

Introduction

- Brief background on Thermo Scientific ICP-MS history
- What are the key aspects making ICP-MS a key tool in many applications and regulated methods?
- Recent trends in single and triple quadrupole ICP-MS



Thermo Fisher Scientific > 35 years of ICP-MS experience



- Quadrupole ICP-MS
 - 1st commercialized Q-ICP-MS in 1983
 - 5th Generation of single quadrupole ICP-MS instruments



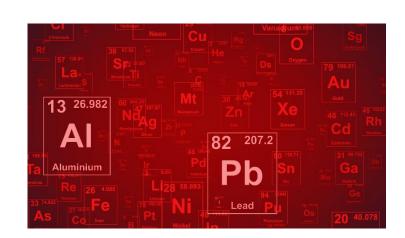
- High Resolution ICP-MS
 - 1st routine HR-ICP-MS introduced in 1994
 - Following instrument generations with significantly improved performance and extended dynamic range



Thermo Fisher

- Triple Quadrupole ICP-MS
 - Added in 2017 with a strong focus on combining superior interference removal and ease of use in a compact system
 - Bringing TQ-ICP-MS into applied analytical testing labs

Advantages of TQ-ICP-MS over SQ-ICP-MS



Better Interference Removal

- Overcome false positives Always an interruption to the daily workplan and a significant cost as well
- Open up new options future proof your laboratory today



Better Sensitivity and LOD's

- Take on new challenges Analysis of emerging contaminants
- More confidence Sometimes there is more than "less than"



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Option to increase productivity

- Reduce sample turnover times –
 Provide right answers faster
- Simple method development Easy and straightforward to leverage new technology from day 1.

There are good reasons for TQ-ICP-MS in a busy laboratory

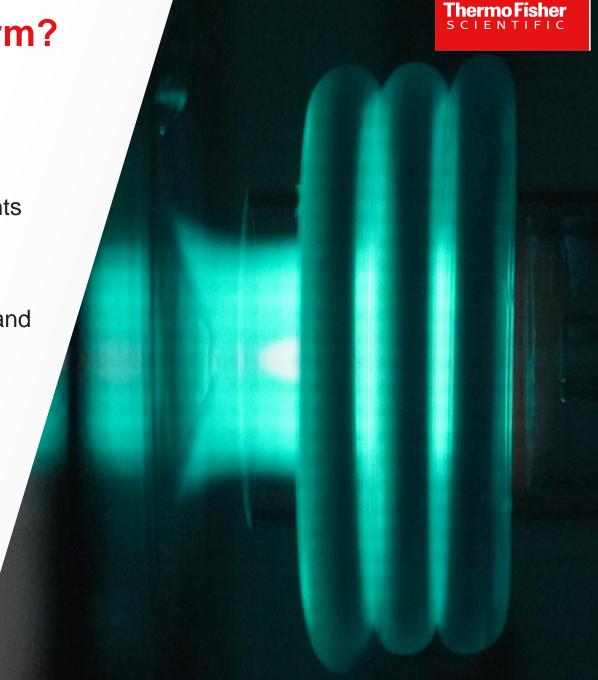
Where do spectral interferences form?

In the argon plasma

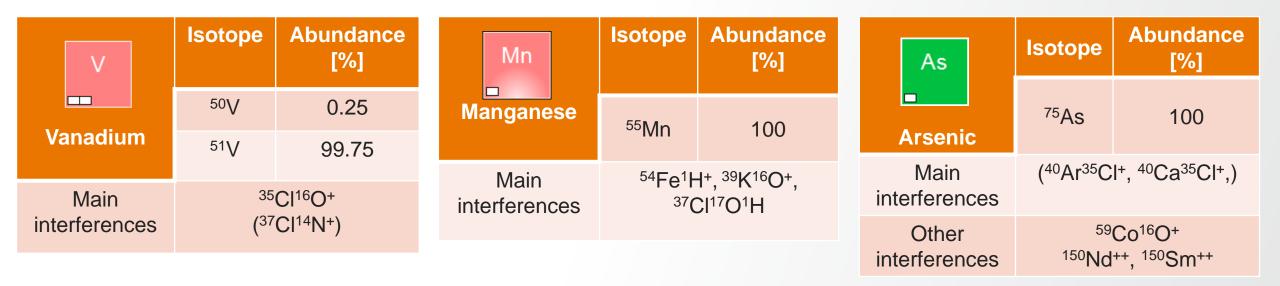
- High temperature environment (6,000 8,000K)
- Ar ions formed and react with sample matrix components (O, Cl, H, Na etc.) to form polyatomic species
- Plasma is hot enough for elements with low enough second ionisation potentials to lose a second electron and become doubly charged

In the sampling interface

- Steep temperature gradient in the boundary layer between the plasma and the cooled sample cone induces recombination reactions
- Temperature drop between the sample and skimmer cone can also lead to recombination processes



How much better is a TQ-ICP-MS?

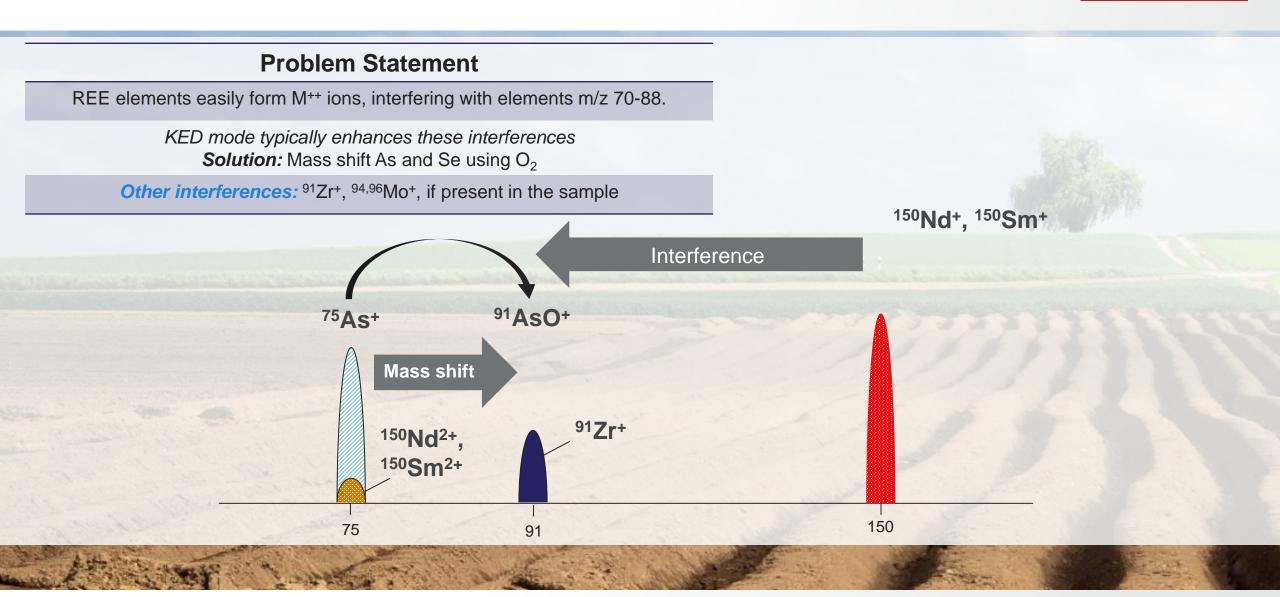


They are monoisotopic or quasi monoisotopic!

V	lsotope	Abundance [%]	Mn	lsotope	Abundance [%]	As	Isotope	Abundance [%]	
Vanadium	50 ∨ 51∨	0.25 99.75	Manganese	⁵⁵ Mn	100	Arsenic	⁷⁵ As	100	
Main	³⁵ Cl ¹⁶ O+ (³⁷ Cl ¹⁴ N+)		Main ⁵⁴ Fe ¹ H ⁺ , ³⁹ K ¹⁶ O ⁺ interferences ³⁷ Cl ¹⁷ O ¹ H		· ·	Main interferences	(⁴⁰ Ar ³⁵ Cl ⁺ , ⁴⁰ Ca ³⁵ Cl ⁺ ,)		
interferences IDL			IDL	IDL order have		Other interferences	⁵⁹ Co ¹⁶ O+ ¹⁵⁰ Nd++, ¹⁵⁰ Sm++		
Improvement	20 times better		Improvement	9 times better		IDL Improvement	t 10 times better		
Sensitivity Improvement	10 times higher		Sensitivity Improvement	4 times higher		Sensitivity Improvement	6 times higher		

Increase in sensitivity and a more significant decrease in detection limit leads to a significantly better signal-to-noise ratio!

Accurate analysis of arsenic in presence of REE



Thermo Fisher

Arsenic analysis

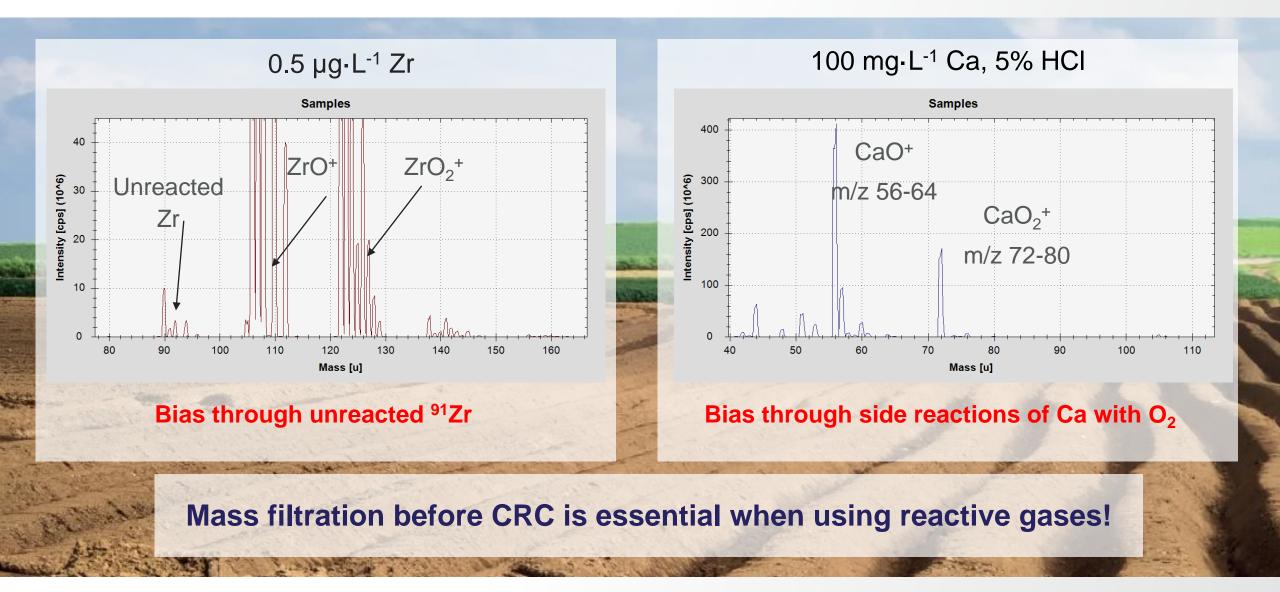
Sample	SQ-KED [µg·L ⁻¹]	TQ-O ₂ [µg·L ⁻¹]	Comment			
100 mg⋅L ⁻¹ Ca, 5% HCl	0.06	0.00	KED is suitable, but not capable to eliminate all CI based interferences			
10 µg⋅L ^{₋1} REE Mix	1.05	0.00				
100 µg⋅L ⁻¹ REE Mix	11.97	0.00	KED shows expected false positive result			

Arsenic analysis

	Sample	SQ-KED [µg∙L⁻¹]	TQ-O ₂ [μg·L ⁻¹]	SQ-O₂ [µg∙L⁻¹]	Comment
	100 mg⋅L ⁻¹ Ca, 5% HCl	0.06	0.00	0.2	KED is suitable, but not capable to eliminate all CI based interferences SQ-O ₂ shows a significant bias
	10 µg∙L⁻¹ REE Mix	1.05	0.00	0.02	KED shows expected false positive result
N	100 µg⋅L⁻¹ REE Mix	11.97	0.00	0.04	SQ-O ₂ is less effective compared to TQ-O ₂
	0.5 mg∙L ⁻¹ Zr	0.00	0.00	148.72	No mass filtration in Q1 leads to a completely wrong result in SQ-O ₂ mode
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What went wrong – Survey Scans in SQ-O₂ Mode



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What causes the complication...

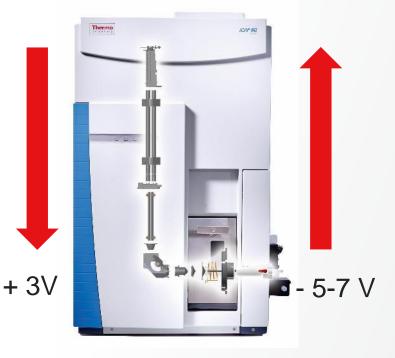
- Reactive gases can provide additional means for interference removal for difficult elements
 - Sulfur, phosphorous etc.
- The reality in a complex sample is usually quite different

KED mode

Positive bias potential between CRC and quad (3V)

Species are slowed down and all polyatomics are removed

This includes ions you normally don't worry about...



Reactive mode

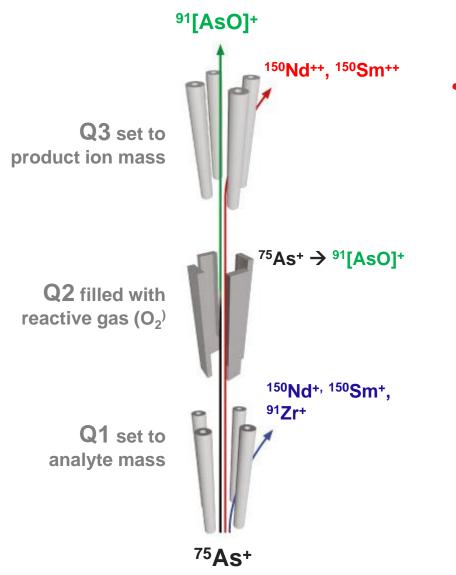
Thermol

Negative bias potential between CRC and quad (5-7V)

All ions are accelerated through the CRC and may react with what ever gas is used!

Some ions can cause significant problems, although not an obvious interference

Accurate analysis of arsenic in presence of REE



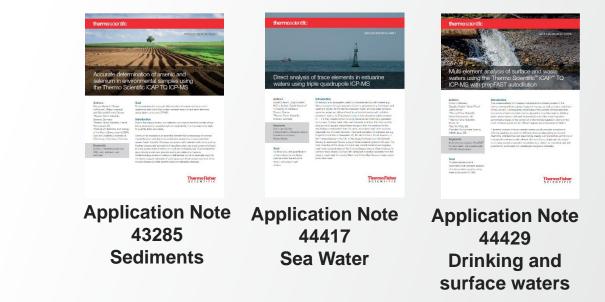
Solution is TQ-ICP-MS:

- Control ions entering the cell using Q1
- Use O₂ to efficiently convert As and Se to AsO and SeO in Q2

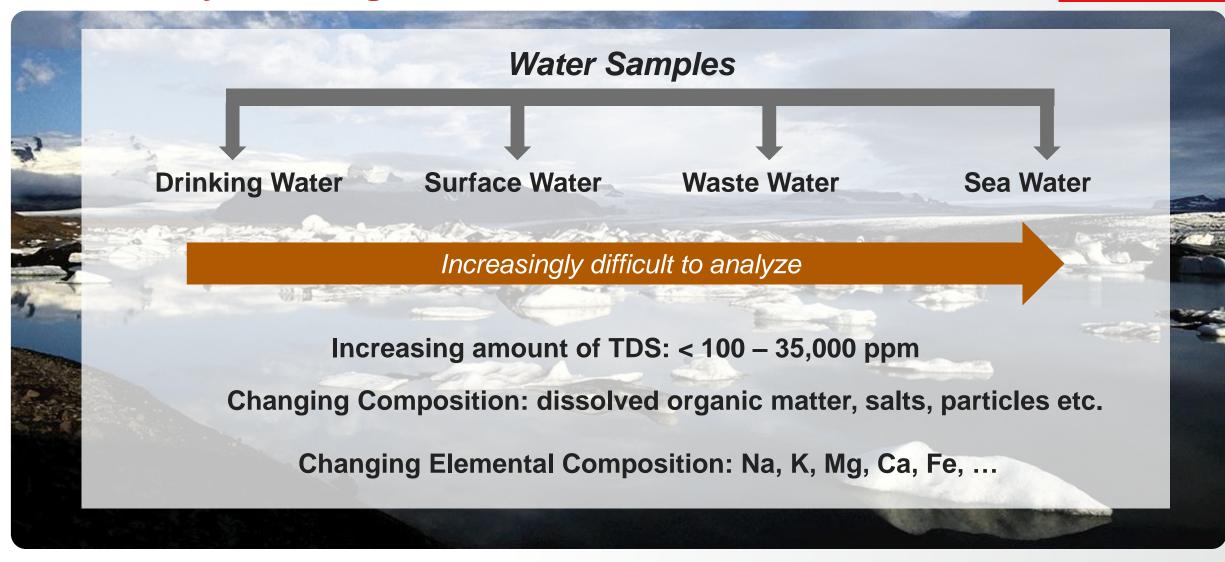
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- Selectively detect AsO free from REE⁺⁺ interference, using Q3
- Better interference removal reduces false positives

Sensitivity and LOD are improved



Water Analysis using ICP-MS



Adding rare earth elements as emerging contaminants

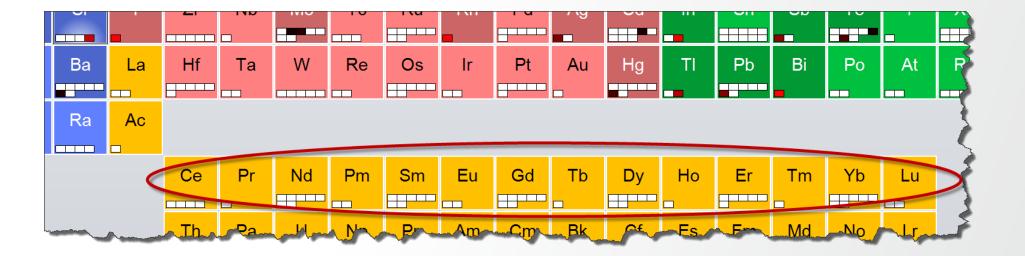


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Challenges with the analysis of rare earth elements

• Are there any?

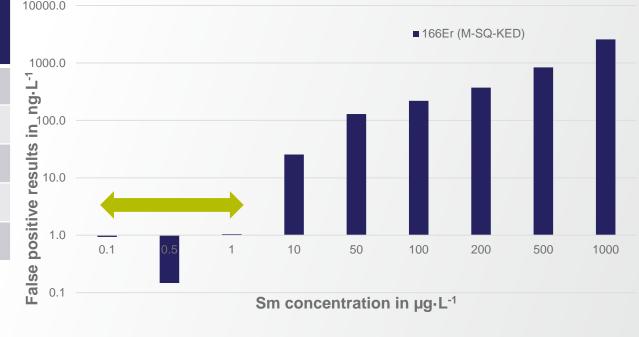


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- Very low concentrations in nature, therefore background contamination is normally negligible
- Mass range between 140-180 is usually free from interferences and offers high sensitivity
- Rare earth elements form oxides quite easily, allowing to use reactive mode for even better sensitivity!
- Rare earth elements do interfere on other elements, but also amongst themselves!

Challenges with the analysis of rare earth elements

Element	Potentially interfering on	Interference
Barium	e.g. Europium	BaO+
Cerium	Gadolinium	CeO+
Neodymium	e.g. Terbium	NdO+
Samarium	e.g. Erbium	SmO+
Gadolinium	Ytterbium	GdO+



Thermol

- ✓ ¹⁶⁶Er is the most abundant isotope (33.60%)
- Although ¹⁵⁰Nd and ¹⁵⁰Sm are not the most abundant isotopes of both elements, significant interferences can be observed when using KED mode

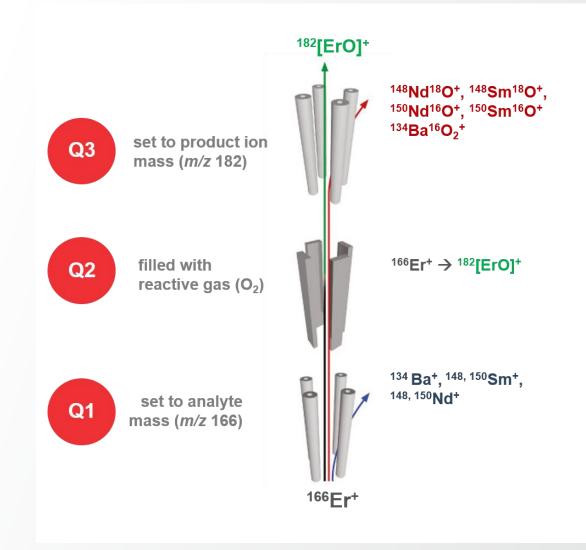
False positive results could be obtained for 166 Er in presence of less than $10\mu g \cdot L^{-1}$ of other REE's (here Sm) when measured in KED mode

Challenges with the analysis of rare earth elements

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Adding REE analysis to a novel ICP-MS method

- Collection of different surface waters in and around Bremen
 - Broad range of TDS and other content
- Method Development
 - Optimize for throughput conscious laboratories
 - Straightforward definition, yet full interference removal
- Preliminary Validation
 - River sediment CRM
 - River Water CRM
 - Spike recovery



Instrument configuation and sample preparation



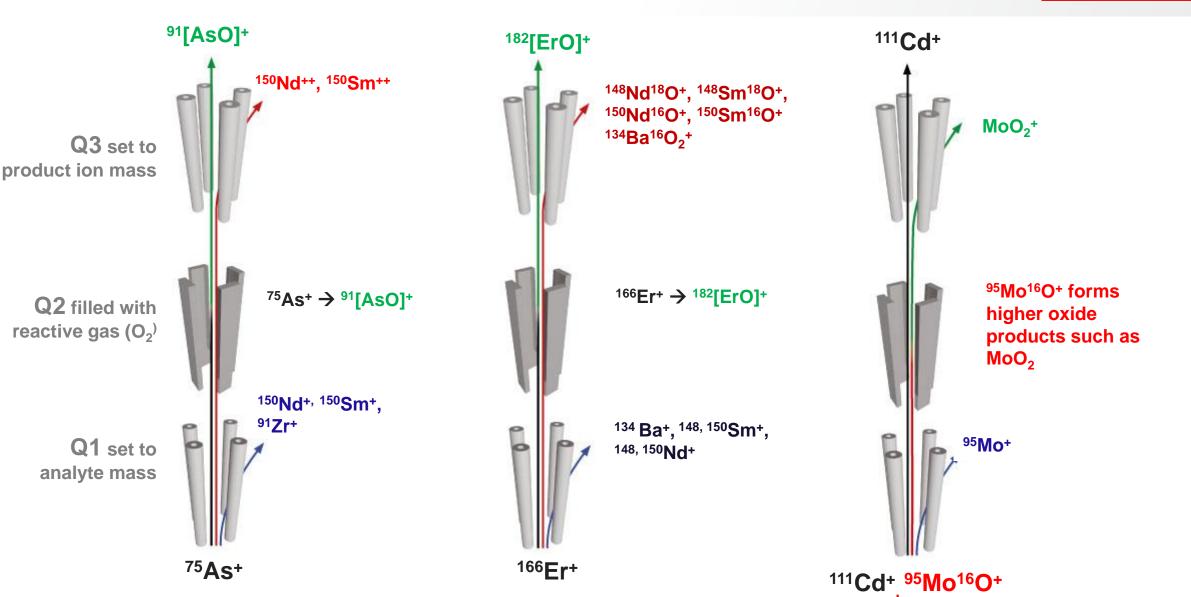
- ✓ Water samples were acidified and filtered
- ✓ BCR-2 was digested (introducing a 2,500 times dilution)

Parameter	Value				
Nebulizer	Borosilicate glass, 400 µL·min ⁻¹				
Spraychamber	Quartz cyclonic, cooled at 2.7 °C				
Injector	2.5 mm i.d., quartz				
Interface	Nickel sampler and nickel skimmer cone with robust insert				
Plasma power	1550 W				
Nebulizer gas	1.04 L∙min ⁻¹				
Analysis Mode	TQ-O ₂				
Gas flow	100% O₂, 0.34 mL⋅min ⁻¹				
Sample uptake and wash	55 sec each				
Total analysis time	2 min 50 sec				



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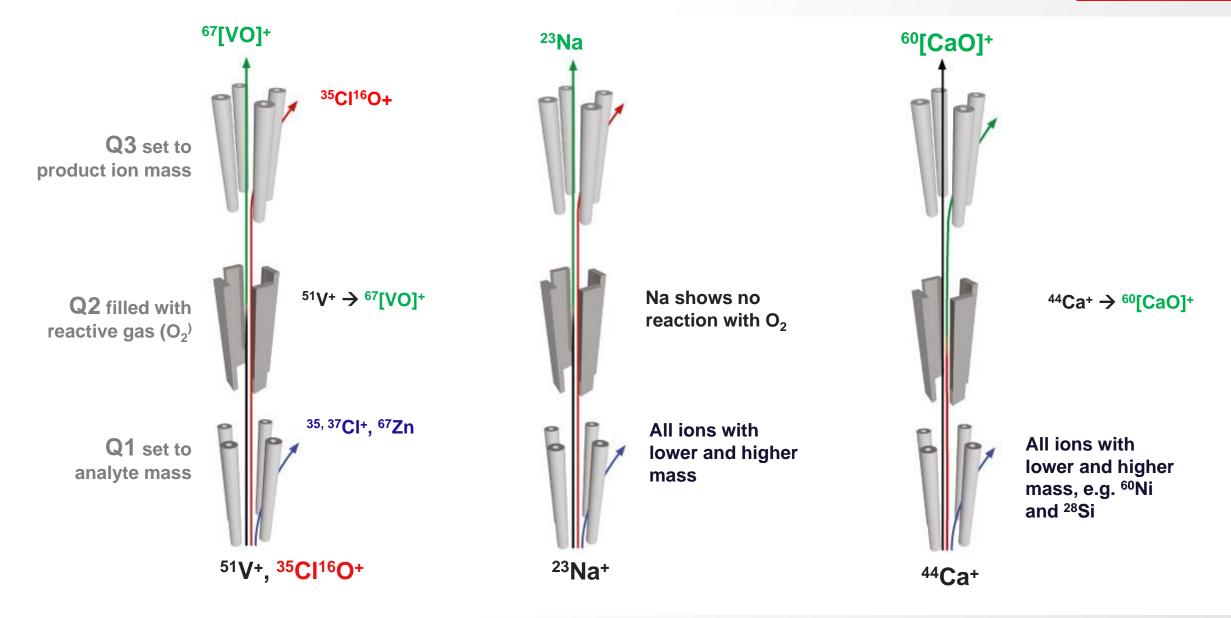
Method Development – Single Mode Analysis



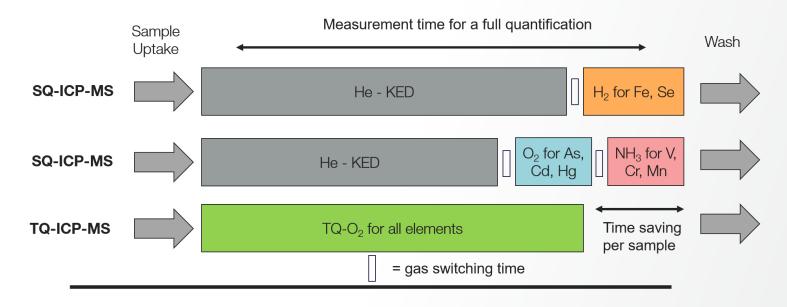
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Method Development – Single Mode Analysis



Let's have a look at throughput



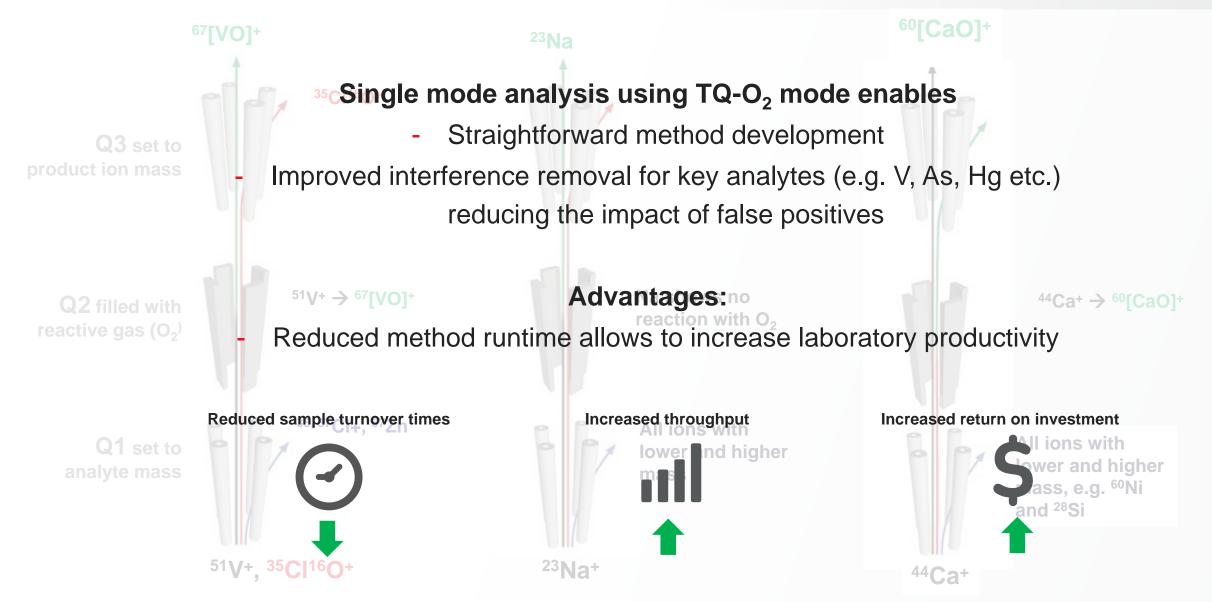
✓ Measurement time per sample reduced at no significant expense on performance

✓ Improved detection limits for a series of elements

✓ Confidence that all interferences are under control

- Often, combinations of different cell gases are used for improving on key analytes on a single quadrupole ICP-MS
- However, every gas switch is adding extra time to the method

Method Development – Single Mode Analysis



Results – Rare earth elements in Columbia River Basalt

Analyte and mass	MDL [µg⋅L⁻¹] incl. DF	Measured [µg⋅g⁻¹]	CRM consensus values [µg⋅g⁻¹]
¹³⁹ La as ¹³⁹ La. ¹⁶ O at m/z 155	0.001	26 ± 0.5	25 ± 1
¹⁴⁰ Ce as ¹⁴⁰ Ce. ¹⁶ O at m/z 156	0.001	55 ± 1	53 ± 2
¹⁴¹ Pr as ¹⁴¹ Pr. ¹⁶ O at m/z 157	0.001	7.0 ± 0.2	6.8 ± 0.3
¹⁴⁶ Nd as ¹⁴⁶ Nd. ¹⁶ O at m/z 162	0.002	30 ± 1	28 ± 2
¹⁴⁹ Sm as ¹⁴⁹ Sm. ¹⁶ O at m/z 165	0.001	6.9 ± 0.5	6.7 ± 0.3
¹⁵³ Eu	0.0003	2.1 ± 0.1	2.0 ± 0.1
¹⁵⁷ Gd as ¹⁵⁷ Gd. ¹⁶ O at m/z 173	0.001	7.1 ± 0.1	6.8 ± 0.3
¹⁵⁹ Tb as ¹⁵⁹ Tb. ¹⁶ O at m/z 175	0.001	1.10 ± 0.03	1.07 ± 0.04
¹⁶³ Dy as ¹⁶³ Dy. ¹⁶ O at m/z 179	0.001	7.1 ± 0.2	-
¹⁶⁵ Ho as ¹⁶⁵ Ho. ¹⁶ O at m/z 181	0.0003	1.45 ± 0.05	1.33 ± 0.06
¹⁶⁶ Er as ¹⁶⁶ Er. ¹⁶ O at m/z 182	0.0003	4.2 ± 0.3	-
¹⁶⁹ Tm as ¹⁶⁹ Tm. ¹⁶ O at m/z 185	0.0003	0.6 ± 0.1	0.54
¹⁷² Yb	0.001	3.7 ± 0.2	3.5 ± 0.2
¹⁷⁵ Lu as ¹⁷⁵ Lu. ¹⁶ O at m/z 191	0.0003	0.55 ± 0.02	0.51 ± 0.02

Excellent recovery with certified concentrations and sub ng-L⁻¹ detection limits for REE's enabling sensitive and reliable analysis at ultra-trace levels

Results – Other elements in SLRS-5 CRM

Analyte and mass (as m/z)	MDL [µg·L ⁻¹]	SLRS-5 [µg⋅L ⁻¹]	SLRS-5 CRM value [µg∙L ⁻¹]	Concentration range in water samples [µg·L ⁻¹]			
⁹ Be	0.006	0.004 ± 0.005	0.005*	0.003 - 0.07			
²³ Na	13.3	5,374 ± 98	5,380 ± 100	10,972 - 110,328			
²⁴ Mg	3	2,443 ± 110	2,540 ± 160	3,332 - 35,128			
²⁷ AI	0.3	50.1 ± 6.0	49.5 ± 5.0	0.002 - 0.2			
³⁹ K	2.1	822 ± 60	839 ± 36	2,216 - 19,681			
⁴⁴ Ca as ⁴⁴ Ca. ¹⁶ O at 60	12.9	10,060 ± 380	$10,500 \pm 400$	18,100 - 48,082			
⁵¹ V as ⁵¹ V. ¹⁶ O at 67	0.002	0.291 ± 0.020	0.317 ± 0.033	0.36 - 0.92			
⁵² Cr as ⁵² Cr. ¹⁶ O at 68	0.012	0.199 ± 0.021	0.208 ± 0.023	0.09 - 0.46			
⁵⁵ Mn	0.005	4.21 ± 0.28	4.33 ± 0.18	0.16 - 519.8			
⁵⁷ Fe	0.57	93.5 ± 2.8	91.2 ± 5.8	50.1 - 1,051			
⁶⁰ Ni	0.024	0.495 ± 0.038	0.476 ± 0.064	0.82 - 1.93			
⁶³ Cu	0.3	18.7 ± 1.8	17.4 ± 1.3	0.77 - 127.02			
⁶⁶ Zn	0.048	0.89 ± 0.018	0.845 ± 0.095	3.8 - 163.3			
⁷⁵ As as ⁷⁵ As. ¹⁶ O at 91	0.0038	0.389 ± 0.03	0.413 ± 0.039	0.06 - 1.08			
⁸⁰ Se as ⁸⁰ Se. ¹⁶ O at 96	0.0041	0.09 ± 0.02	-	0.04 - 0.12			
⁸⁹ Y as ⁸⁹ Y. ¹⁶ O at 105	0.0009	0.11 ± 0.006	-	0.01 - 0.77			
⁹⁸ Mo as ⁹⁸ Mo. ¹⁶ O at 114	0.0082	0.5 ± 0.1	0.5*	0.1 - 1.2			
¹⁰⁷ Ag	0.002	0.005 ± 0.001	-	0.004 - 0.019			
¹¹¹ Cd	0.0016	0.0069 ± 0.0012	0.0060 ± 0.0014	0.001 - 0.031			
¹²¹ Sb	0.0016	0.29 ± 0.02	0.3*	0.029 - 0.31			
²³⁸ U as ²³⁸ U. ¹⁶ O ₂ at 270	0.0003	0.100 ± 0.003	0.1*	0.014 - 0.596			

Excellent recovery with certified concentrations for all elements!

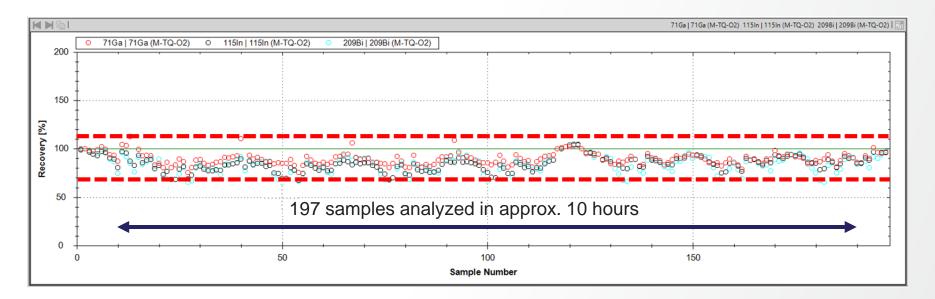
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⁵² Cr as ⁵² Cr. ¹⁶ O at 68	0.012	0.199 ± 0.021	0.208 ± 0.023	0.09 - 0.46			
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²³⁸ U as ²³⁸ U. ¹⁶ O ₂ at 270	0.0003	0.100 ± 0.003	0.1*	0.014 - 0.596			

Samples covered a broad range of matrix load (~ $20 \text{ mg} \cdot \text{L}^{-1}$ to > $200 \text{ mg} \cdot \text{L}^{-1}$)!

Robustness

• Typical parameters characterizing robustness: Internal standard and QC reoveries!



- Internal standard recovery well within 80-120% in a 10 hour long sequence
- Quality control samples (CCV) (every 20 samples) did read out with ± 3 %



Full intereference removal accomplished for all analytes

Good agreement between certified concentrations for both certified reference materials
 No false positive results observed for As and Se in BCR-2

Consistent internal standard and QC recovery throughout a 10 hour sequence

- Collected water samples were significnatly differnet in total matrix content and composition
- For example, the concentration for calcium varied between 11 and 110 mg·L⁻¹, up to 20-fold concentation differences for Fe and Ni, up to a 100-fold difference for Cu

Comparison of detection limits

⁹ Be	²³ Na	²⁴ Mg	²⁷ AI	³⁹ K	⁵¹ V	⁵² Cr	⁵⁵ Mn	⁶⁰ Ni	⁶³ Cu	⁶⁶ Zn	⁷⁵ As	Se	¹⁰⁷ Ag	¹¹¹ Cd	¹²¹ Sb	²⁰⁸ Pb	²³⁸ U
+++	0	0	0	0	++	+	++	0	0	+	+	+	++	+	0	0	++

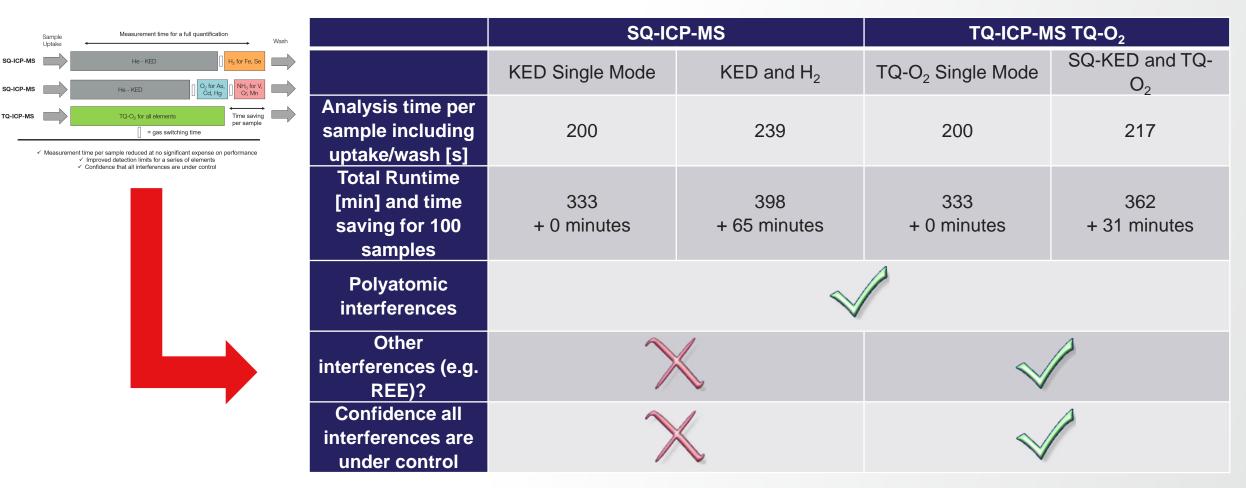
O Equivalent or sligtly higher DL

- + Detection Limit improvement factor > 2; ++ Detection Limit Improvement factor > 5; +++ Detection Limit Improvement factor > 10
- An improvement in the detection limit is most obviously observed for critical contaminnats, often combined with a better interferecne remvoval
- Typical major elements are not affected significantly



Application Note 44358 (iCAP RQ ICP-MS)

Let's have a look at throughput



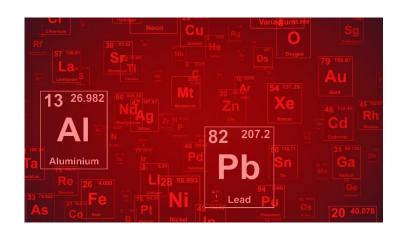
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- The data shown corresponds to a sequence containing 40 individual elements
- For 100 samples, a time saving of at least 30 minutes can be realized when using a single mode
- Compared to methods using additional gases, the time savings can be even more significant

SQ-ICP-MS

TQ-ICP-MS

Advantages of TQ-ICP-MS over SQ-ICP-MS



Better Interference Removal

- There are many elements that could be biased unexpectedly
- Existing methods can be combined with TQ modes where needed!



Better Sensitivity and LOD's

- New applications come with new challenges (i.e. interferences)
- Especially critical contaminants can benefit from a TQ-ICP-MS



Option to increase productivity

- Sample turnover times can be as fast as a single quad – or faster
- Method development can't be easier!

There are good reasons for TQ-ICP-MS in a busy laboratory

Summary and Conclusion

- There are many reasons why a TQ-ICP-MS should be found in a laboratory
 - Superior interference removal avoids unnecessary interruptions
 - Better detection limits and increased confidence
- TQ-ICP-MS is all but complicated
 - Straightforward method development using Reaction Finder Method Development Assistant
 - Easy install and cost-efficient operation with He and O₂ as collision/reaction gases only
- It has never been easier to future-proof
 - Similar and often faster turnover times can be achieved compared to SQ-ICP-MS methods
 - Increase productivity and performance







Download our TQ-ICP-MS toolkit including a Method Development Guide under www.thermofisher.com/tqtoolkit

New Resource

Guide for environmental sample analysis by ICP-MS

If your laboratory is

- Experiencing analytical challenges, inaccurate results, and sample reruns
- Seeking to streamline current methodologies and workflows
- Starting up or preparing for environmental sample analysis by ICP-MS

our eBook, "Guide for Environmental Sample Analysis by ICP-MS: Recommendations for Getting Started and Best Practices to Streamline Workflow," serves as a helpful resource

Topics include:

- Considerations and tips for selecting laboratory apparatus, equipment, reagents, and standard solutions
- Best practices for the entire elemental analysis workflow
- Best practices and tips to streamline sample and standard preparations
- Recommended pre-calibration routines and instrument inspections
- · General instrument maintenance and troubleshooting tips and tricks

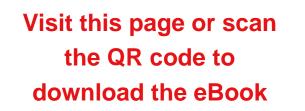
https://www.thermofisher.com/us/en/home/global/forms/ind ustrial/environmental-sample-analysis-by-icp-ms-ebook.html



Guide for environmental sample analysis by ICP-MS:

Recommendations for getting started and best practices to streamline workflow

by Sabrina Antonio





New Story - The Flint Community Lab

Turning The Taps On Without Fear

The Flint Community Lab was born from the Flint water crisis where tens of thousands of residents were exposed to dangerous levels of lead in their tap water. The consequences were devastating and to this day basic use of tap water is not possible. To restore trust in the water supply and maintain vigilance, an old school was converted to the Flint Community Lab where water samples collected from homes are tested for lead, copper, and other metal contaminants. Thermo Fisher Scientific assisted with this initiative and donated an <u>ICP-MS instrument</u> enabling high school and college chemistry students to learn about the technology and perform the testing.

To learn more and watch a video about this compelling story, please visit the following pages:

Webpage: <u>https://www.thermofisher.com/us/en/home/step-beyond/turning-the-taps-on-without-fear.html</u>

YouTube: https://www.youtube.com/watch?v=NWMhgRrPG11

www.thermofisher.com/flint-michigan

Turning The Taps On Without Fear

Going a step beyond means returning hope to the community of Flint, Michigan.



athing your ohild. Diniving a glass of water, Even brushing your teeth with tap water is no longer possible for the community of Flint, Michigan. "It can't be one," says Michael Harris, founder of The McKenzie Patrice-Croom Flint Community Lab.

2014, the oity of Fint failed to treat its water with corrosion inhibitors, resulting in lead leaching out of the water pipes and into the water that supplied the omes of tens of thousands of people. The consequences were devastating. Once lead enters your system, it stays in your blood for around two weeks, before septing into your bones and brain, where it can cause neurological disorders, behavioral issues in children, and sterilization and miscarriages in women. "What append in fint was a huge breakdown of trust," Candice Mushatt, head of the Fint Community Lab, explains. "We needed a way to restore the trust in our ster and we needed a way for our residents to be empowered."

And so, the Flint Community Lab was born.





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