ICP-MS measurements of enriched ⁸²Se samples for the LUCIFER experiment

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- Thanks a lot!
- It was a great experience!

Project objective

LUCIFER – is an experiment for the search of neutrinoless double beta decay with the help of scintillating bolometers (e.g. ZnSe).

Two samples of enriched ⁸²Se material were sent to the LNGS Chemistry laboratory for **ICP-MS analysis of contamination** by the elements which are important for crystal growth and scintillation properties of the detectors.

Besides that we were able to check the **isotopic composition** of enriched and natural selenium.

THERMO Finnigan ELEMENT2



J. S. Becker, Inorganic mass spectrometry: principles and applications, Wiley, 2007

THERMO Finnigan ELEMENT2



Schematic of the ELEMENT2 ICP-MS



cooling gas \longrightarrow

auxiliary gas —

load coil

Skimmer cone

Sampling cone

plasma

spray chamber (Ar + sample)





Interference

Interference is a masking of the peak of interest by another mono/polyatomic ions peak with close mass-to-charge ratio.

Solution: Resolution R = m / Δm (= const)

 $\Delta m = FW(5\%)M$

Low res: **R = 300**

Medium res: **R = 4000**

High res: **R = 10000**

Low resolution (R=300)

🔺 Interfe	rences : basic			
A				? 📘
Mass	Formula	Relative Intensity - 0 1 10 100		
55.45209	111Cd++			
55.93494	Fe			
55.95138	112Cd++			
55.95241	112Sn++			
55.95729	40Ar 160			
55.95750	40Ca16O			
55.95999	20Ne36Ar			
55.96284	39K17O			
56.45203	113ln++			
56.45220	113Cd++			
		-		
			55.83669	56.06109
			Mass Resolution	Normalize
			© Select © 10000 ○ 4000 ④ 300 ○ Quad	• Interference
			30000 Calculate	1
			Select resolution Calculate elemental formula from mass	Close

Medium resolution (R=4000)

🔺 Interfe	rences : basic			
A			?	R
Mass	Formula	Relative Intensity 0 1 10 100		
55.45209	111Cd++			
55.93494	Fe			
55.95138	112Cd++			
55.95241	112Sn++			
55.95729	40Ar 160			
55.95750	40Ca16O			
55.95999	20Ne36Ar			
55.96284	39K17O			
56.45203	113in++			
56.45220	113Cd++			
			55.92020	55.97758
			Mass Resolution	nalize lement
			© Select © 10000 € 4000 © 300 © Quad ● I	nterference
			30000 Calculate	1
			Select resolution	Close

High resolution (R=10000)

🔺 Interfe	rences : basic			
A				? 📘
Mass	Formula	Relative Intensity - 0 1 10 100		
55.45209	111Cd++			
55.93494	Fe			
55.95138	112Cd++			
55.95241	112Sn++			
55.95729	40Ar 160			
55.95750	40Ca16O			
55.95999	20Ne36Ar			
55.96284	39K17O			
56.45203	113in++			
56.45220	113Cd++	Γ		
			55.92905	55.96873
			Mass Resolution	Normalize C Element
			○ Select ● 10000 ○ 4000 ○ 300 ○ Quad	Interference
			30000 Calculate Select resolution Calculate elemental formula from mass	Close

Enriched ⁸²Se samples

Sample	Sample preparation	Dilution
Sample #1	m = 65 mg, dissolved in 1 ml HNO ₃ up to 10 ml, diluted 12 times with UP water	1846
Sample #2	m = 99 mg, dissolved in 1 ml HNO ₃ up to 10 ml, diluted 18 times with UP water	1818







Contamination of samples

	Sample #1,	Sample #2,
Isotope	ppb	ppb
Mg24(LR)	< 90	< 90
Mn55(LR)	< 20	< 20
Co59(LR)	< 20	< 20
Cu63(LR)	< 27	18
Cd110(LR)	< 20	< 20
W182(LR)	33	18
Pb206(LR)	< 40	< 40
Bi209(LR)	< 1	< 1
Th232(LR)	< 1	< 1
U238(LR)	< 2	< 2
Na23(LR)	< 1000	3300
Sb121(LR)	< 2	< 2
Hg202(LR)	< 2	< 3
Te125(LR)	3300	5400
Mo95(LR)	24	14
Sm152(LR)	< 2	< 2

	Sample #1,	Sample #2,
Isotope	ppb	ppb
Al27(MR)	< 1800	< 1800
Ca44(MR)	< 7000	< 7000
Cr52(MR)	< 20	< 20
Fe56(MR)	< 500	< 500
Si28(MR)	14000	< 10000
V51(MR)	< 90	< 90
S32(MR)	185000	180000
Ni58(MR)	< 100	< 100
As75(MR)	< 90	< 50
Co59(MR)	< 10	< 10
K39(HR)	< 1800	< 1800

The concentrations refers to the solid samples; the uncertainty is about 5%.

Isotopic composition

Se Isotope	Ref. Nat., %	Exp. Nat., %	Exp. Enr., %
74	0.89	0.72 ± 0.02	< 0.001
76	9.37	8.66 ± 0.26	< 0.08
77	7.63	6.74 ± 0.20	< 0.004
78	23.77	23.31 ± 0.70	< 0.02
80	49.61	50.94 ± 1.53	4.8 ± 1.4
82	8.72	9.63 ± 0.29	95.1 ± 2.9

Summary

- Within this project I got in touch with ICP-MS and different chemical research tools
- The contamination of selenium samples was studied and results were sent to the LUCIFER collaboration
- Isotopic composition was measured and compared with natural selenium. The values are compatible with previous measurements.
- ICP-MS is often a necessary technique for low-background experiments and the knowledge I acquired I find very useful

BACKUP SLIDES

Calibration: ³⁹K (HR)



Natural Se sample



Enriched ⁸²Se sample

🛃 [1] Spectrum No.1 👘 [-	48.073 sec]:5E82C.D	/ Tune #1 [Count] [Li	inear]						
1.0E6	7								
5.OE5									
						ul			
m/z->	68	70 72	2 74	76	78	, <u> </u> ,, 80	82	84	86



Challenge 2 - background

Presence of the isotope of interest in the blank sample

Blank sample spectrum: ⁵⁶Fe



Test sample spectrum: 56Fe







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