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Optimizing the chemical separation procedure of ^{79}Se for its application in decommissioning activities

TRISKEM
Expertise in Separation Chemistry

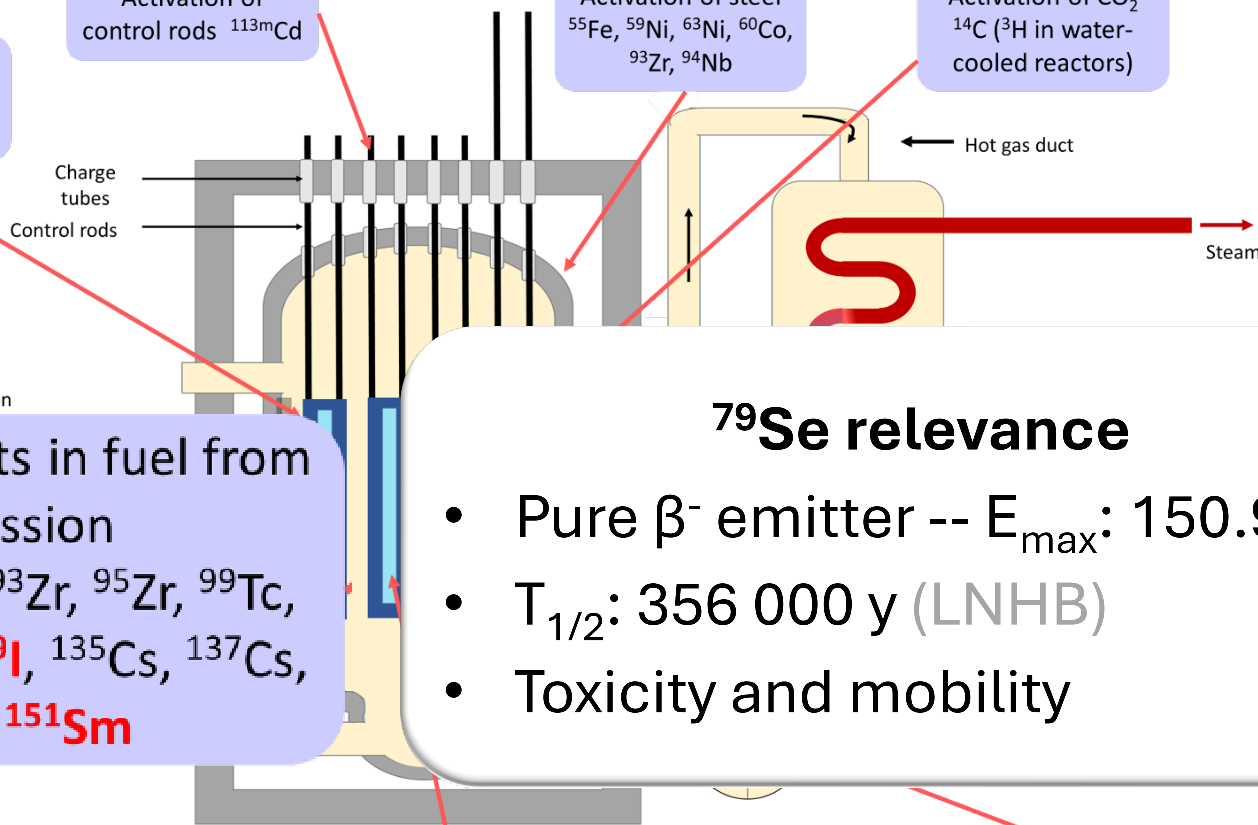
Radiological waste characterization

Activation graphite
^{14}C and **^{36}Cl** (from trace Cl impurities)

Activation of control rods ^{113m}Cd

Activation of steel
^{55}Fe, ^{59}Ni, ^{63}Ni, ^{60}Co, ^{93}Zr, ^{94}Nb

Activation of CO_2
^{14}C (^{3}H in water-cooled reactors)



Fission products in fuel from ^{235}U fission
^{3}H, **^{79}Se**, ^{90}Sr, ^{93}Zr, ^{95}Zr, ^{99}Tc, ^{107}Pd, ^{126}Sn, **^{129}I**, ^{135}Cs, ^{137}Cs, **^{147}Pm**, **^{151}Sm**

^{79}Se relevance

- Pure β^- emitter -- E_{max}: 150.9 keV
- $T_{1/2}$: 356 000 y (LNHB)
- Toxicity and mobility

Actinides in fuel from neutron capture by U
^{236}U, ^{237}Np, ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, ^{241}Am

Activation of concrete bioshield
^{3}H, ^{152}Eu, **^{41}Ca**, **^{151}Sm**

Based on (Warwick et al., 2022)

Challenges for ^{79}Se determination

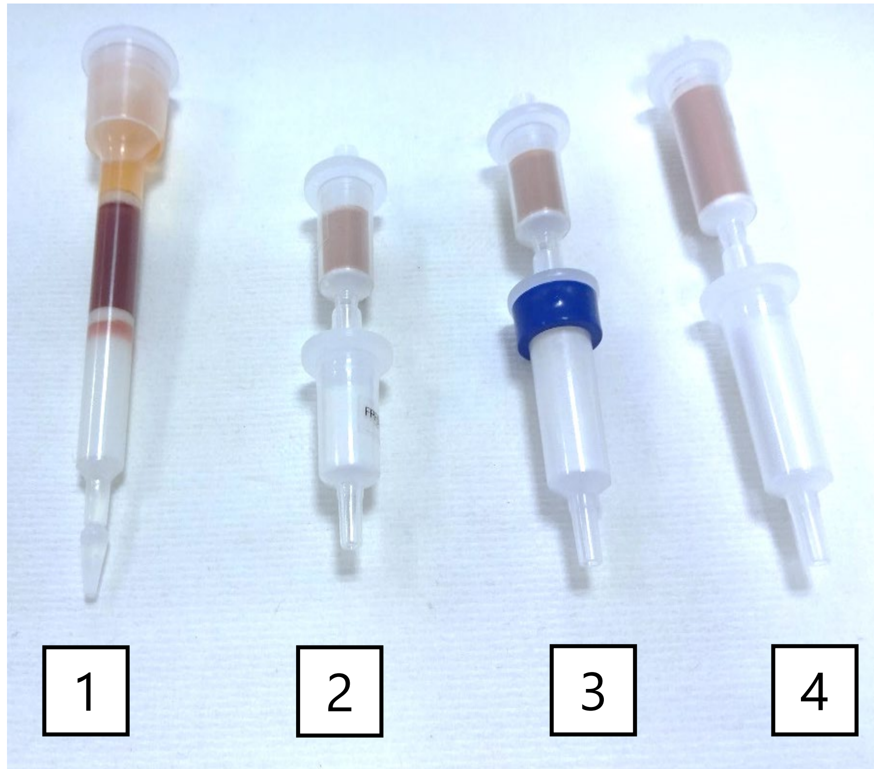
Development of a **new resin-based** material for the concentration and chemical separation of Se

On the determination of ^{79}Se

Se resin-based material

New SE Resin

TRISKEM



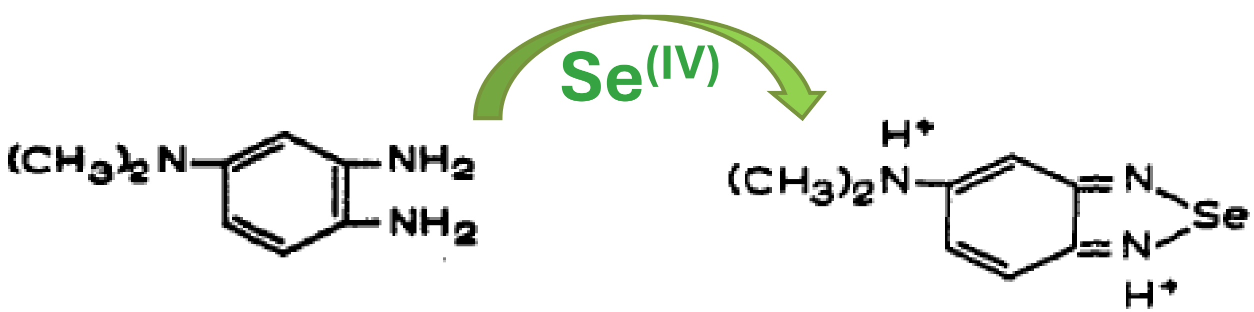
SE Resin

Se retention

Prefilter Resin

↓

extractant bleeding



Acid media (H^+)

Influence Se oxidation state

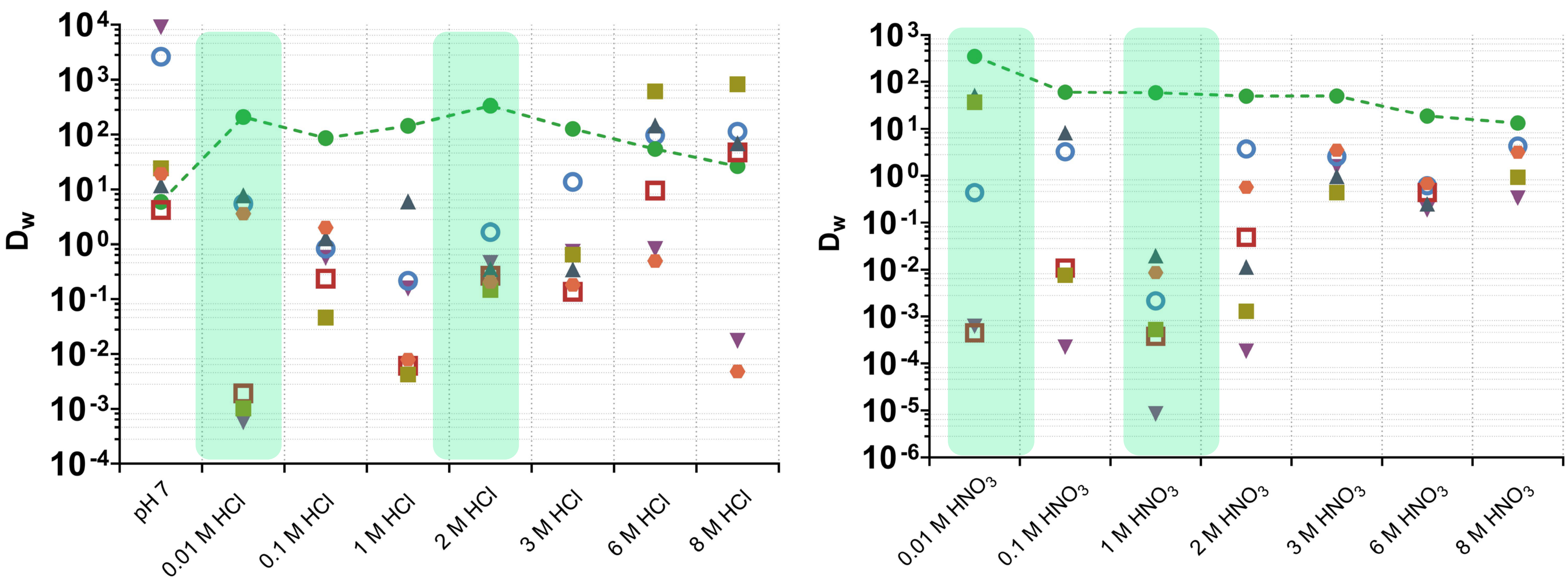
On the determination of ^{79}Se

Initial Se selectivity

Batch experiments

As Sb Se In
Ga Ge Te

reported by Dirks et al. 2016



HCl

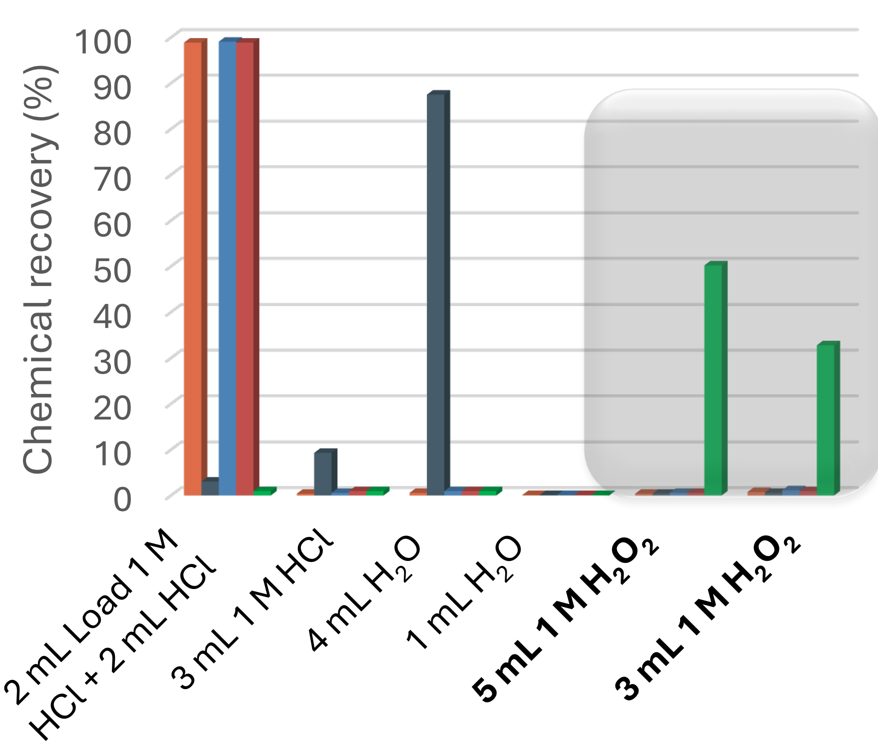
HNO_3

On the determination of ^{79}Se

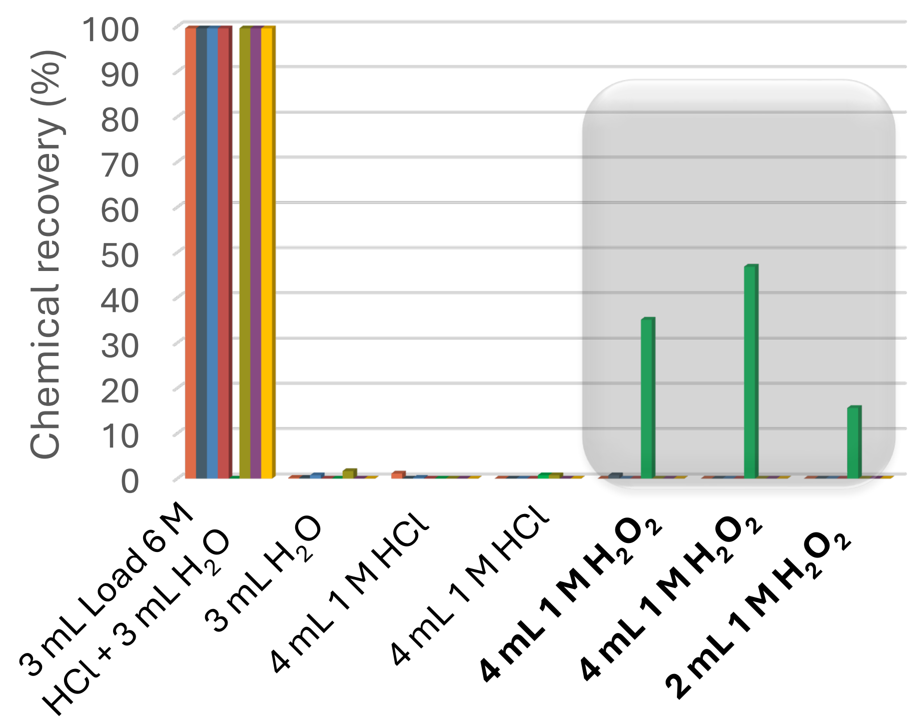
Initial Se profiles

reported by Dirks et al. 2016

As Sb Se In Ga Ge Te Cd



350 mg Se selective Resin **Load 1 M HCl**

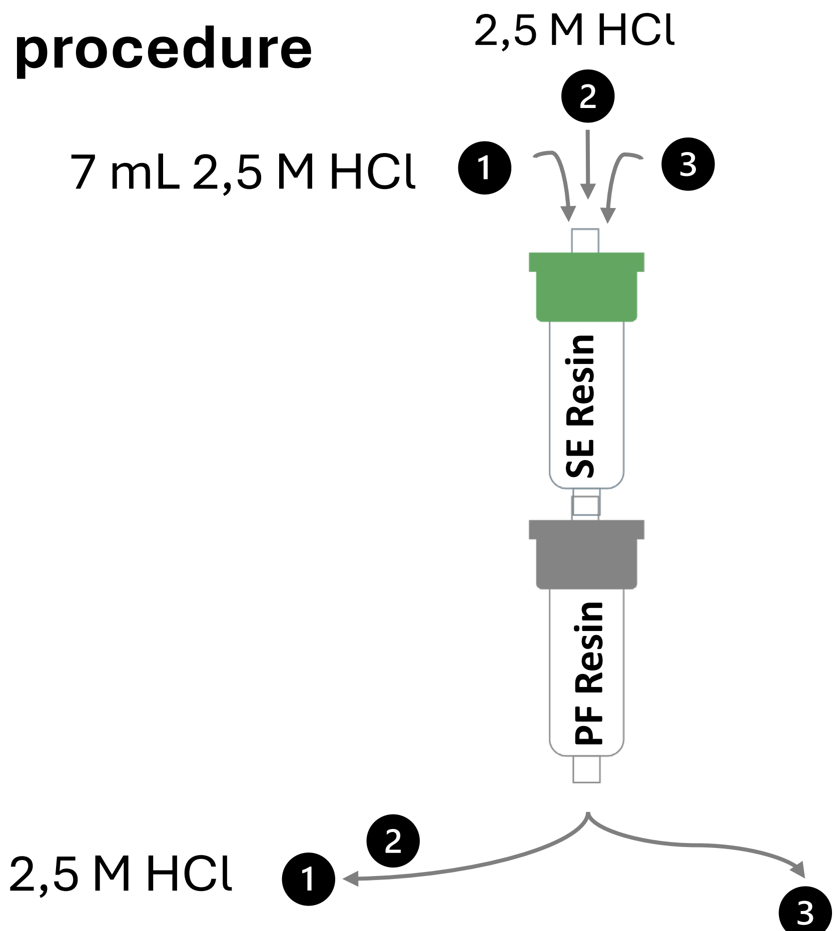


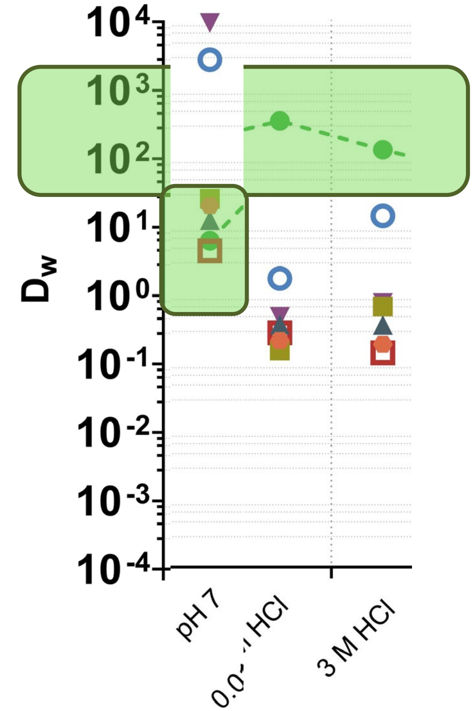
300 mg Se selective Resin **Load 6 M HCl**

On the determination of ^{79}Se

Initial Se profiles

“standard” procedure

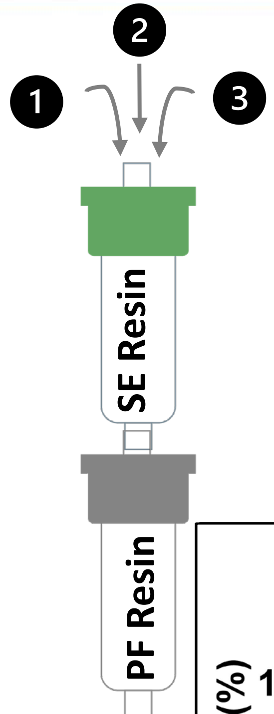




Se

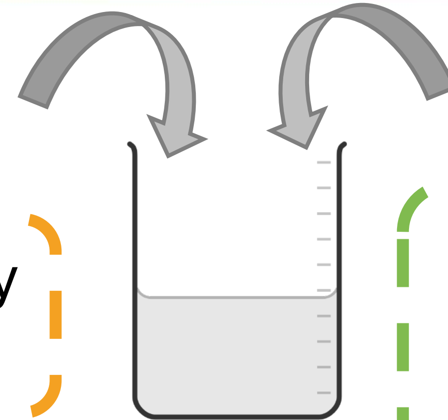
2x10 mL 1 M NaOH

On the determination of ^{79}Se
Using different tracers



1 mg
stable Se

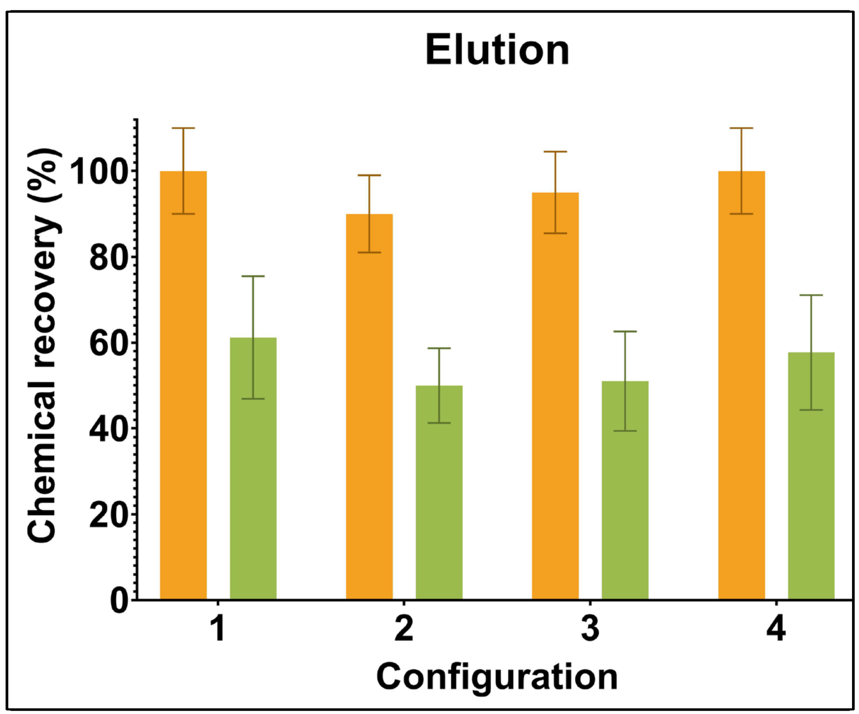
Measured by
SF-ICP-MS



6 Bq ^{75}Se

Measured by
gamma-ray
spectrometry

1. 7 mL 2.5 M HCl
2. 10 mL 2.5 M HCl
3. 2x10 mL 1 M NaOH



Difference in Se
retentions

On the determination of ^{79}Se
Contributing factors

Difference in Se retentions

Incomplete mixing / spike conditioning

Spike separately ^{75}Se and stable Se

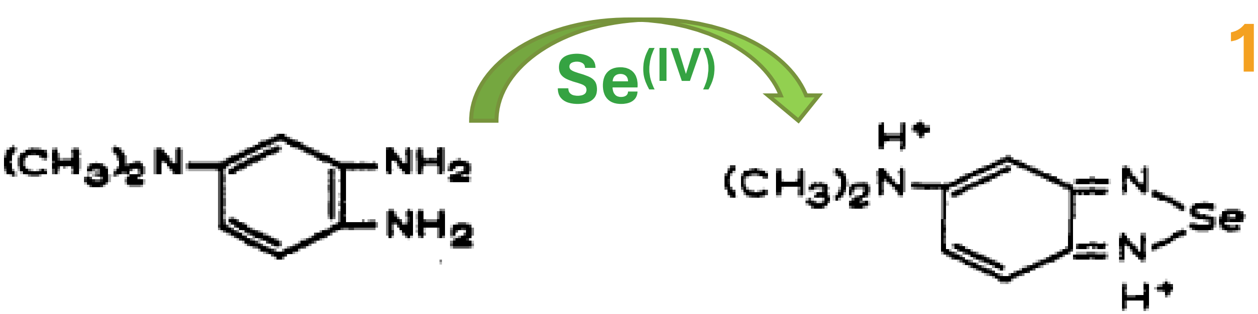
Different measurement techniques / calibration issues

Total of 100 % with all fractions collected

Resin capacity / breakthrough

Spike separately ^{75}Se and stable Se

Different chemical forms / speciation



100% | 1 mg stable Se | Se$^{(IV)}$

50% | 6 Bq ^{75}Se | Se$^{(IV/VI)}$

On the determination of ^{79}Se

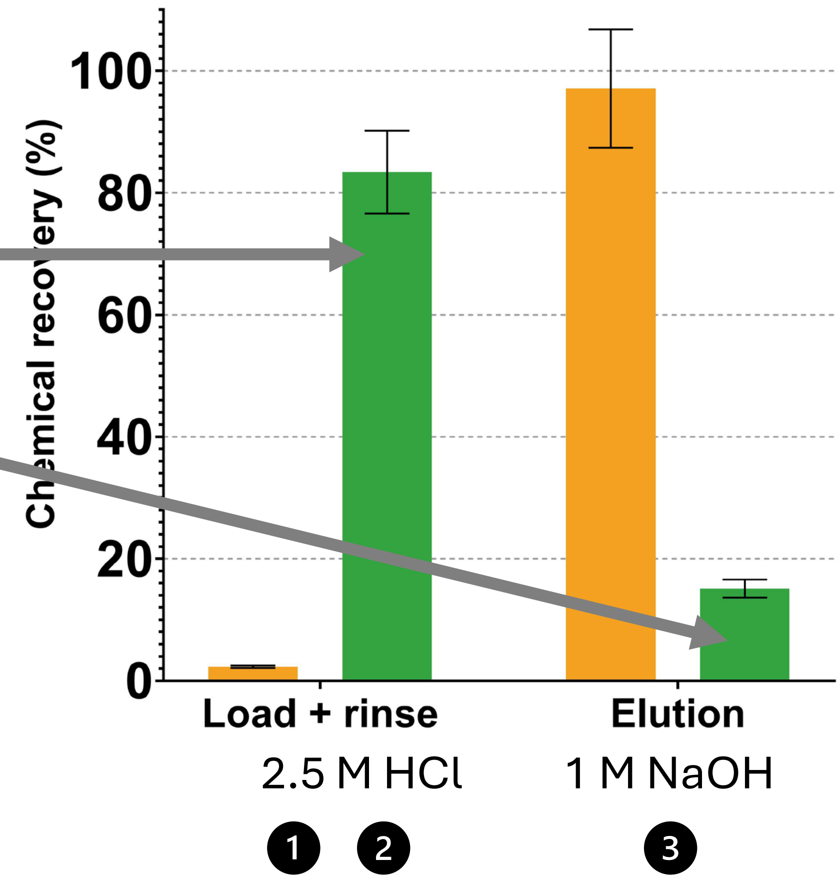
Se oxidation states

$Se^{(IV)}$ standard (2% HNO_3)

$Se^{(VI)}$ standard (in H_2O)

- 85% $Se^{(VI)}$
- 15% $Se^{(IV)}$

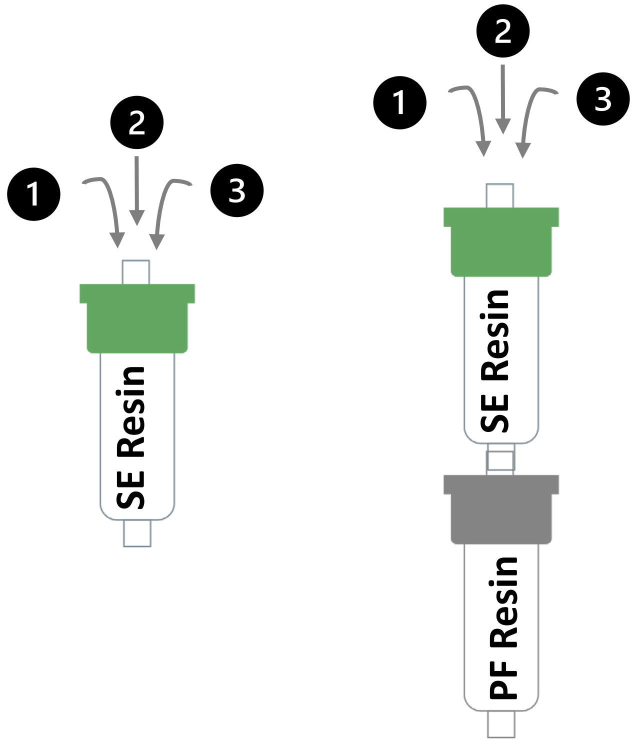
1. 7 mL 2.5 M HCl
2. 10 mL 2.5 M HCl
3. 2x10 mL 1 M NaOH



Different behavior of Se(IV) and Se(VI)

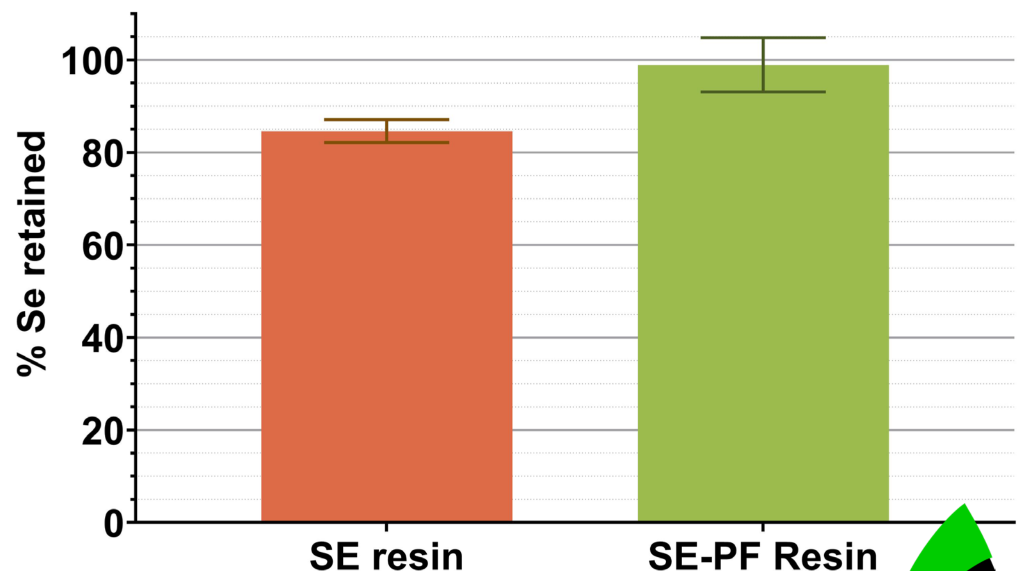
❖ Se(IV) fully retained

On the determination of ^{79}Se
Influence of PF Resin



1. 7 mL 2.5 M HCl
2. 10 mL 2.5 M HCl
3. 2x10 mL 1 M NaOH

$Se^{(IV)}$ standard ($2\%\ HNO_3$)



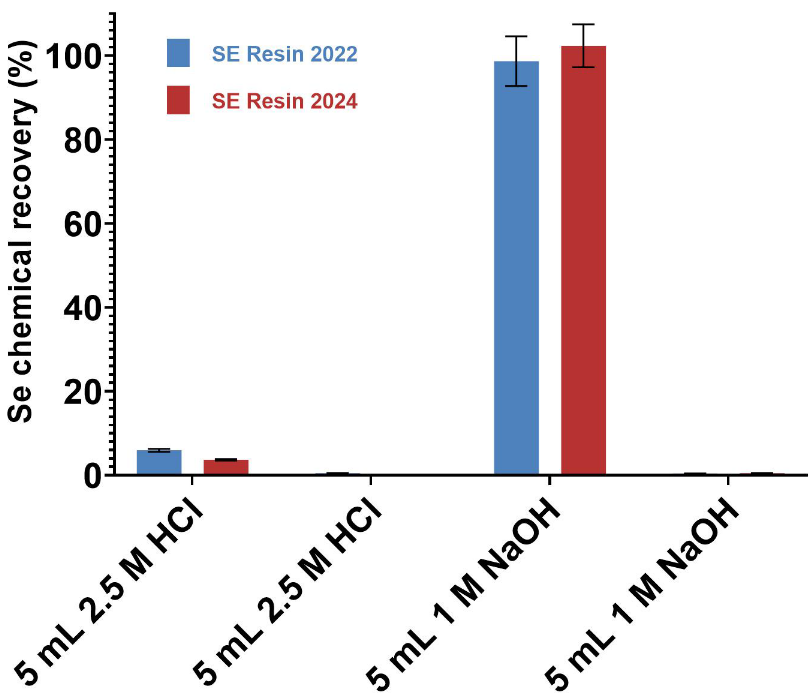
PF reduced **extractant bleeding** on fractions collected

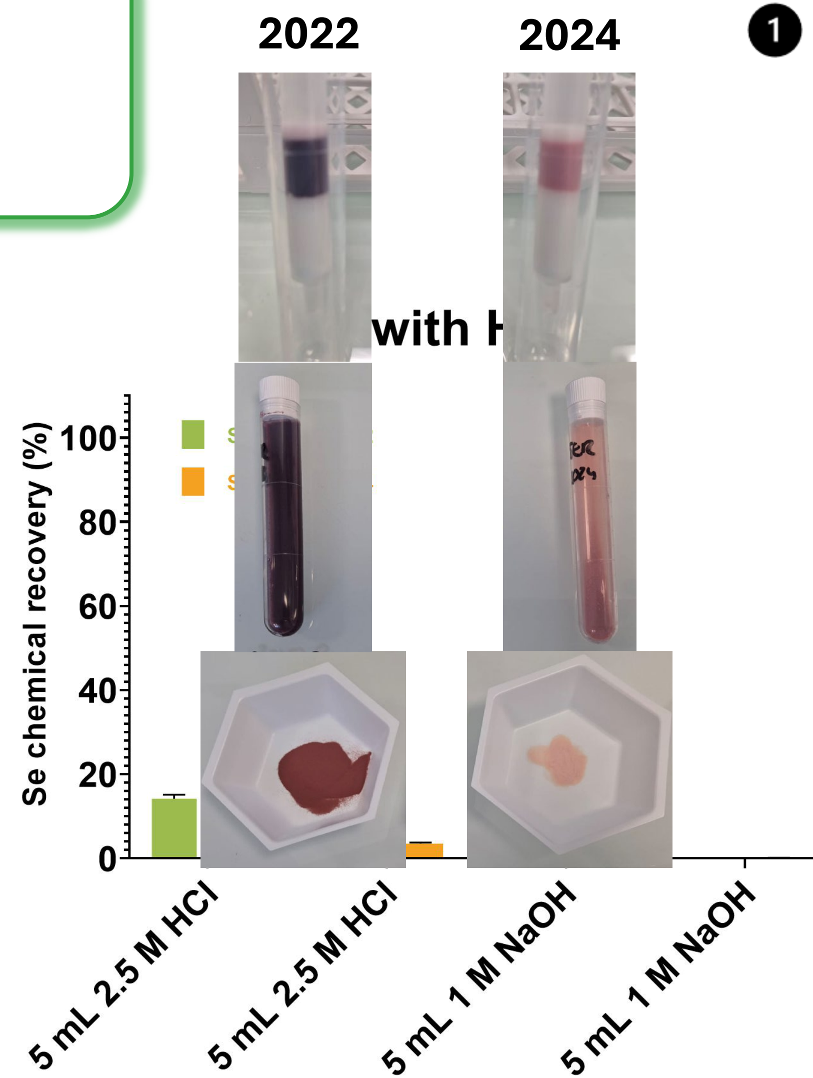
On the determination of ^{79}Se

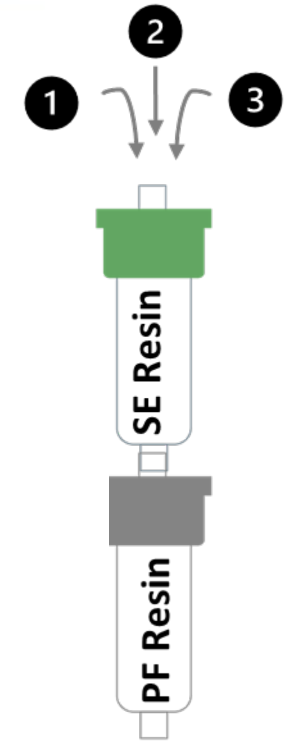
Ageing effect

- ❑ Stability SE Resin over time
- ⚠ **Need to fix Se oxidation state**

No treatment







On the determination of ^{79}Se

Dealing with Se oxidation states

Influence of pH on Se retention

Bosca & Mot, 2021 ➔ relevance of pH on *piazselenol* formation (**pH~1.3**)

Se$^{(IV)}$ standard

Se$^{(VI)}$ standard

1. 1 mL 2.5 M HCl
2. 5 mL 2.5 M HCl
3. 2x10 mL 1 M NaOH

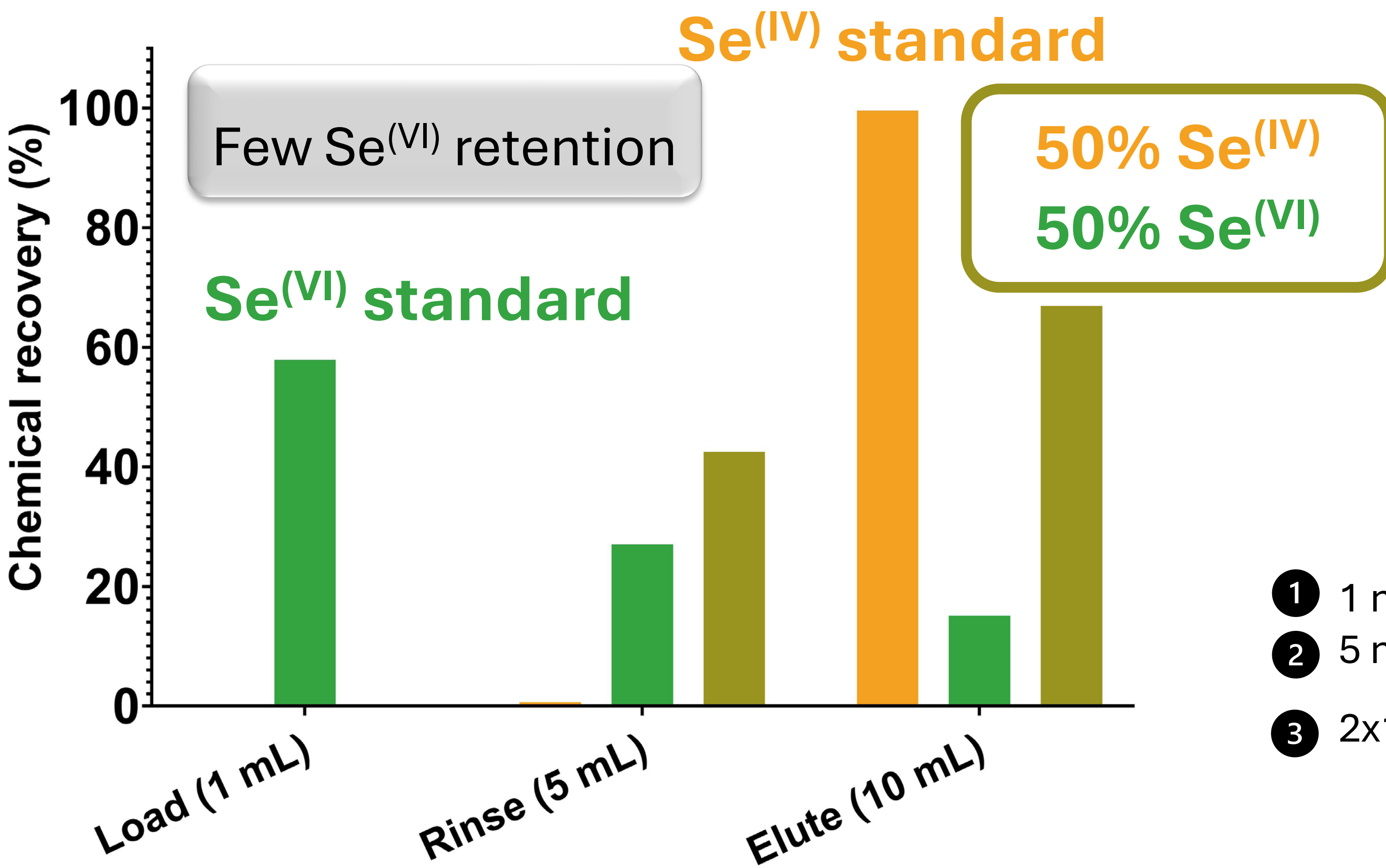
Clear **differences** between Se^{+4} and Se^{+6} retention

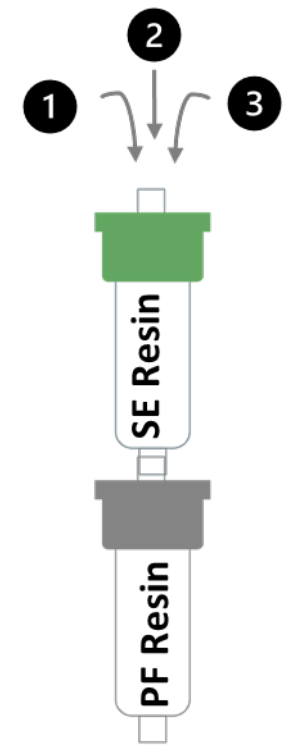
Se **reduction cycle** still needed

On the determination of ^{79}Se

Dealing with Se oxidation states

Loading different Se oxidation states





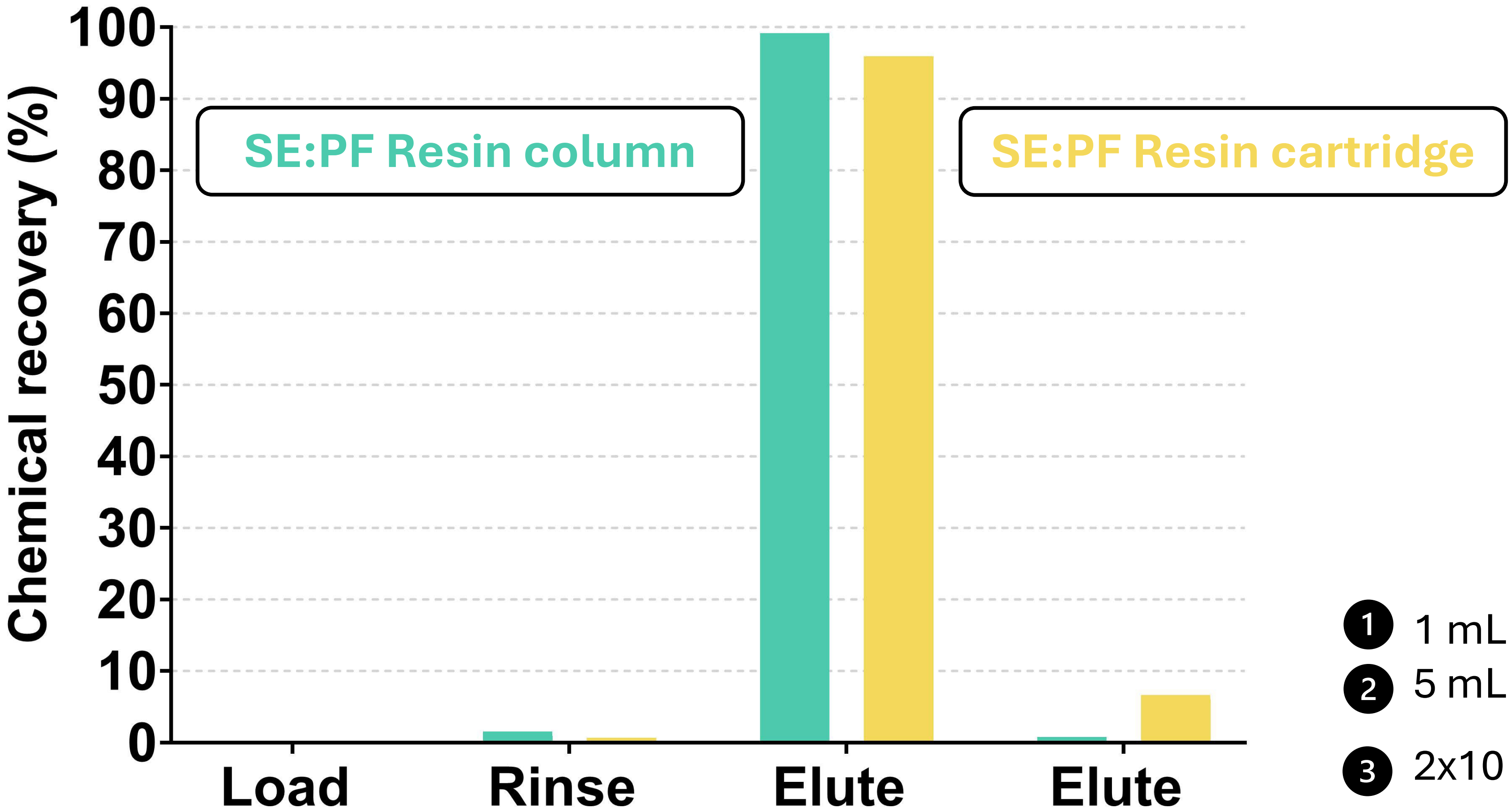
1. 1 mL 2.5 M HCl
2. 5 mL 2.5 M HCl
3. 2x10 mL 1 M NaOH

On the determination of ^{79}Se

Dealing with Se oxidation states

Comparison column vs. cartridge

$Se^{(IV)}$ standard



1. 1 mL 2.5 M HCl
2. 5 mL 2.5 M HCl
3. 2x10 mL 1 M NaOH

On the determination of ^{79}Se
Dealing with Se oxidation states

Se reduction using HCl

Ragnar Bye and Waiter Lund, 1988 → relevance of HCl concentration, temperature and time on Se reduction

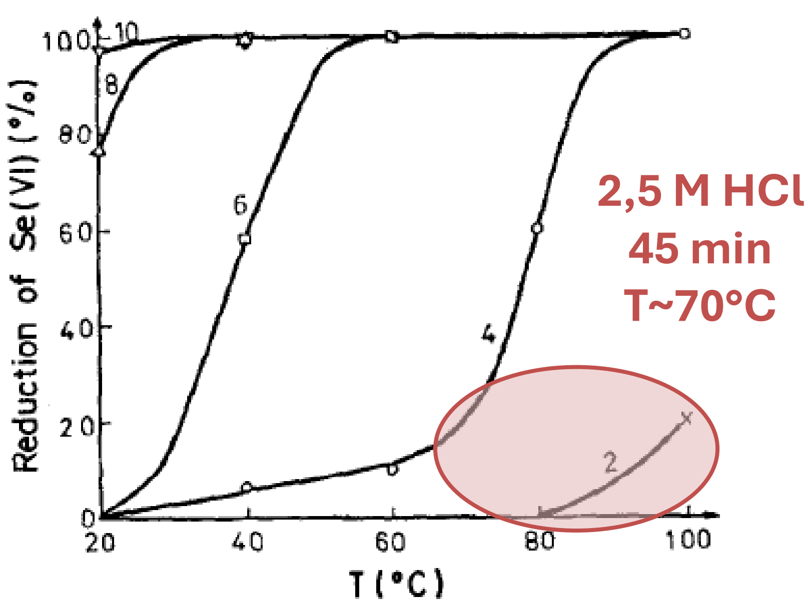


Fig. 2. Reduction of 1.6 µg Se(VI) with 10 ml HCl of various concentrations (mol/l) after 30 min in dependence on temperature

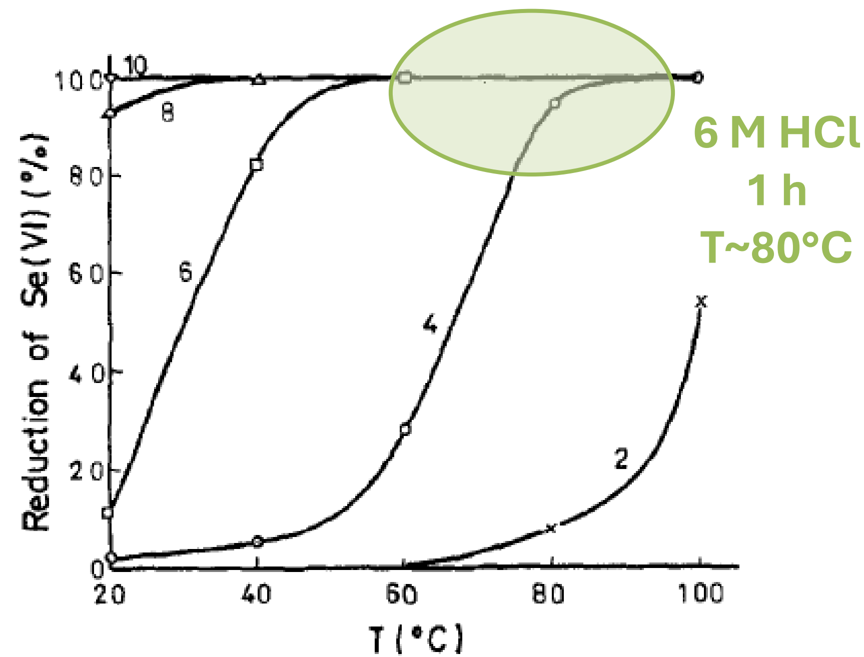
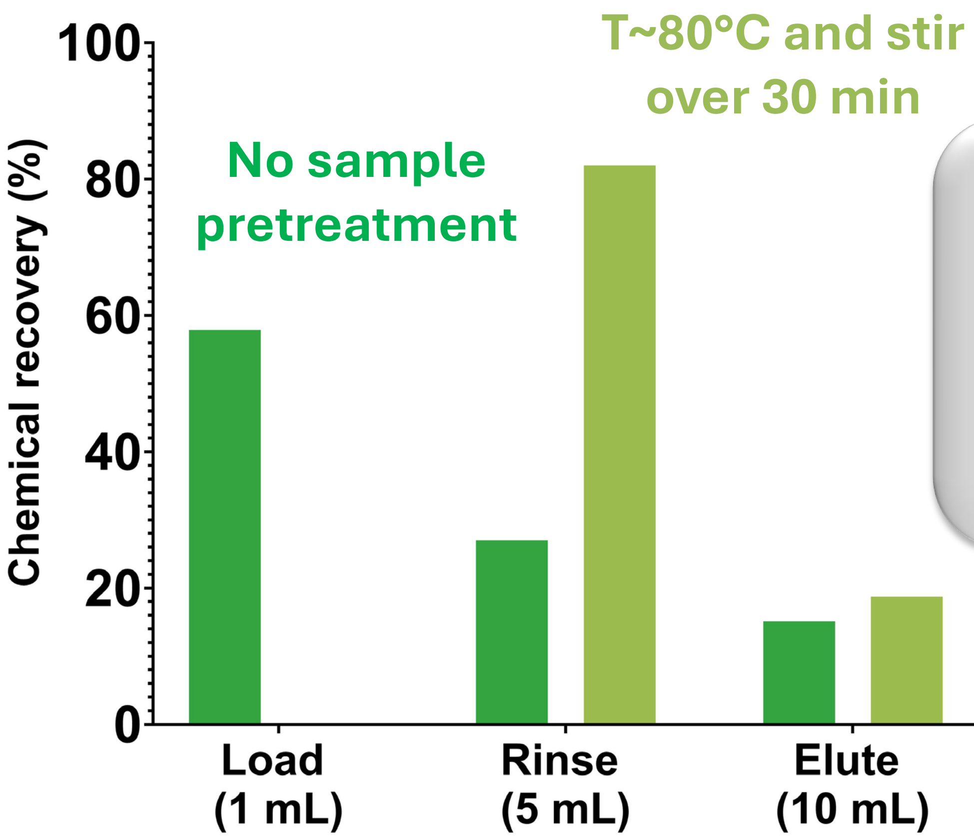


Fig. 4. Reduction of 1.6 µg Se(VI) with 10 ml HCl of various concentrations (mol/l) after 2 h in dependence on temperature

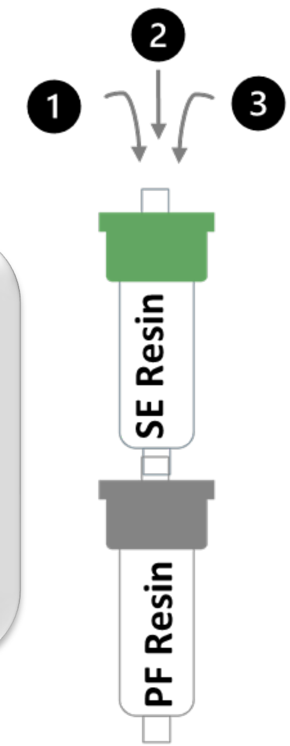
On the determination of ^{79}Se

Dealing with Se oxidation states

Se reduction using HCl



Higher $Se^{(VI)}$ retention when warming the sample before loading

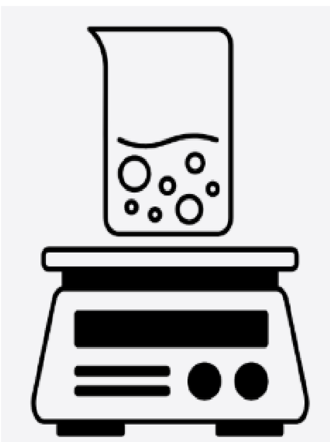


1. 1 mL 2.5 M HCl
2. 5 mL 2.5 M HCl
3. 2x10 mL 1 M NaOH

On the determination of ^{79}Se

Dealing with Se oxidation states

Se reduction using HCl



T=80°C
60 min

1. Load
2. 5 mL 2.5 M HCl
3. 2x10 mL 1 M NaOH

100% Se$^{(VI)}$ retention when the sample is heated and loaded under high acid concentrations

On the determination of ^{79}Se
Dealing with Se oxidation states

Interferences removal

$Se^{(VI)}$
Fe
Ni
Co

T=80°C
60 min

Step	Fraction
Load	1 mL
Rinse	5 mL 2,5 M HCl
Elution	5 mL 1 M NaOH
Elution	5 mL 1 M NaOH

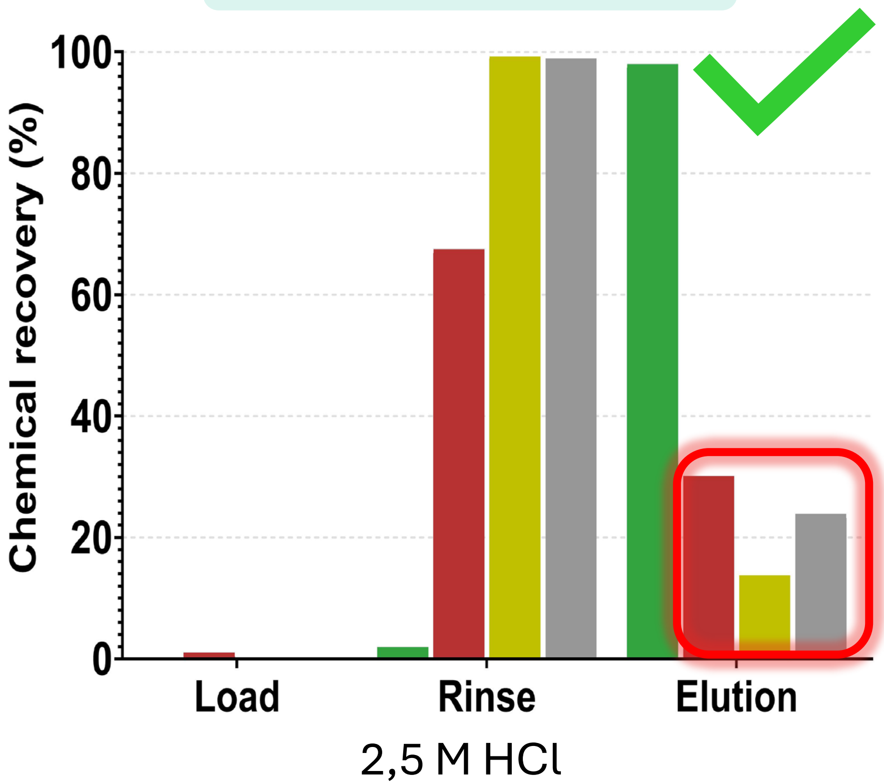
5.5 M HCl
7 M HCl

On the determination of ^{79}Se
Dealing with Se oxidation states

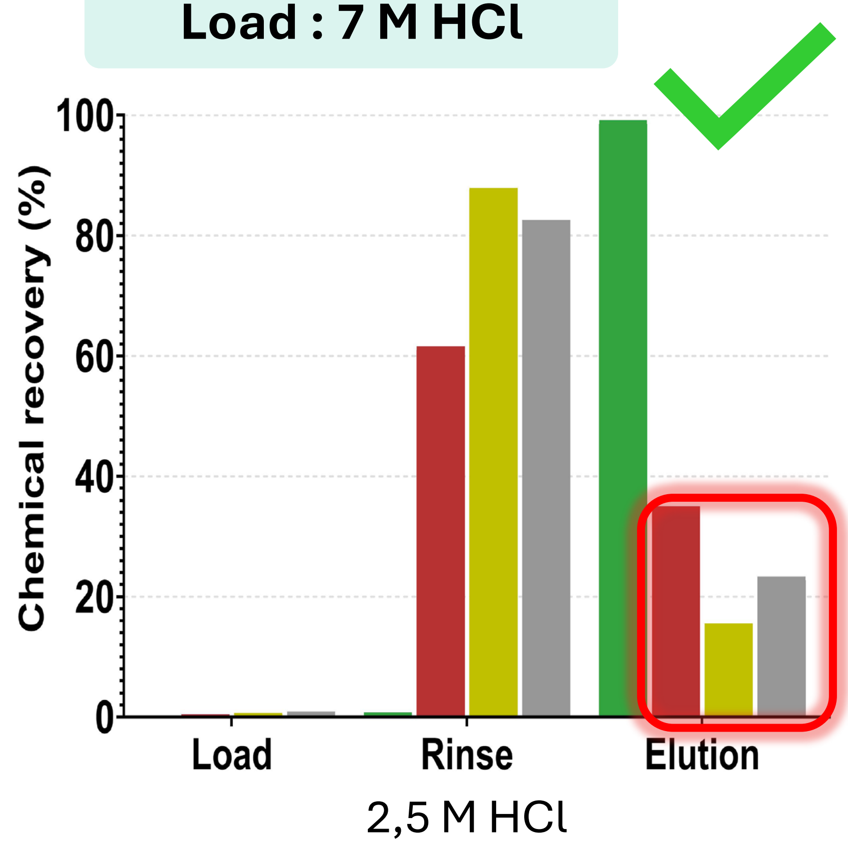
Interferences removal

Se(VI) Fe Ni Co

Load : 5.5 M HCl



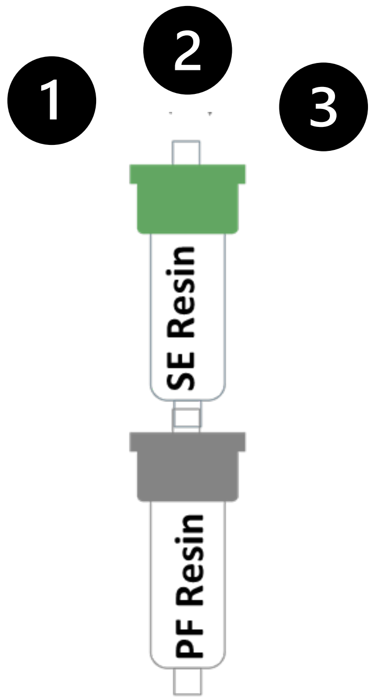
Load : 7 M HCl



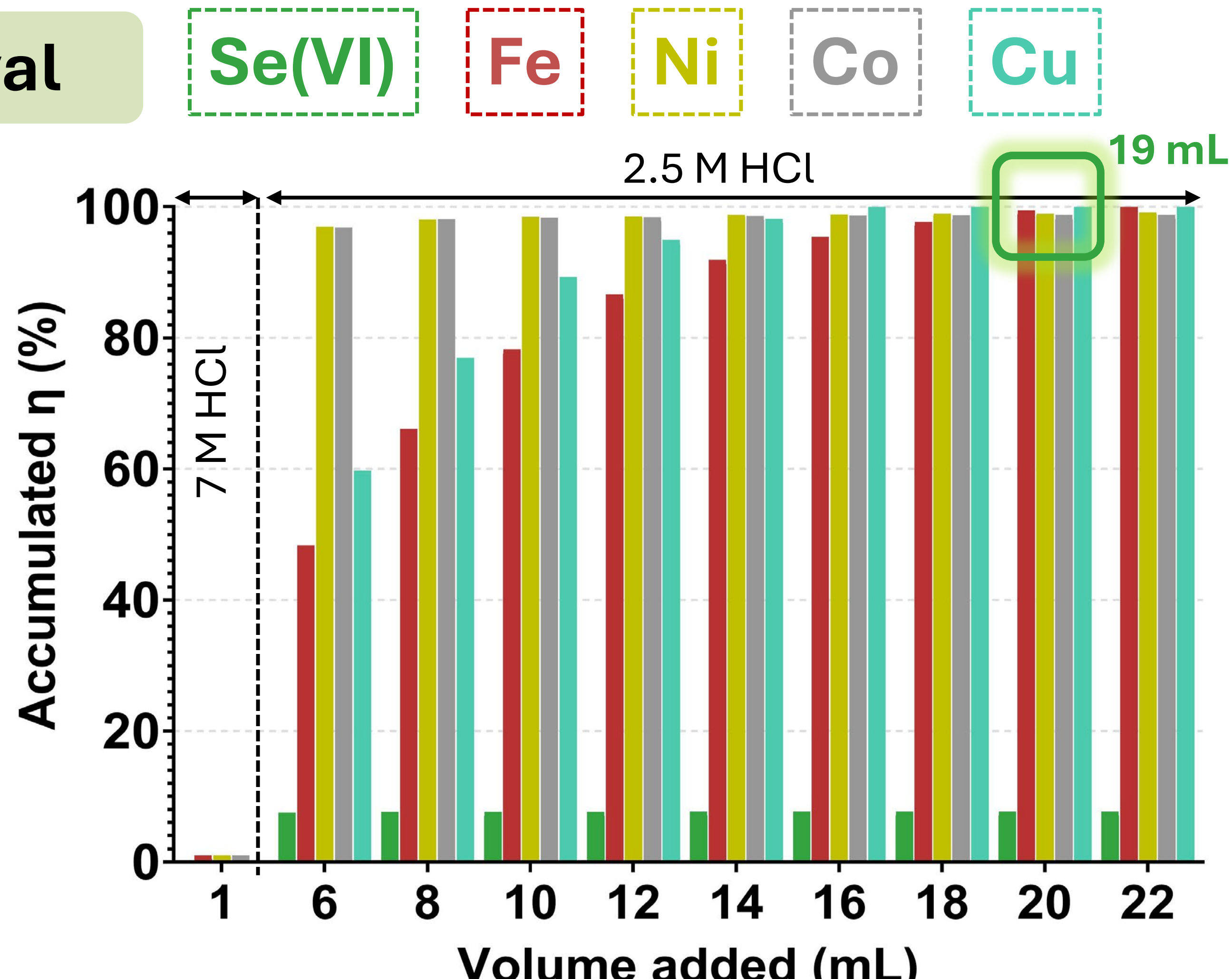
Se(VI) fully reduced when using ↑ [HCl] ➔ Full Se retention

On the determination of ^{79}Se
Dealing with Se oxidation states

Interferences removal



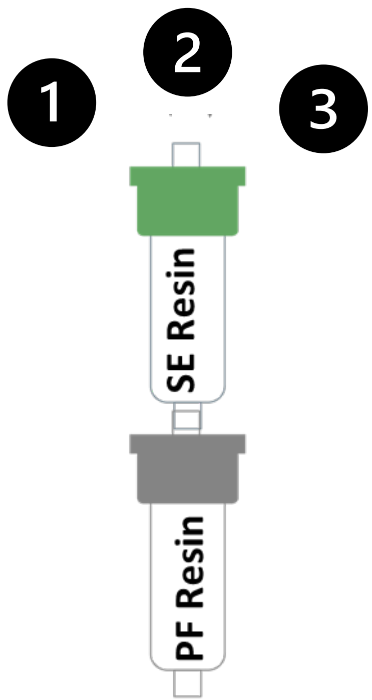
1. Condition: 2.5 M HCl
2. Load: 7 M HCl
3. Rinse: 2.5 M HCl

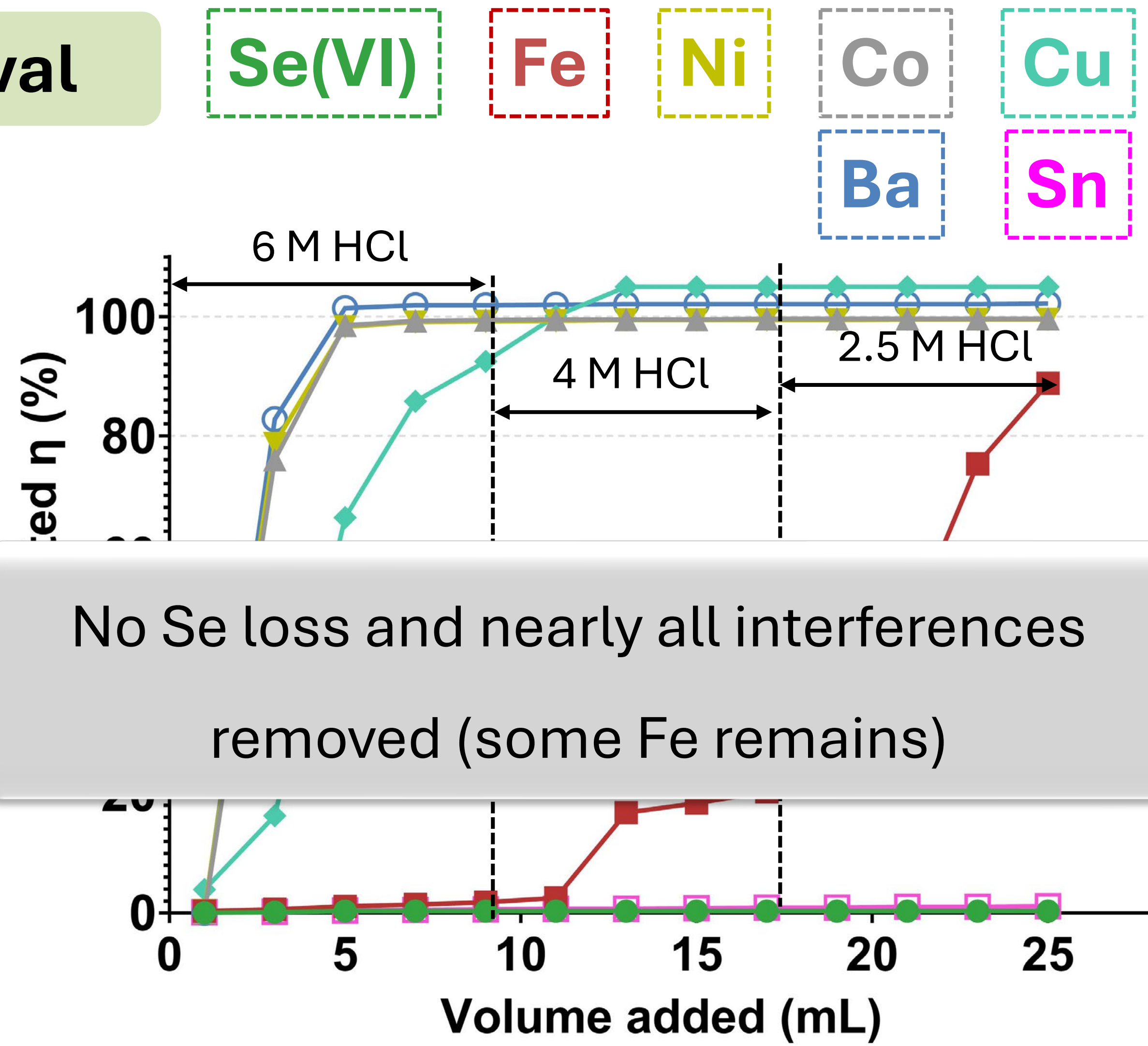


<10 % Se lost and interferences removed

On the determination of ^{79}Se
Dealing with Se oxidation states

Interferences removal





No Se loss and nearly all interferences removed (some Fe remains)

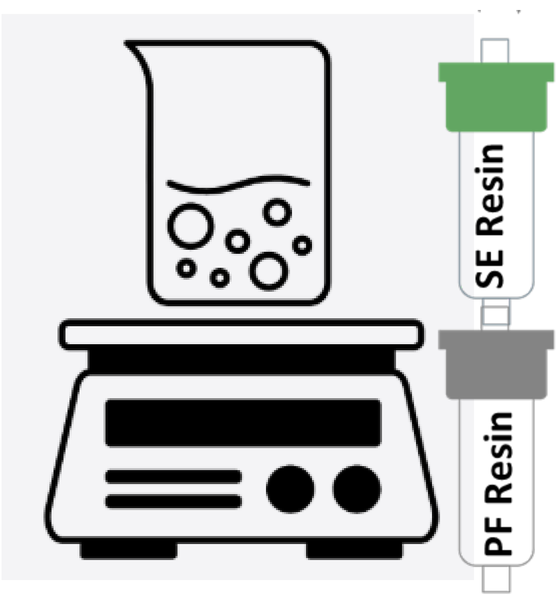
1. Condition: 6 M HCl
2. Load: 6 M HCl
   6 M HCl
3. Rinse: 4 M HCl
   2.5 M HCl

On the determination of ^{79}Se

Dealing with Se oxidation states

Final chemical separation procedure

Se(IV) Fe Ni
Co Sn



T=80°C
60 min

Se(VI) Fe Ni
Co Sn

T=80°C
60 min

1 Load

6 M HCl

2 Rinse

8 mL 6 M HCl

8 mL 2.5 M HCl

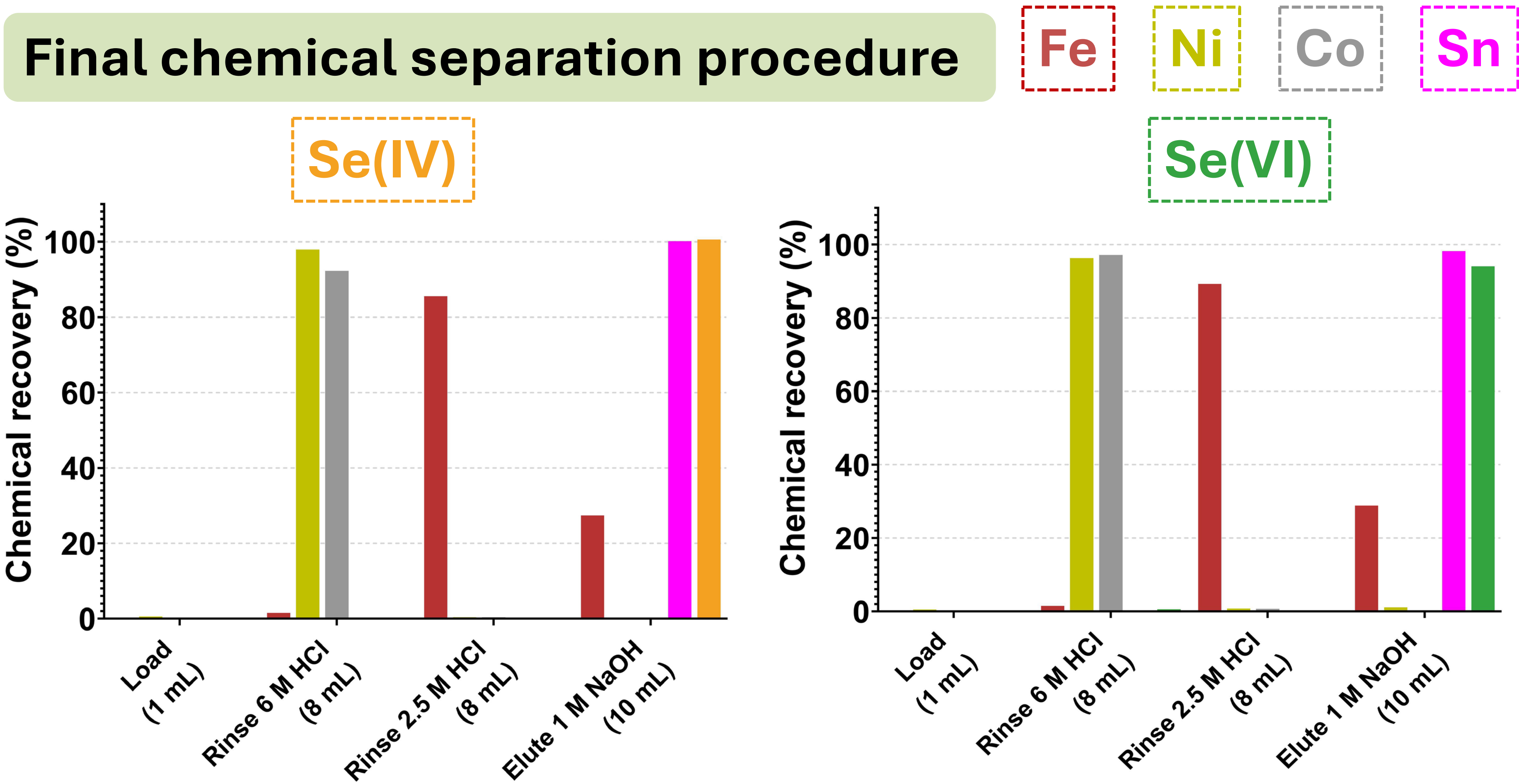
3 Elution

2 * 5 mL 1 M NaOH

On the determination of ^{79}Se

Dealing with Se oxidation states

Final chemical separation procedure



Complete Se retention (presence of Sn and few Fe)

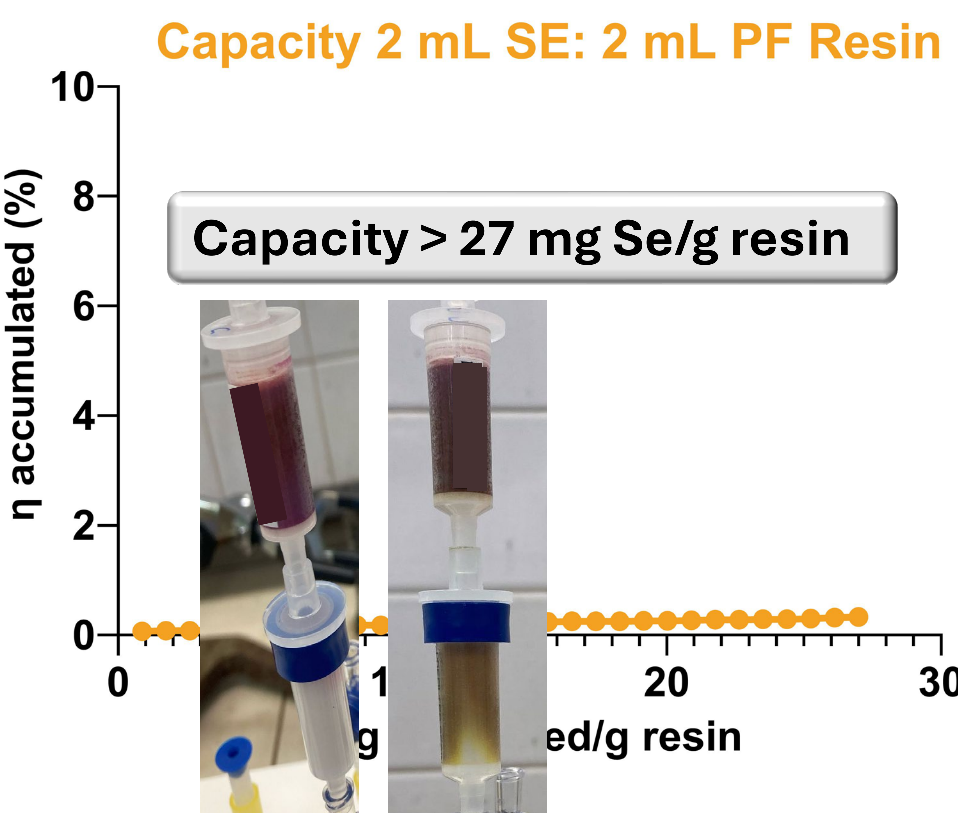
On the determination of ^{79}Se

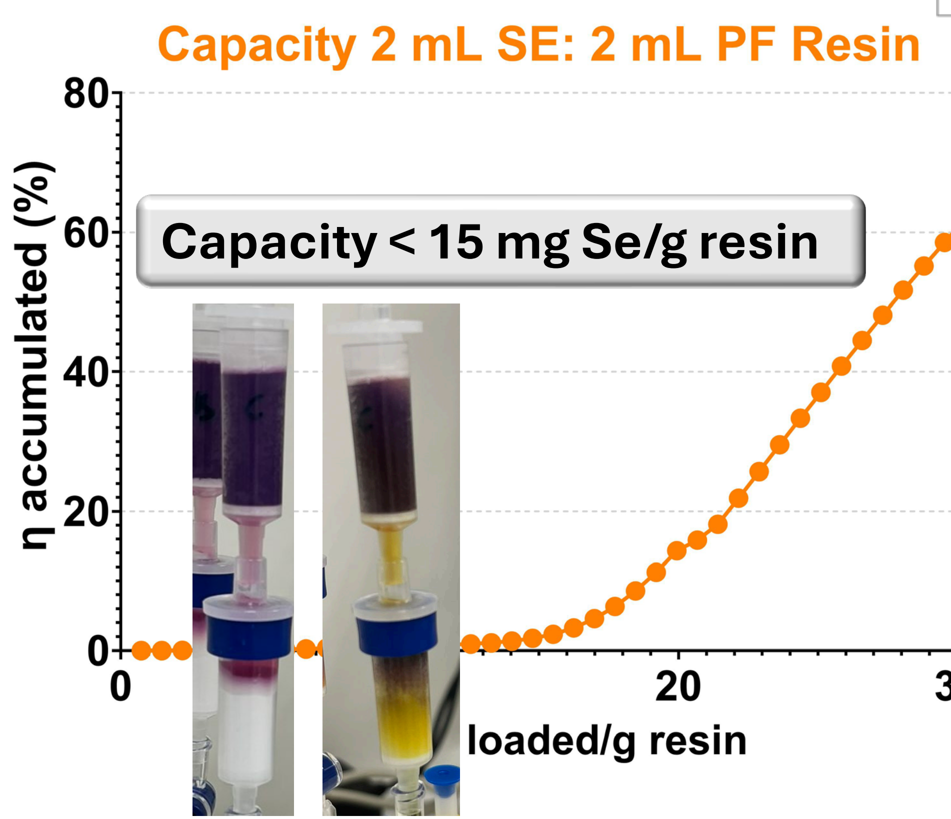
Parameters investigated

Capacity of SE resin

Loading with **2.5 M HCl** (100 % retention Se(IV))

Loading with **6 M HCl** (complete Se reduction)





On the determination of ^{79}Se

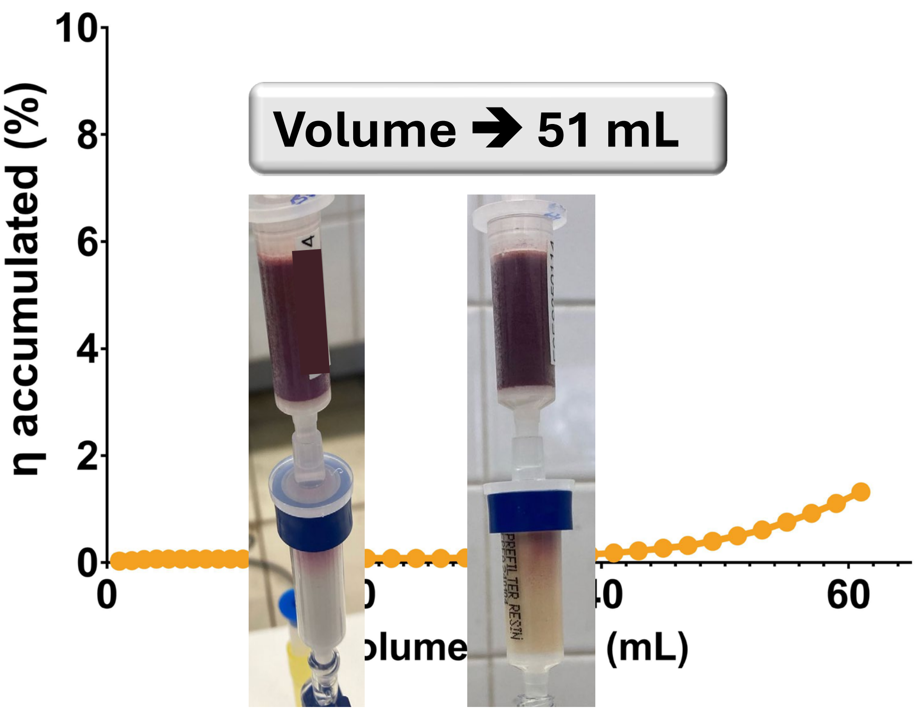
Parameters investigated

Breakthrough volume SE Resin

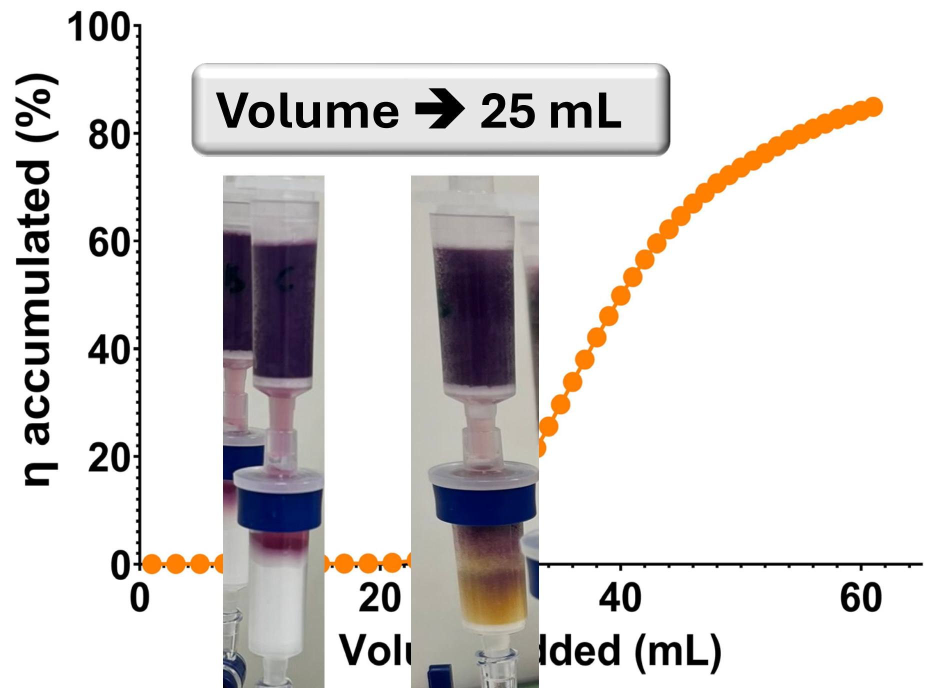
Loading with **2.5 M HCl** (100 % retention Se(IV))

Loading with **6 M HCl** (complete Se reduction)

Breakthrough volume 2.5 M HCl



Breakthrough volume 6 M HCl

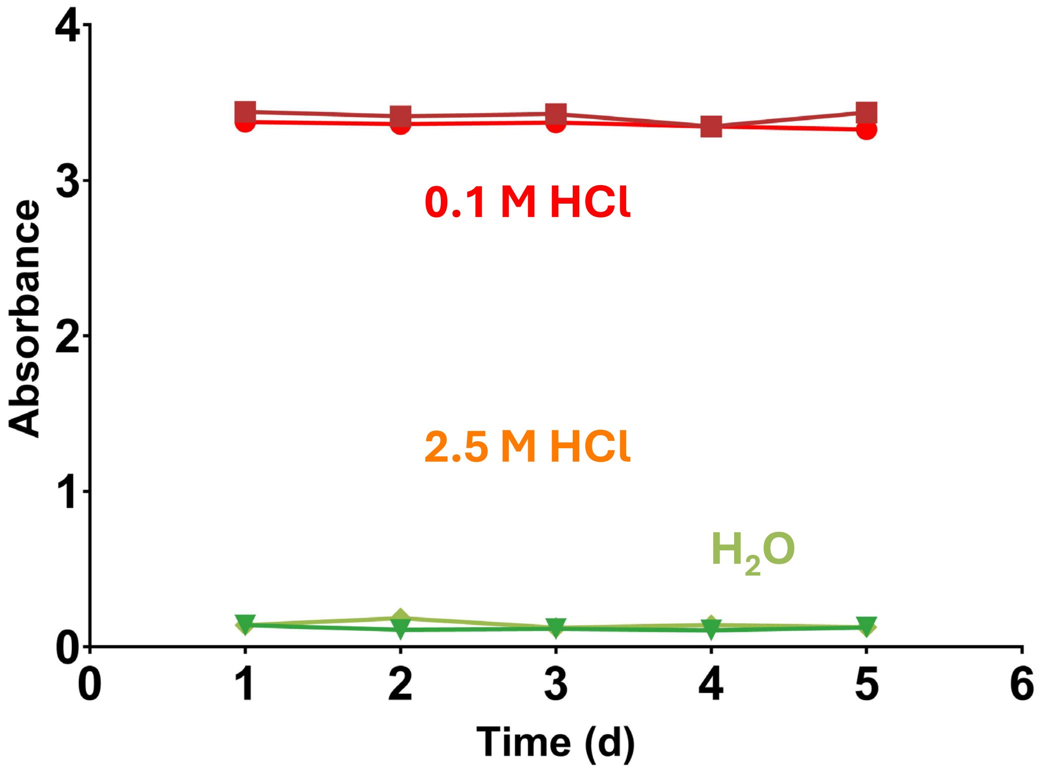


On the determination of ^{79}Se
Parameters investigated

Stability of the resin in different media

In collaboration with Matic Dokl

- No change in absorbance over time
- Leaching is the most prominent in 0.1M HCl and stable in water
- Shaking doesn't affect the leaching



Wavelength [nm]

Absorbance of solutions after soaking SE Resin with different media

Analytical methods

On the determination of ^{79}Se

Current challenges

Fixation of Se oxidation state — Redox conditions

Interference removal studies — ^{99}Tc, ^{147}Pm, ^{93}Zr, ^{63}Ni, ^{55}Fe

Application of the procedure

⬆ Chemical recovery

⬇ LOD

^{75}Se diagnostic

NUCLEAR MEDICINE

Thank you for your attention!!

Questions?

TRISKEM
Expertise in Separation Chemistry