}Mg	KED	2665	2540	105
^{27}Al	KED	55.3	49.5	112
^{39}K	KED	863	839	103
^{56}Fe	KED	93.2	91.2	102
^{59}Co	KED	0.05	0.05	107
^{60}Ni	KED	0.52	0.48	110
^{63}Cu	KED	18.2	17.4	105
^{75}As	TQ-O2	0.43	0.41	104
^{78}Se	TQ-O2	0.10		
^{208}Pb	KED	0.08		
^{238}U	KED	0.10	0.09	109

Determination of trace elements in metals and alloys

28
Ni
Nickel
58.6934

34
Se
Selenium
78.971

NiO^{+} interference

Nickel alloys are used for industrial applications where resisitance to high temperature is required e.g. aircraft turbine blades. Selenium content is critical, as this leads to weakness in the alloy composition

Solid Samping

Liquid Sampling

LA-ICP-MS

GD-MS

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

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
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	$^{82}Kr^+$

KED or H_2

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
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	$^{82}Kr^+$	$^{64}Ni^{18}O^+$, $^{64}Ni^{16}O^{1}H^+$

KED or H_2

O_2 conversion into SeO

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	$^{82}Kr^+$	$^{64}Ni^{18}O^+$, $^{64}Ni^{16}O^{1}H^+$	$^{81}Br^{1}H^+$

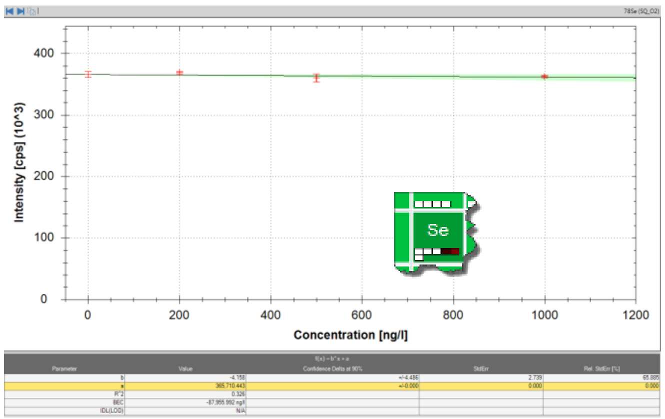
KED or H_2

O_2 conversion into SeO

KED or H_2 don’t help

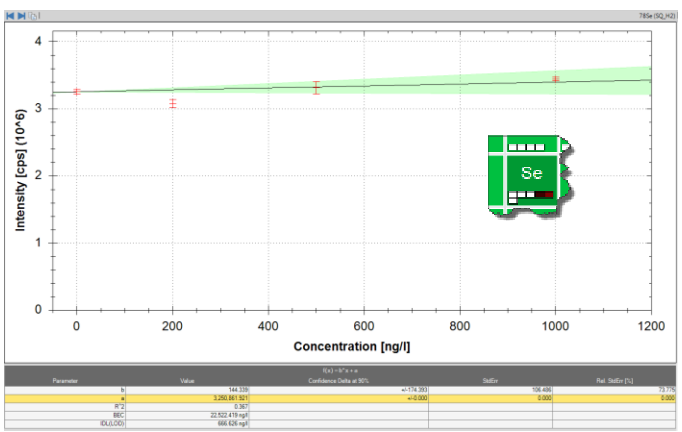
Interference removal using SQ-ICP-MS

Sample: Se in 100 ppm Ni



O_2

H_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

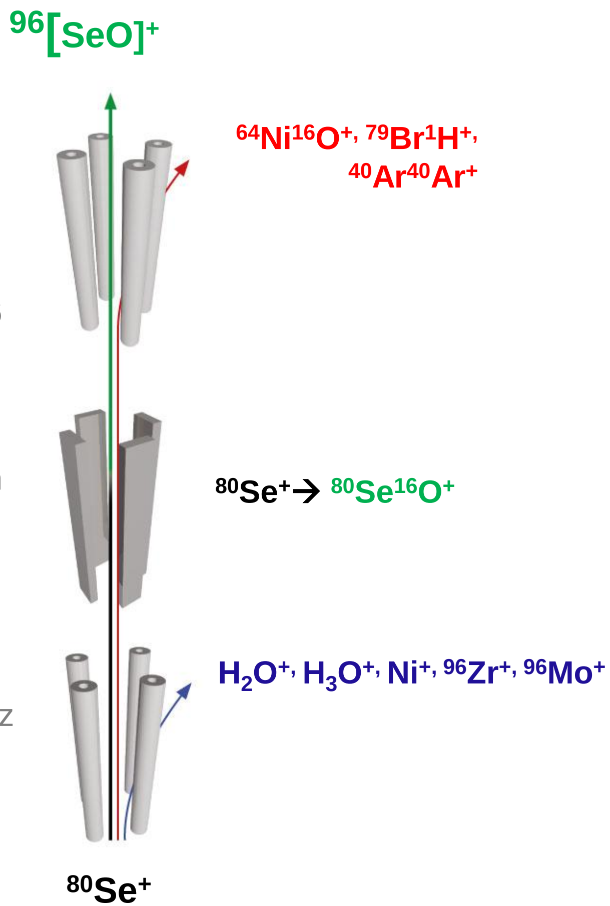
Ion Mass	Identifier	Interference
92	$^{76}Se^{16}O^+$	$^{58}Ni^{16}O(H_2O)^+$
93	$^{77}Se^{16}O^+$	$^{58}Ni^{16}O(H_3O)^+$
94	$^{78}Se^{16}O^+$	$^{60}Ni^{16}O(H_2O)^+$, $^{58}Ni^{18}O(H_2O)^+$
96	$^{80}Se^{16}O^+$	$^{62}Ni^{16}O(H_2O)^+$, $^{60}Ni^{18}O(H_2O)^+$
98	$^{82}Se^{16}O^+$	$^{64}Ni^{16}O(H_2O)^+$,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

Interference removal using TQ-ICP-MS

Q3 Q3 set to product ion mass m/z 96

Q2 Q2 filled with reactive gas O_2

Q1 Q1 set to m/z 80



Only initial separation of lower mass ions enables effective and complete removal of all interferences on Se – using both H_2 and O_2 reactive gases

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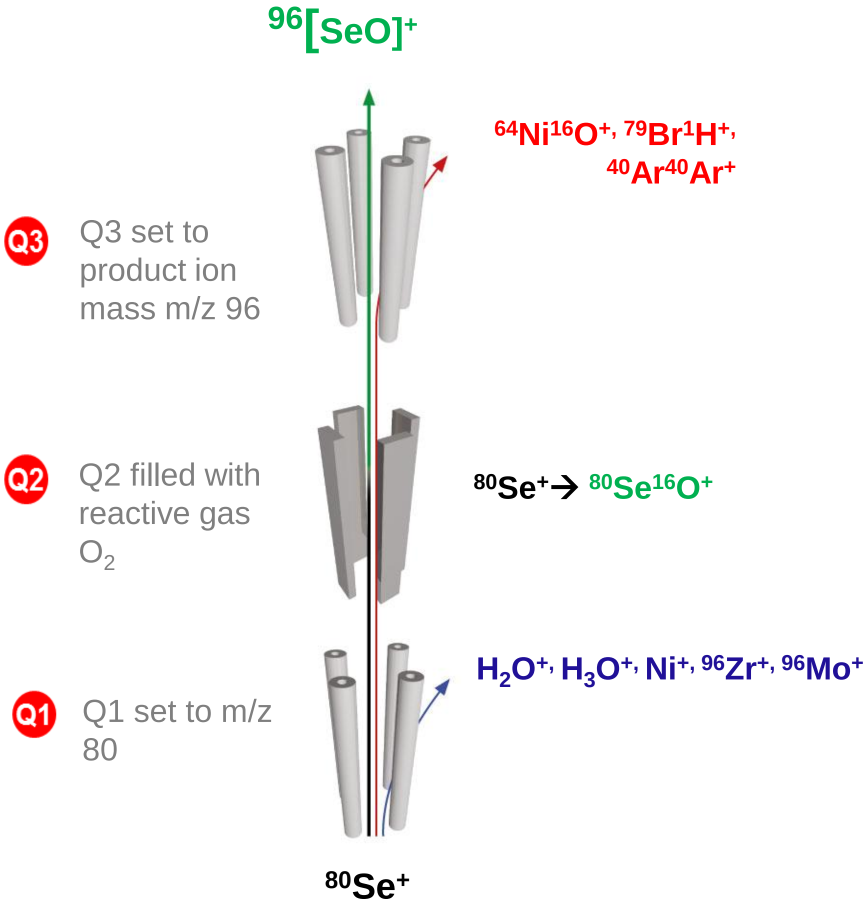
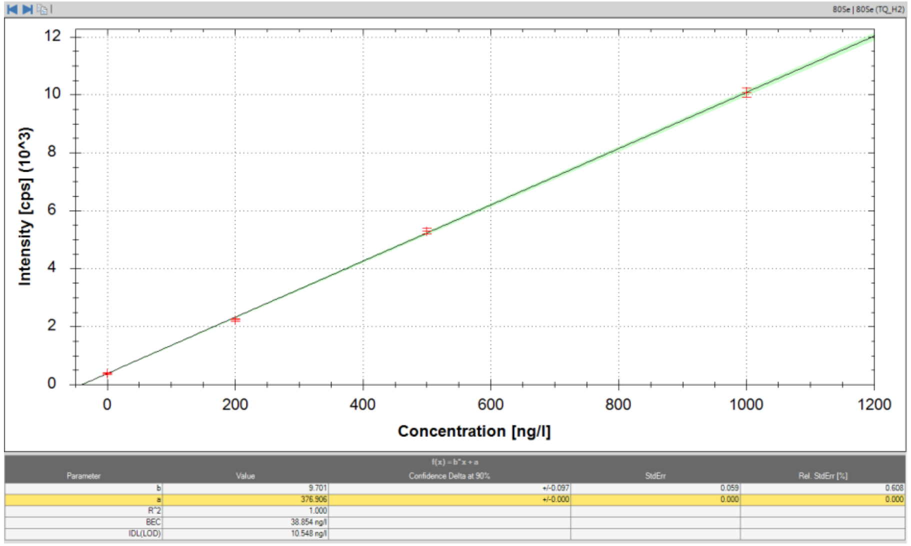
Interference removal using TQ-ICP-MS



Only initial separation of lower mass ions enables effective and complete removal of all interferences on Se – using both H_2 and O_2 reactive gases



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

Determination of Ti in biological samples using ICP-MS

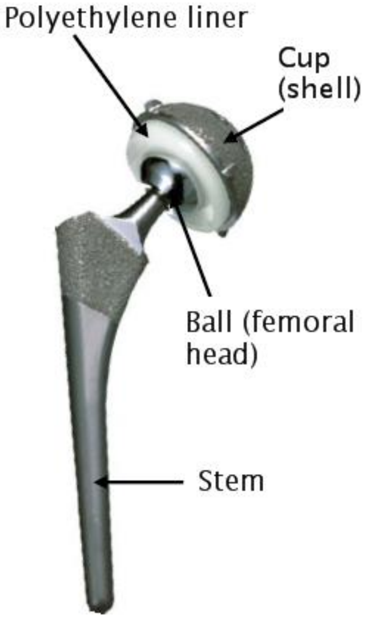
22

Ti

Titanium

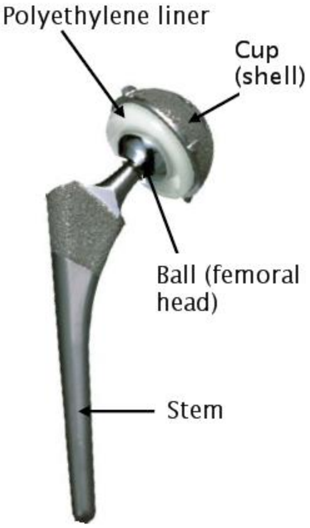
47.867

- Titanium based components used for orthopedic and dental implants.
- Degradation of these implants releases Ti (and Co, Ni and Cr too) into the body
- $^{48}Ca^+$, PO^+, SO^+, SOH^+ interference on Ti isotopes
- HR-ICP-MS effective technique, but expensive



Determination of Ti in biological samples using ICP-MS

- Preliminary work started to measure titanium in hip samples, via serum samples
- Three modes compared:- He KED, SQ NH_3 and TQ NH_3
- Aim: To test if TQ mode gives low enough LOQ to enable determination of the normal Ti levels in patient samples
- Lowest LOQ only possible with Ti isotope at m/z 48 (abundance 73.8%), but serum high in Ca (^{48}Ca interference
- Solution: Use ammonia as the reaction gas to isolate m/z 48 Ti from Ca

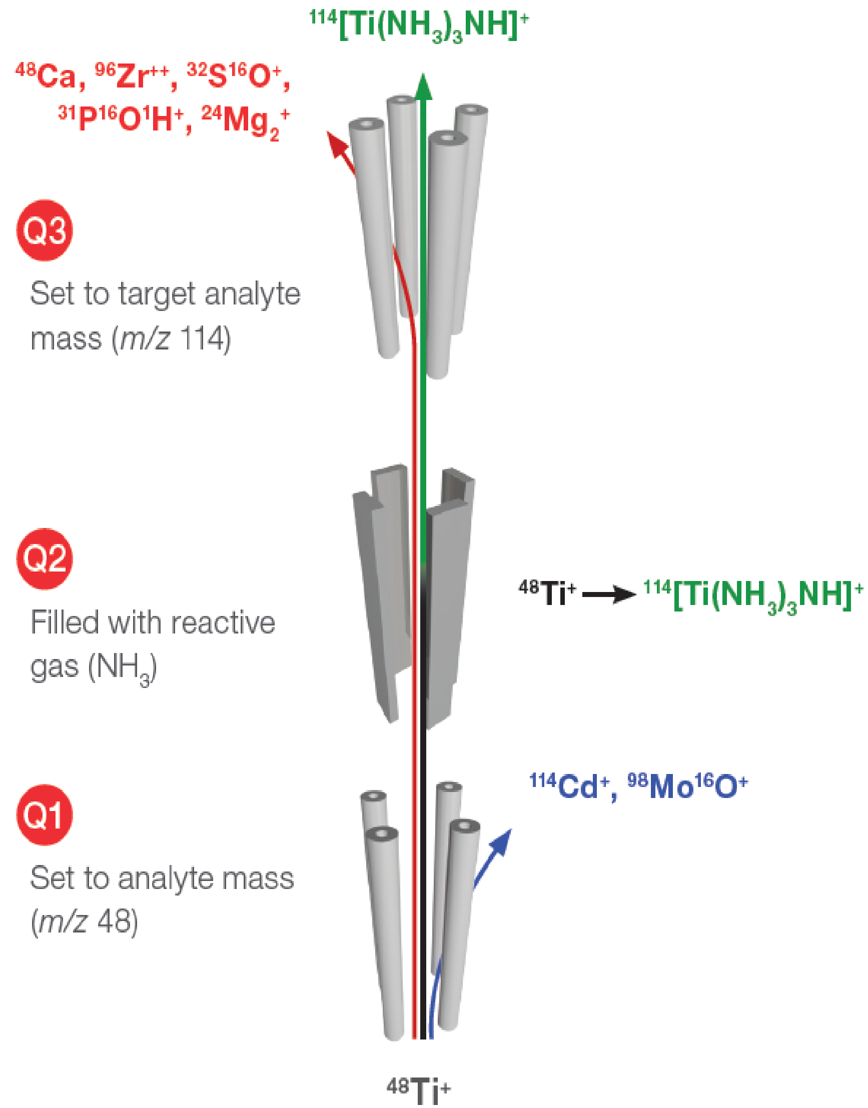


Reaction of Ti with NH_3: how it works

- **Q1** – set to transmit Ti, potential interferents on the product ion (e.g. ^{114}Cd) and lower mass interference precursors (e.g. ^{31}P, ^{16}O) rejected.

- **Q2** – filled with NH_3. Ti collides and generates a range of adducts including $^{48}Ti(NH_3)_3NH^+$ at mass 114

- **Q3** – set to transmit mass 114, other masses rejected.



Comparison of different ICP-MS modes for Ti analysis

Sample matrix - 1:10 diluted serum plus 1ppm Cd, all data in µg/L

Sample i.d.	He KED mode, on mass at ^{48}Ti	Ti SQ NH_3 mode, at mass 114	Ti TQ NH_3 mode, at mass 114	Ti reported value, measured at ^{47}Ti using HR-ICP-MS
Serum L-1	167	1800	6.64	6.8
Serum L-1	262	1850	6.38	6.8

^{48}Ca interference plus residual PO^+ etc.

Contribution from ^{114}Cd

Only TQ NH_3 mode is capable of providing the correct Ti result

Accurate multi-element, multi-mode analysis with the iCAP TQ

Standard mode (i.e. no cell gas) with SQ operation

He KED single quadrupole mode with cell pressurised with He and KED applied

TQ NH_3 / H_2 / O_2 triple quadrupole mode with CRC pressurised with reaction gas Q1 set to analyte mass and Q3 set to either analyte mass (on mass analysis) or product ion (mass shift analysis)

- Flexibility and usability of both single and triple quadrupole modes
  - Full multielemental analysis with dedicated TQ interference removal for difficult analytes and simple He KED mode for everything else **in one analytical run**

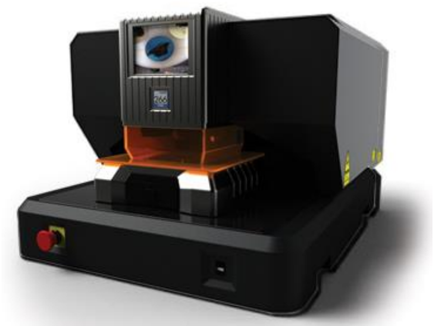
Redefining TQ-ICP-MS - accessories

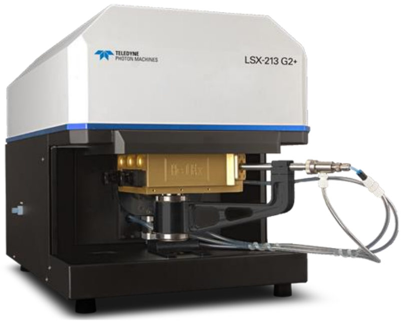
Fully integrated autosampler and autodilution solutions

Elemental Scientific prep*FAST*

CETAC SDX_{HPLD}

Fully integrated speciation (IC and LC) and laser solutions





Questions?

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Thermo Scientific iCAP TQ ICP-MS

Redefining triple quadrupole ICP-MS
with unique ease of use