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ICP-MS applications for spectral interference resolution

Maciel Santos Luz

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Instituto de Pesquisas Tecnológicas do Estado de São Paulo
S/A - IPT
Av. Prof. Almeida Prado, 532 | Cidade Universitária ou
Caixa Postal 0141 | CEP 01064-970
São Paulo | SP | Brasil | CEP 05508-901
Tel 11 3767 4374/4000 | Fax 11 3767-4099

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III WORKSHOP OF INORGANIC MASS SPECTROMETRY
I WORKSHOP ON TECHNOLOGY-CRITICAL ELEMENTS
June 18 – 22, 2023 • Rio Claro (SP) • Brazil

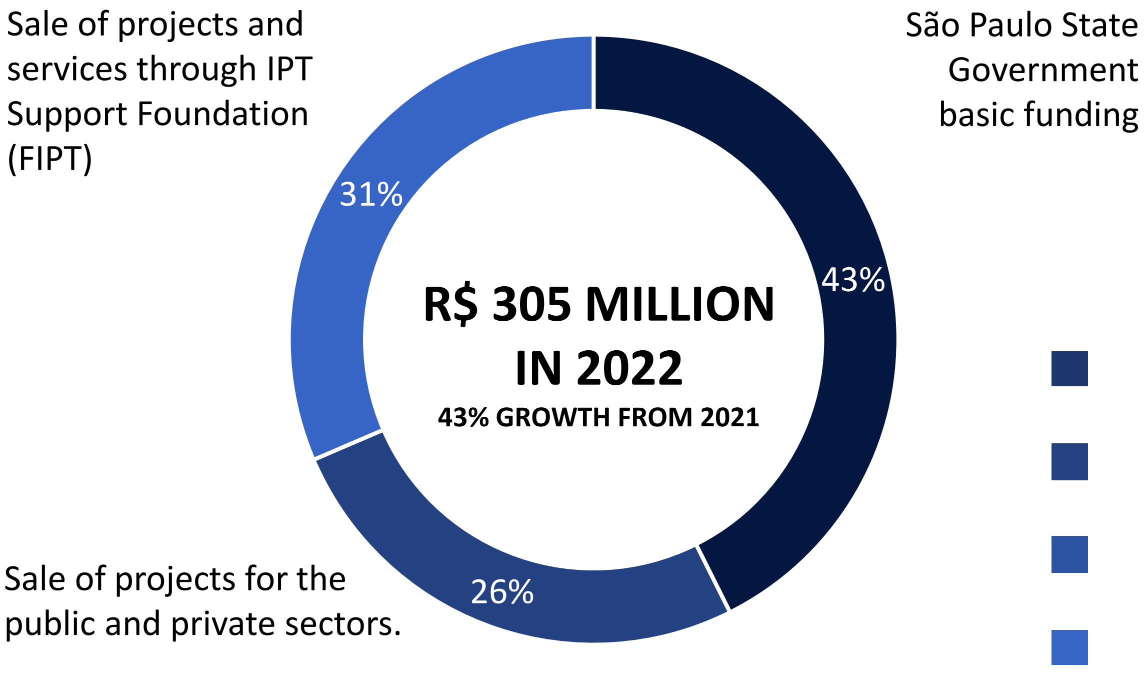
ICP-MS applications for spectral interference resolution

Maciel Santos Luz

WHO ARE WE ?

IPT PROVIDES TECHNICAL SOLUTIONS FOR INDUSTRY, GOVERNMENTS AND SOCIETY, ENABLING THEM TO OVERCOME THE CHALLENGES OF OUR TIME

INCOMES



OUR NUMBERS (2022)

- 123 YEARS OF CONTRIBUTIONS TO SOCIETY
- > 1,000 EMPLOYEES AND PARTNERS
- 41% REVENUE IN INNOVATION PROJECTS
- > 1,830 CUSTOMERS SERVED
- SATISFIED CUSTOMERS NPS 84 (LEVEL OF EXCELLENCE)
- > 19,900 TECHNICAL DOCUMENTS ISSUED
- > 2,000 TESTING AND ANALYSIS PROCEDURES IN THE PORTFOLIO

WHAT WE DO ?

RESEARCH, DEVELOPMENT AND INNOVATION

PRODUCTS AND PROCESSES

SOFTWARES

FROM THE BENCH TO THE PILOT

FUNDING

EMBRAPII

TESTS, TRIALS AND ANALYSIS

TECHNICAL ANALYSIS OF PRODUCTS AND MATERIALS

PRODUCT EVALUATION

PRODUCT CERTIFICATION

INSPECTION AND MONITORING

CONSTRUCTION AND SCTRUCTURES

MACHINERY AND EQUIPMENT

ACCREDITED INSPECTION BODY

METROLOGICAL DEVELOPMENT, MEASUREMENTS AND CALIBRATIONS

PROFICIENCY PROGRAMS

STANDARDS DEVELOPMENT

ADVANCED METROLOGY

CERTIFIED REFERENCE MATERIALS

METALS

CERAMIC

MINERAL

VISCOSITY

NORMAL SAND

TECNOLOGICAL EDUCATION

PROFESSIONAL MASTER

EXTENSION COURSES

COURSES ON DEMAND

BUSINESS UNITS

BIONANOMANUFACTURING
Processes, Chemistry, PPEs, Biotech, Nanotech, Microfabrication

CITIES, INFRASTRUCTURE AND ENVIRONMENT
Territorial planning, Sustainability, Risks, Civil works

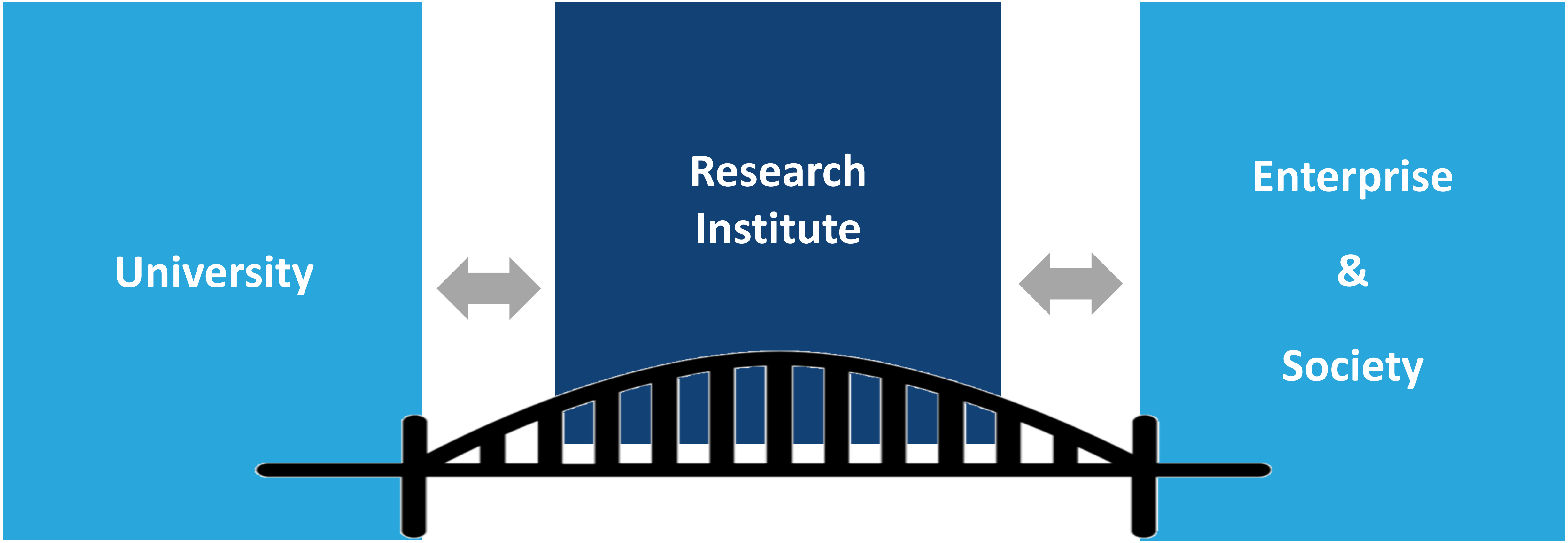
ENERGY
Generation, Infrastructure, Efficiency, Clean energy

BUILDING AND HOUSING
Confort, Performance, Safety, Materials, Sustainability

ADVANCED MATERIALS
Metallic, Polymeric, Composite, Cellulosic, Corrosion

DIGITAL TRANSFORMATION
IoT, Embedded Systems, Intelligent Transport Systems, AI, Analytics

METROLOGICAL AND REGULATORY TECHNOLOGIES
Mechanics, Electrical, Flow Measurement, Aerodynamics, Chemistry


University
Research Institute
Enterprise
&
Society

Advanced Materials
Laboratory of Metallurgical Processes

IPT on the news

A future for rare earths in Brazil

With the help of IPT, Brazilian companies can become part of the rare earths value chain and offer alternatives to comply with the world markets supply and demand

Brazil has the second largest reserve of rare earths in the world, 22 million tonnes, according to the U.S. Geological Survey, Mineral Commodity Summaries, January 2016. One of the sites has already resulted in the industrial scale production of a concentrate of rare earths, due to an important productive advantage: the rare earths are extracted from the tailings of the niobium produced by the Companhia Brasileira de Metalurgia e Mineração - CBMM, the largest niobium producer in the world and the first mining company to be certified by ISO 14.001. As a result, the extraction cost is deducted from the total cost of production.

Electrolytic cell adapted for didymium oxide reduction

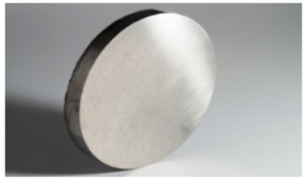
In 2015 IPT, CBMM and the Brazilian Industrial Research and Innovation Company (Embrapii) established a partnership for the process development of the neodymium-praseodymium alloy production from the oxides of those elements.

After obtaining the first batches of metallic "didymium" produced in Brazil, from rare earth oxides produced by CBMM, and after developing the didymium-iron-boron alloy, which are key steps in

METALLURGY

The first step towards supermagnets

IPT is developing a process to transform rare earth elements into metal for use in more powerful magnets for industry



A sample of metallic didymium developed at IPT

Eduardo Cesar

An important step was taken to allow Brazil to produce supermagnets in the future. The development of technology to produce didymium — a mixture of two metals and the precursor of alloys for magnets with a higher magnetic flux density — opens the way for the manufacture of this product, not yet made in Brazil. Didymium consists of two rare earth elements, praseodymium (Pr) and neodymium (Nd), from the lanthanide group. The high-power magnets are used, for example, in electric vehicle motors and power generators in wind turbines. The news announced in February 2016 is the result of a partnership between the Institute for Technological Research (IPT), the Companhia Brasileira de Metalurgia e Mineração (CBMM) and the Brazilian Agency for Industrial Research and Innovation (Embrapii), linked to the Ministry of Science, Technology and Innovation (MCTI).

In the project begun in 2014, the group of IPT researchers from the Metallurgical Processes Laboratory, led by metallurgical engineer João Batista Ferreira Neto, developed technology to transform didymium oxide, a coffee-colored power, into ingots of pure metal. "We developed the reduction stage, which means transforming the oxide into metal by removing the oxygen. In order to do this, we assembled reactors that operate at 1,200 degrees Celsius (°C) and produce bars of metallic didymium. In a subsequent stage — for which we also intend to develop technology — this material will be used to produce a metallic alloy of didymium, iron and boron for later manufacture of the supermagnet," explains Ferreira Neto. The project to develop metallic didymium cost R$9 million, with R$3 million from the CBMM, R$3 million from Embrapii, and IPT's contribution consisting of equipment, infrastructure and the salaries of seven researchers.

NEWS

Brazil's CBMM, IPT rare earth magnet tech push advances

Bnamericas

Published: Friday, April 28, 2017

Market Prices ... | Metals proces... | Rare earth ele... | Studies | Niobium

An R&D partnership b
CBMM (pictured) and
institute IPT has ente

The two entities are
and processes neede
the processing of rar

Metallic neodymium is employed in the manufacture of mag
memory for quantum computers. But no Brazilian company
magnets, local paper Folha de S. Paulo reported.

The joint initiative was launched by the two entities in Nover
the reduction of neodymium oxide into pure metal.

Now, CBMM and IPT are developing an alloy comprising ne
the key ingredients in the magnets.

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INVESTMENT PROMOTION AGENCY

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WHY SÃO PAULO ▾ | BUSINESS SECTORS ▾ | INVEST IN ▾ | EXPORT ▾ | NEWS ▾ | EVENTS ▾

Latest News > Brazil begins developing rare-earth alloys

NEWS

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02/05/2016 14h15

Brazil begins developing rare-earth alloys

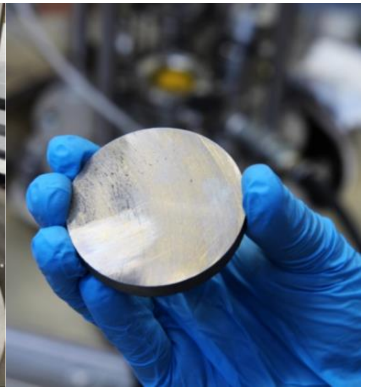
Valor International

You've probably never heard of neodymium, but the metal is the basis of supermagnets used in a variety of products – from personal computers to electric motors, generators and wind turbines. Three times as powerful as common magnets but cheaper to produce, they've become an almost exclusive Chinese export since the country concentrates the biggest global reserves of rare earths, a group of 17 minerals that includes neodymium. But Brazil has just taken an important step toward nationalizing supermagnet production and perhaps turning into a global heavyweight.

The Institute of Technological Research (IPT) in São Paulo has produced in a lab the first 100 grams of a crucial metallic alloy for supermagnet manufacturing. A compound of neodymium and another lanthanide metal, praseodymium, the alloy is known as metallic didymium. The innovation stems from a partnership with Companhia Brasileira de Metalurgia e Mineração (CBMM), which realized it was wasting a business opportunity.

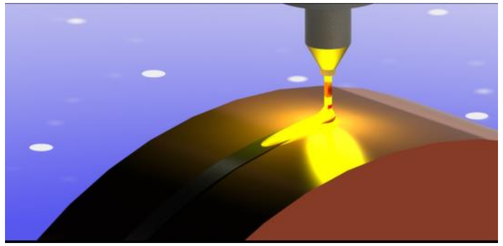
Rare earth production chain

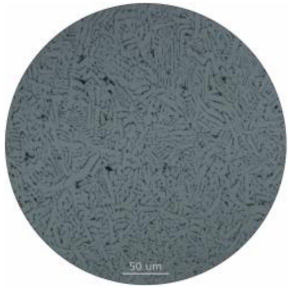
Electrolytic cell



Nd,Pr – 99.7 % purity

Strip casting





NdFeB alloy strips

CBMM Niobium

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WEG

- Nd and Pr (didymium) oxide
- Obtaining the metal (Nd,Pr)
- (Nd,Pr)FeB alloy preparation
- Hydrogenation
- (Nd,Pr)FeB powder
- Orientation
- Compression
- Sintering
- Heat treatment
- Corrosion coating
- Application

EMBRAPII UE - IPT MATERIAIS - ALTO DESEMPENHO

Finep INOVAÇÃO E PESQUISA EMPRESA PÚBLICA DO MCTI

BNDES

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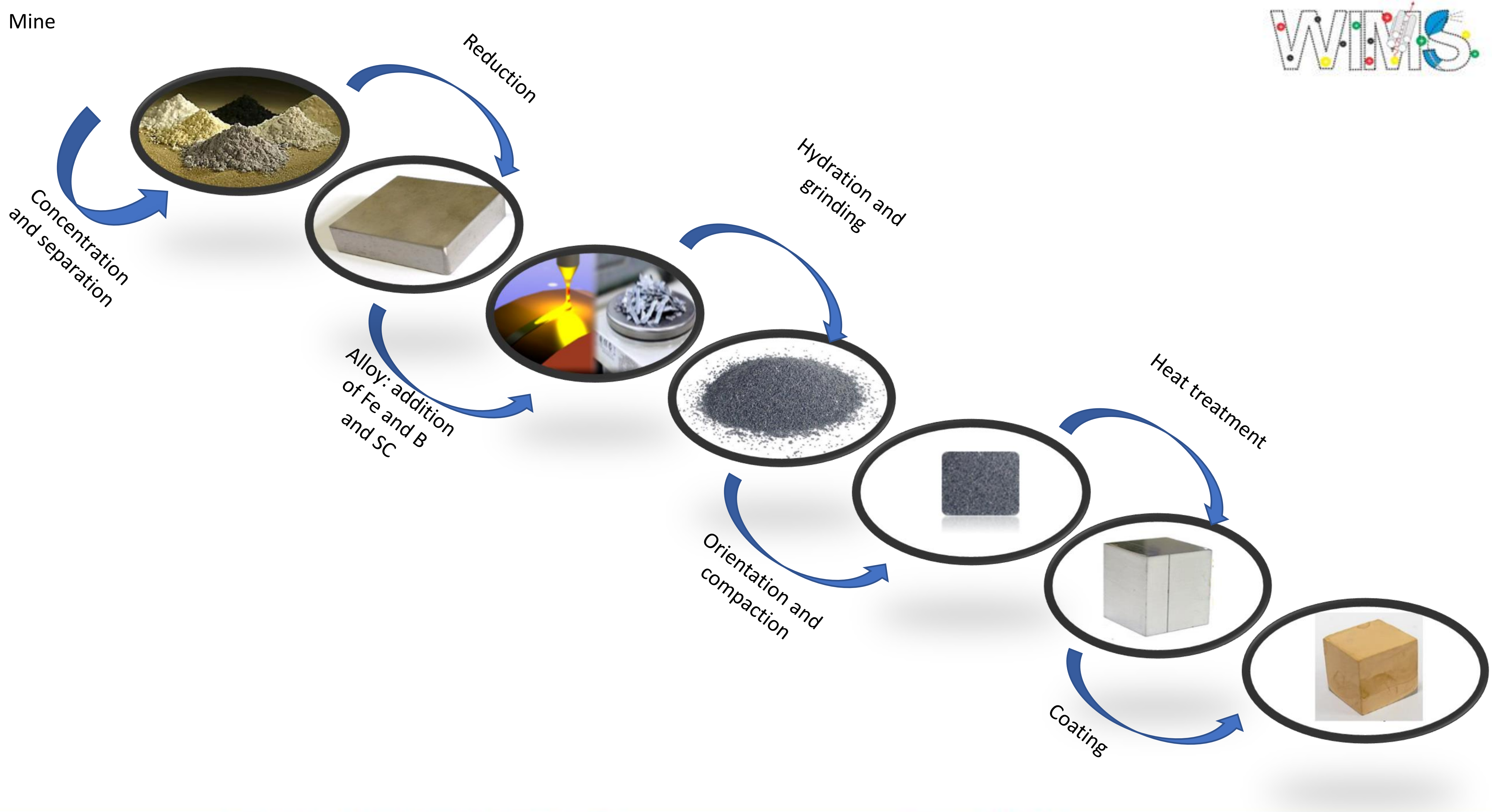
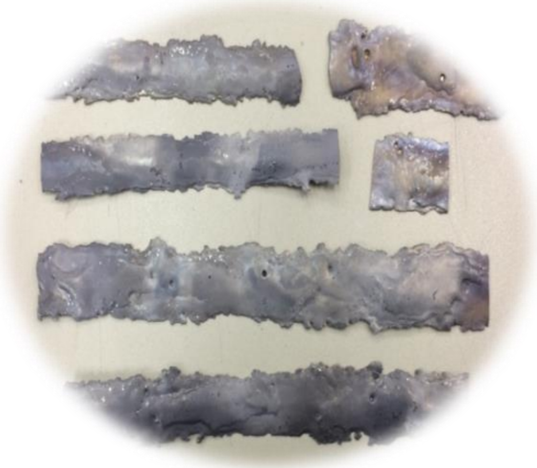

Mine
Concentration and separation
Reduction
Alloy: addition of Fe and B and SC
Hydration and grinding
Orientation and compaction
Heat treatment
Coating

Samples

Nd_2O_3



Nd-Fe-B

Nd^0

How to analyze them?

Spectral interferences in ICP OES

Analito	**Sc**		**Y**							
λ (nm)	337,215		371,030		324,228			224,306		
λ Nd (nm)	337,235	337,200	371,040	371,010	324,245	324,238	324,208	224,368	224,281	224,235

Analito	**La**										
λ (nm)	333,749				412,323		387,164		442,990		
λ Nd (nm)	333,800	333,760	333,725	333,700	412,333	412,300	387,194	387,134	443,058	443,018	442,932

Analito	**Ce**							
λ (nm)	413,380			413,765		446,021	429,667	
λ Nd (nm)	413,422	413,400	413,336	413,799	413,746	446,014	429,677	429,636

Analito	**Pr**										
λ (nm)	414,313				422,535			411,846			
λ Nd (nm)	414,356	414,325	414,300	414,271	422,556	422,520	422,485	411,900	411,836	411,816	411,779

Analito	**Sm**					**Eu**					**Gd**	
λ (nm)	442,434					272,778		443,556			342,247	
λ Nd (nm)	442,479	442,456	442,434	442,395	442,375	272,818	272,758	443,605	443,560	443,509	342,267	342,230

Spectral interferences in ICP OES

Analito	Tb									Dy		
λ (nm)	350,917						367,635			340,780		
λ Nd (nm)	350,950	350,924	350,896	350,950	350,924	350,896	367,660	367,635	367,589	340,802	340,755	340,738

Analito	Ho								Er					
λ (nm)	345,600				339,898				337,271			349,910		
λ Nd (nm)	345,617	345,600	345,577	345,558	339,941	339,919	339,888	339,865	337,309	332,870	337,230	349,955	349,908	349,955

Analito	Tm								
λ (nm)	313,126			336,261			250,962		
λ Nd (nm)	313,117	313,110	313,100	336,315	336,270	336,226	250,962	250,900	250,875

Analito	Yb									Lu			
λ (nm)	369,419				289,138					261,542			
λ Nd (nm)	369,481	369,439	369,409	369,359	289,170	289,145	289,127	289,110	289,084	261,602	261,562	261,527	261,482

ICP-MS

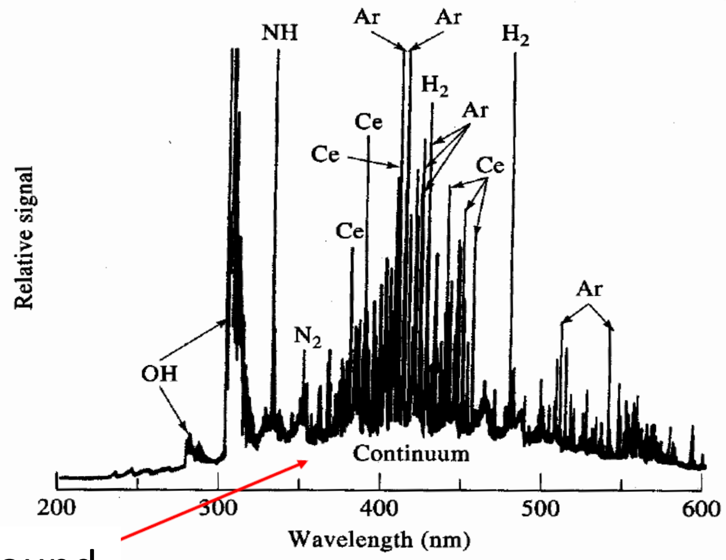
ICP OES Vs. ICP-MS

Elements	Emission lines	Isotopes
Li	30	2
Cs	645	1
Mg	173	3
Ca	662	6
Cr	2277	4
Fe	4757	4
Ce	5755	4

Fonte: F. J. Holler, D. A. Skoog & T. A. Neiman. Principles of Instrumental Analysis

The spectra of Ce

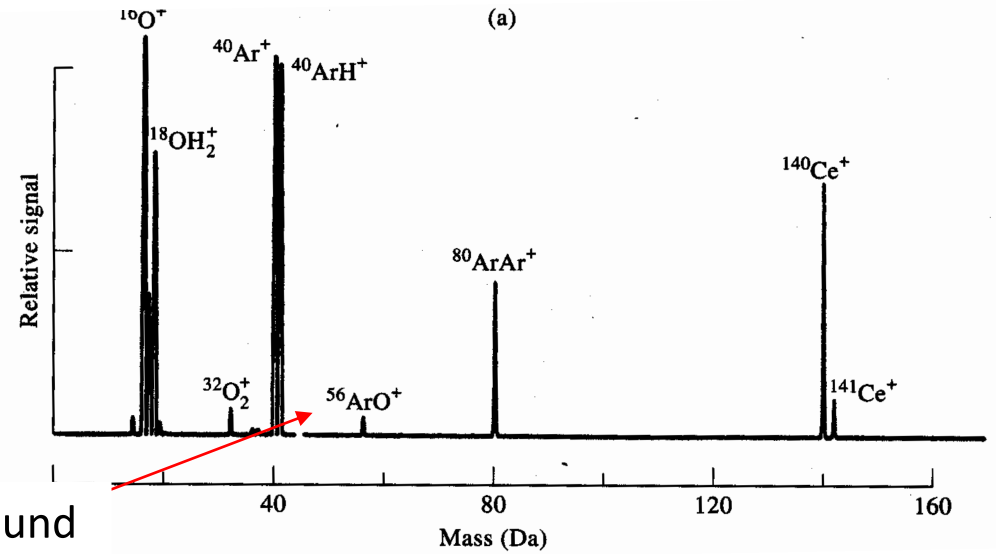
Light emission



Background

ICP OES

Mass-to-charge ratio



Background

ICP-MS

Fonte: F. J. Holler, D. A. Skoog & T. A. Neiman. Principles of Instrumental Analysis

Interferences in ICP-MS

Interference	m/z	Analyte	Isotopic abundance (%)	Interfering
Isobaric overlap	40	$^{40}Ca^{+}$	96,9	$^{40}Ar^{+}$
	50	$^{50}Ti^{+}$	5,4	$^{50}Cr^{+}$, $^{50}V^{+}$
Polytatomic ions	75	$^{75}As^{+}$	100	$^{40}Ar^{35}Cl^{+}$
	80	$^{80}Se^{+}$	49,6	$^{40}Ar^{40}Ar^{+}$
Doubly charged ion	69	$^{69}Ga^{+}$	60,1	$^{138}Ba^{2+}$
	70	$^{70}Ge^{+}$	21,2	$^{140}Ce^{2+}$

How to deal with interference?

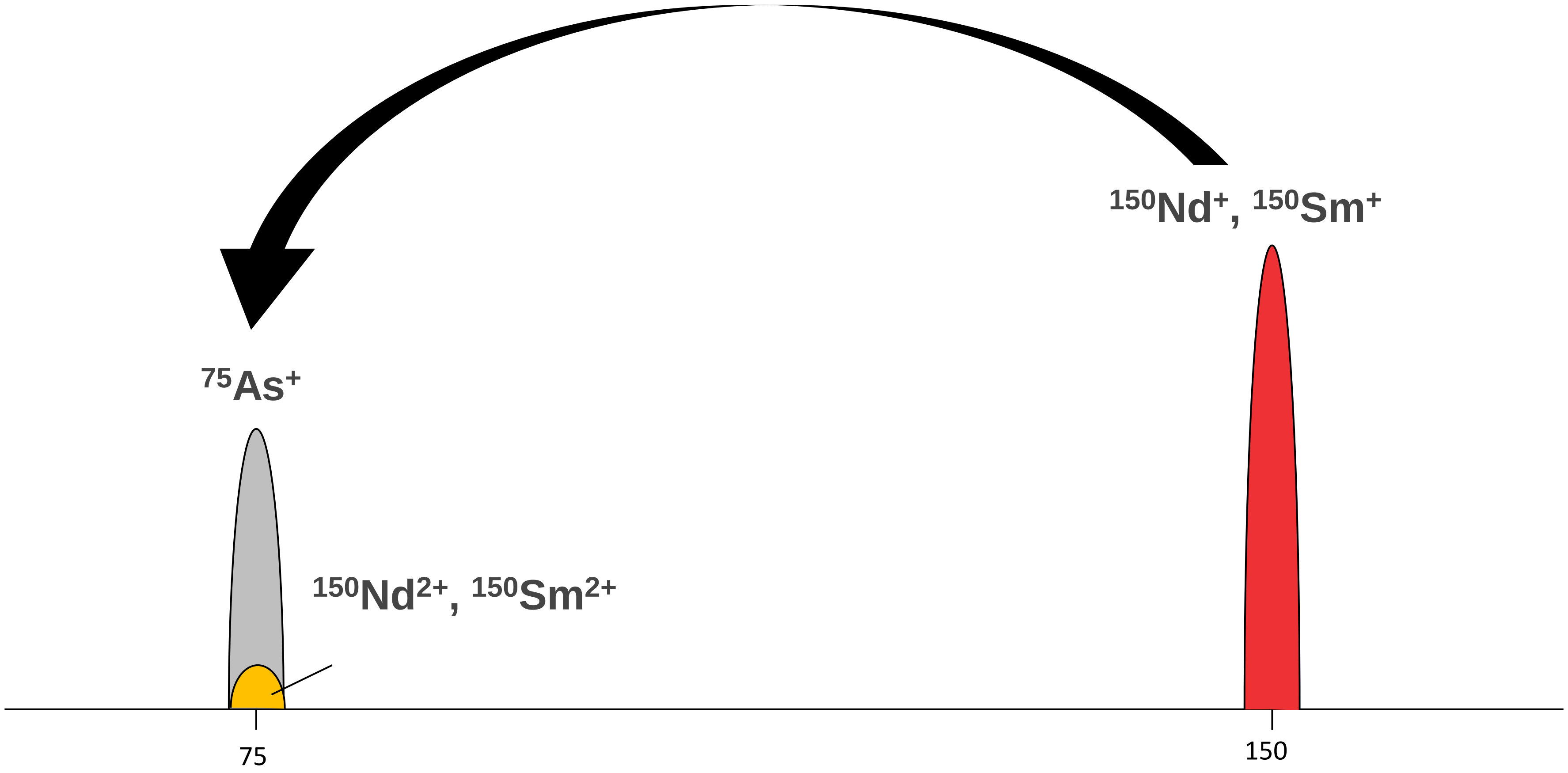
- Alternative sample introduction techniques
  - Aerosol desolvation to minimize the population of precursor ions at the source of ions

- Cold plasma
  - Reduces plasma power to reduce the amount of Ar ionization

- Mathematical correction equations
  - They measure the isotope of interest, an interference-free isotope, a polyatomic isotope and mathematically decovolute it to reach the Interference-free value

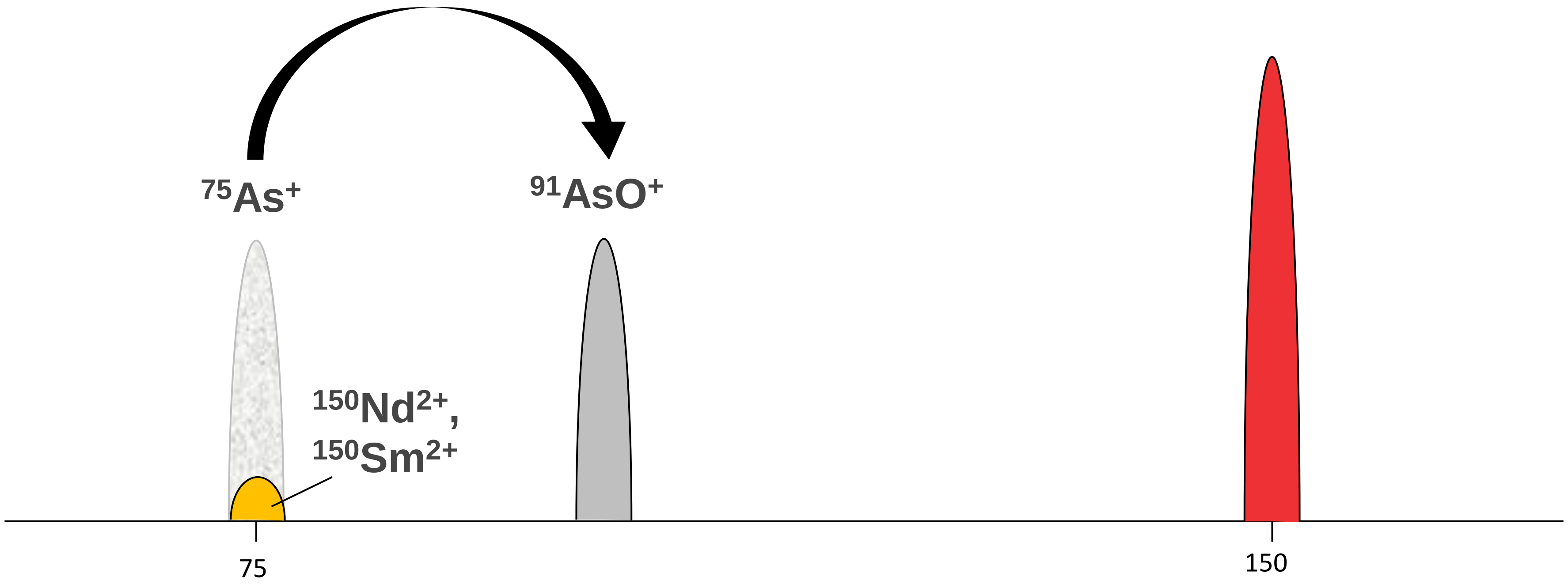
- Collision and reaction cells

As determination in REE matrix



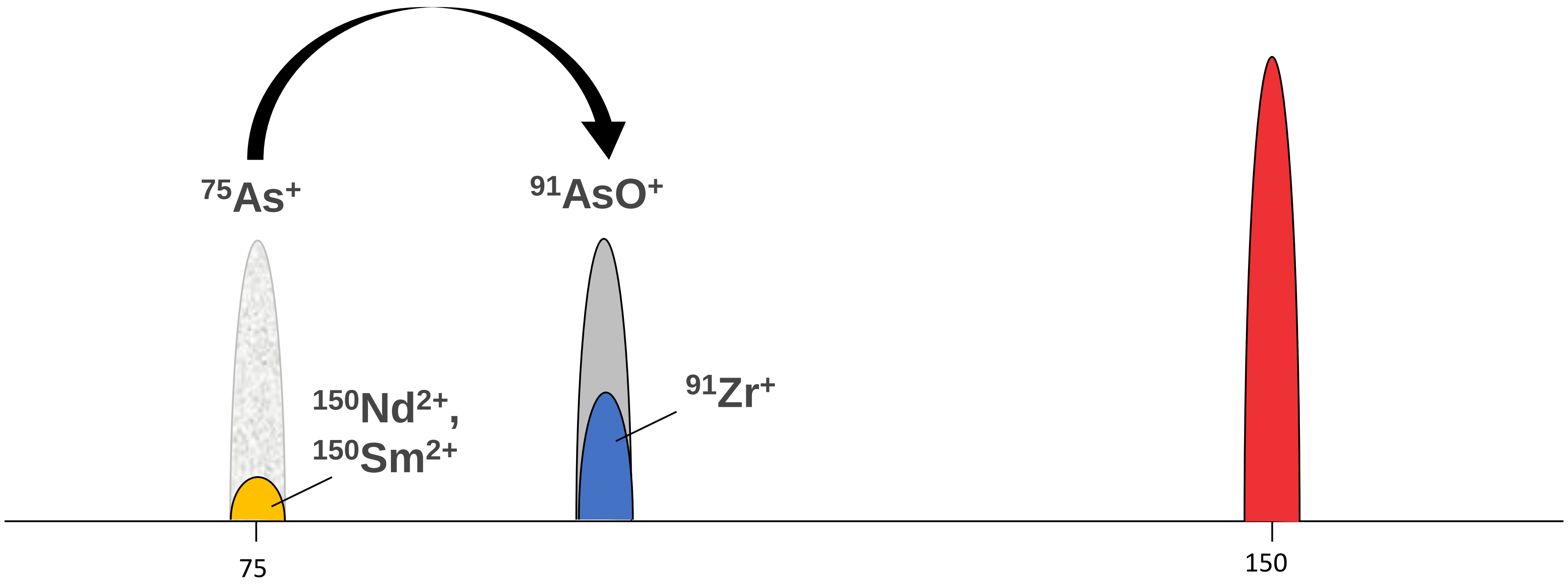
Fonte: Thermo

As determination in REE matrix



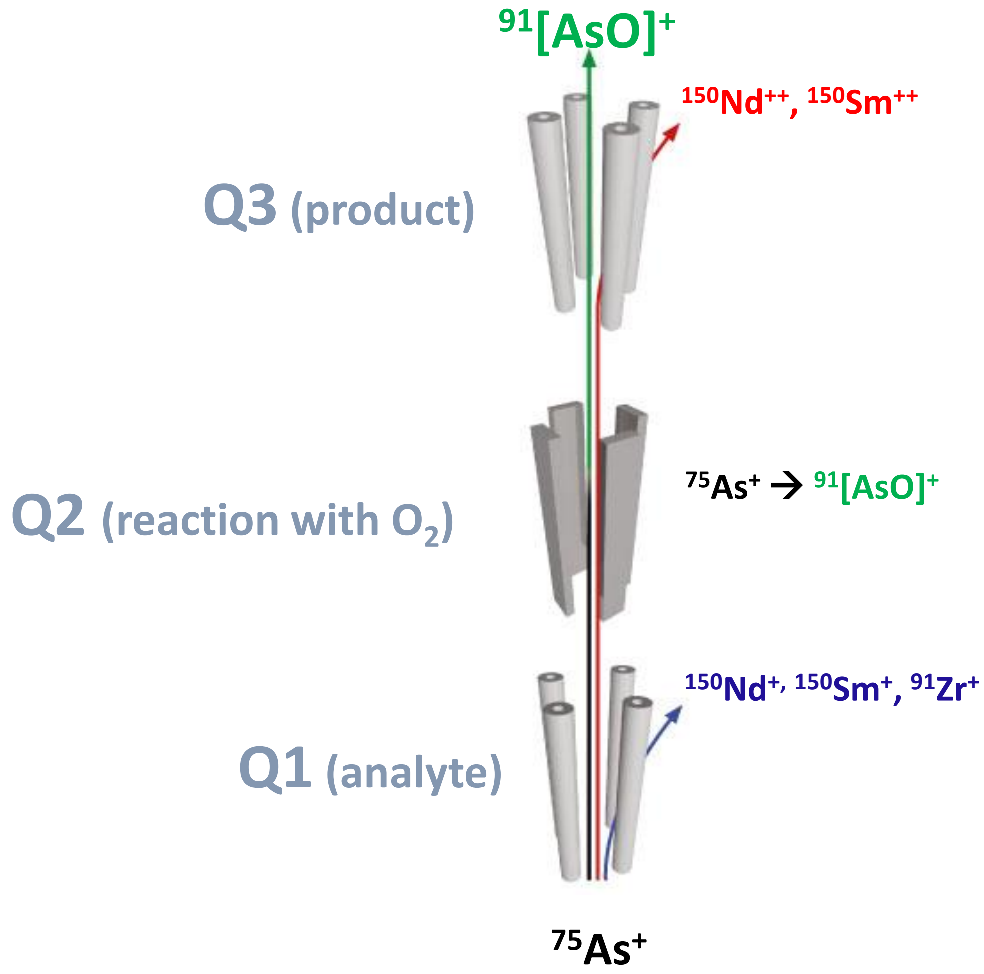
Fonte: Thermo

As determination in REE matrix



Fonte: Thermo

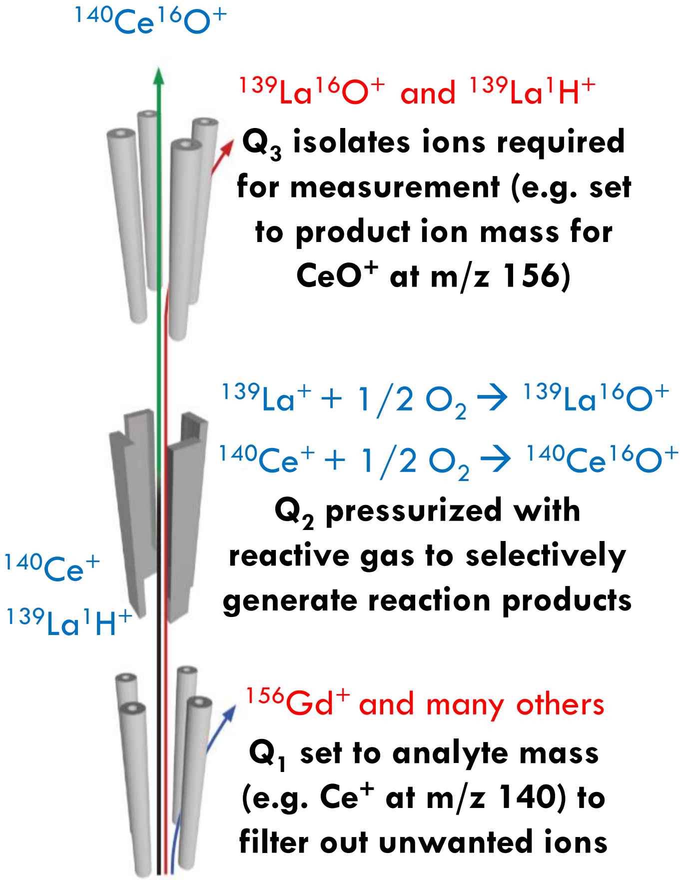
As determination in REE matrix



Fonte: Thermo

REEs determination in water CRMs

- →TQ Technology promotes interference removal and maintains high detectability
- →BEC and SNR were improved compared to SQ mode
- →Low concentrations achieved (linear range: 0.01 - 1000 ppt for most REE)



	Concentrations ($ng\ L^{-1}$)			
	NRC		NIST	SCP EnviroMAT
Element	AQUA-1	SLRS-6	1640A	EU-L
Sc	157 ± 4	171 ± 7	196 ± 5	141 ± 8
Y	27 ± 2	141 ± 6	47 ± 6	72 ± 6
La	27 ± 3	248 ± 4	< 0.01	85 ± 10
Ce	11 ± 4	222 ± 15	< 0.01	< 0.01
Sm	4.5 ± 0.5	34 ± 4	3.9 ± 0.8	0.17 ± 0.03
Eu	2.2 ± 0.2	7.1 ± 1.4	21 ± 4	161 ± 5
Gd	2.9 ± 0.6	23 ± 4	<0.01	0.33 ± 0.11
Tb	0.27 ± 0.07	2.5 ± 0.3	<0.01	0.09 ± 0.01
Dy	0.54 ± 0.04	14.5 ± 1.3	<0.01	< 0.01
Ho	0.06 ± 0.01	0.44 ± 0.05	0.07 ± 0.04	0.02 ± 0.01
Er	1.52 ± 0.12	9.2 ± 0.8	7.7 ± 1.7	0.19 ± 0.05
Tm	0.24 ± 0.05	1.2 ± 0.2	1.8 ± 0.3	0.08 ± 0.01
Yb	1.7 ± 0.3	8.7 ± 0.8	21 ± 2	0.44 ± 0.11
Lu	0.31 ± 0.08	1.3 ± 0.2	4.8 ± 0.9	0.25 ± 0.07

Spike and Recovery test with Tap Water (ppt level)	94-108%

Ti nanoparticles for MRI contrast agent

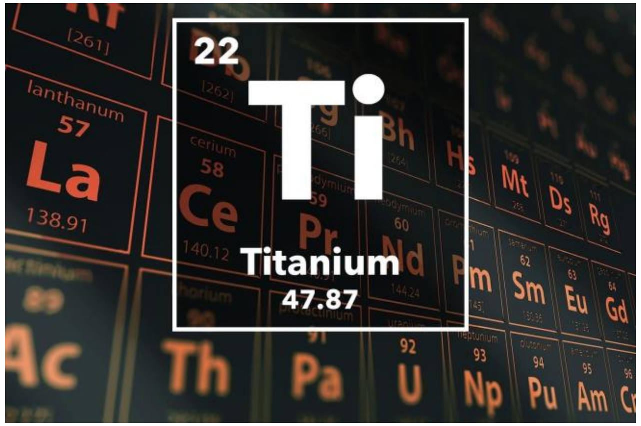
MRI: Magnetic Resonance Imaging

ICP-MS (Ti in biological samples)

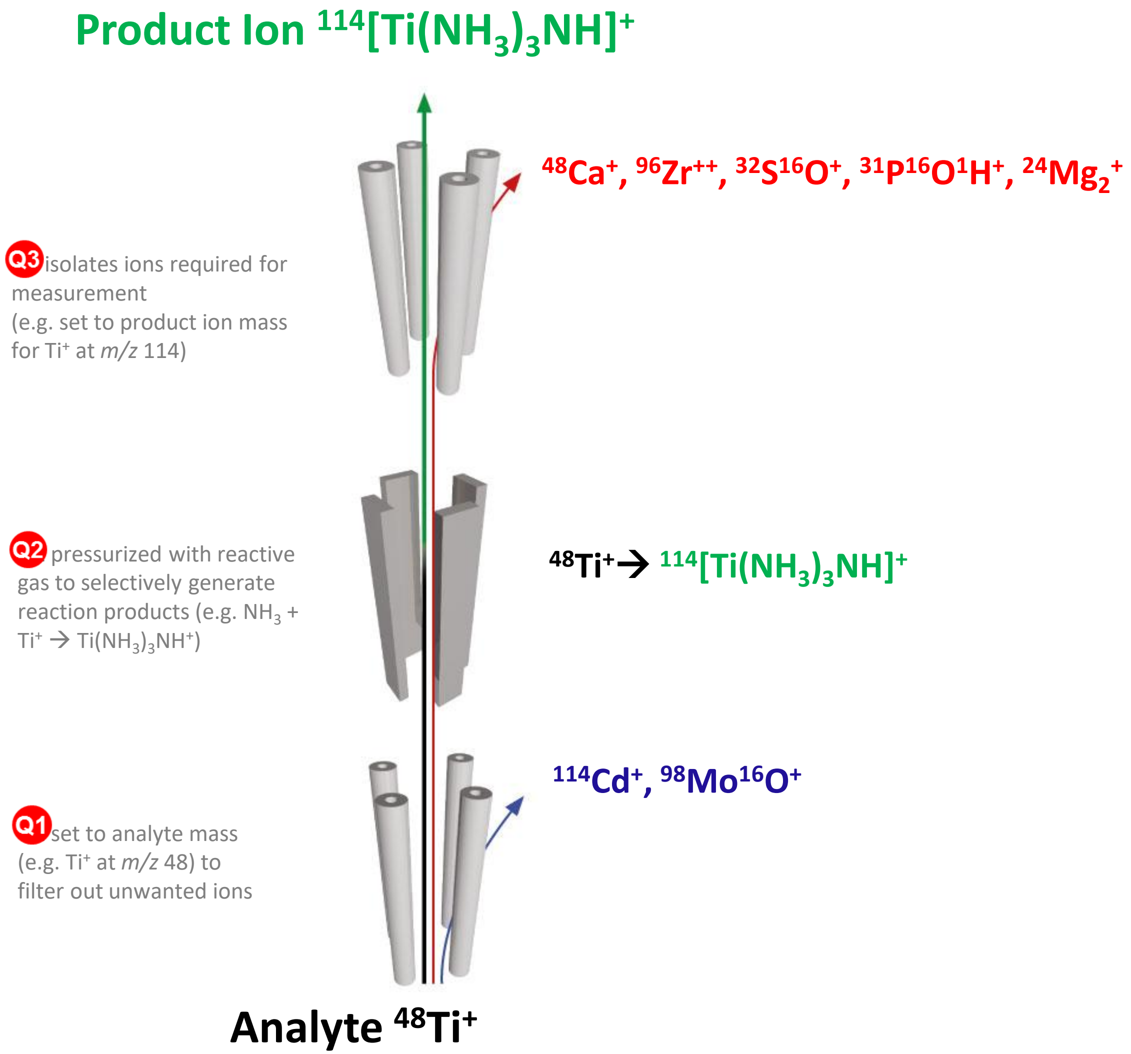
- $^{48}Ti^{+}$ (most abundant isotope)
- $^{48}Ca^{+}$, $^{96}Zr^{++}$, $^{32}S^{16}O^{+}$, $^{31}P^{16}O^{1}H^{+}$, $^{24}Mg_2^{+}$

No reaction

- $^{114}[Ti(NH_3)_3NH]^{+}$ Reaction with ammonia
- $^{114}Cd^{+}$, $^{98}Mo^{16}O^{+}$

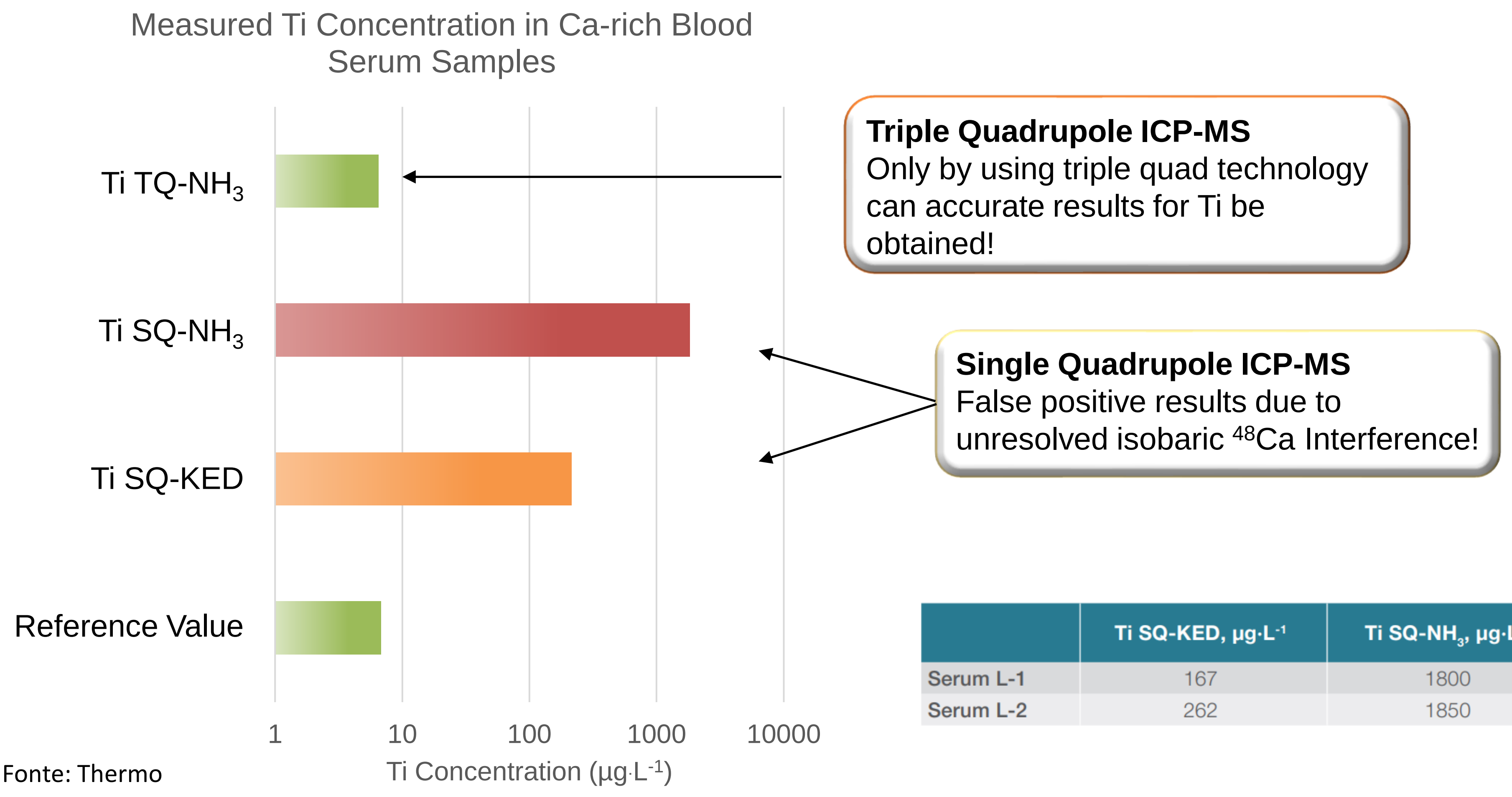


Ti determination in biological matrices



Fonte: Thermo

Ti determination in biological matrices



Fonte: Thermo

	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

Ti determination in biological matrices

	Spike and Recovery test (%)		
Spike concentration	**1 (µg/kg)**	**10 (µg/kg)**	**50 (µg/kg)**
Spleen	94	104	106
Spleen	81	114	111
Brain	89	106	115
Brain	83	116	111
Liver	74	108	109
Liver	87	107	105
Lung	115	126	124
Lung	98	112	119
Kidney	112	122	119
Kidney	108	120	114

* digested in microwave oven system

Ti determination in biological matrices

	Spike and Recovery test (%)			
Spike concentration	**0.5 (µg/L)**	**1 (µg/L)**	**5 (µg/L)**	**10 (µg/L)**
Urine	111	106	107	108
Urine	114	109	106	107

- Installed inside an ISO 8 cleanroom
- Equipped with all reaction (O_2, H_2 and NH_3) and collision gases (He)
- Nanoparticles analysis system
- Analysis in hydrofluoric acid medium

IPT

Location

Av. Prof. Almeida Prado, 532 - Cidade Universitária
05508-901 - São Paulo - SP

ipt

Thank you for your attention

Maciel Santos Luz

macielluz@ipt.br

WIMS

analítica 30 anos

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