# Characterisation of a Cu selective resin for use in the production of Cu isotopes

# Outline

- Resin characterisation
  - Selectivity
  - Interferences with Ni or Zn
- Column Experiments
  - Optimisation
  - Simulated targets
  - Decontamination factors
- Conversion of Cu resin eluate via AIX
- Other applications

# $D_w$ Cu resin in HCl

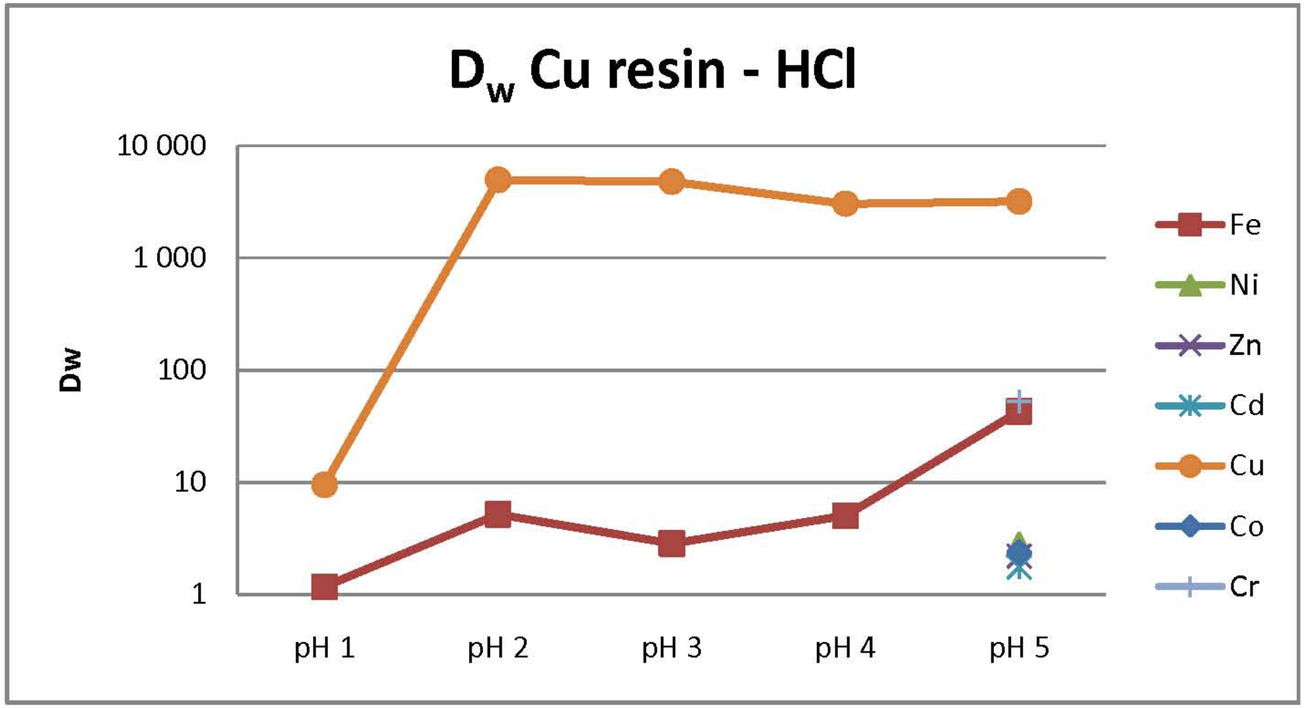


Figure 1: $D_w$ of Cu and selected elements on Cu resin in HCl, varying pH values, measurement via ICP-MS, each element 10µg/ml

Loading conditions (> pH 2):

- Good selectivity for Cu over other elements tested,
- Some selectivity for Fe at high pH

Elution:

- Low Cu $D_w$ at low pH, further tests with 4, 6 and 8 M HCl

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# $D_w$ Cu resin in $HNO_3$

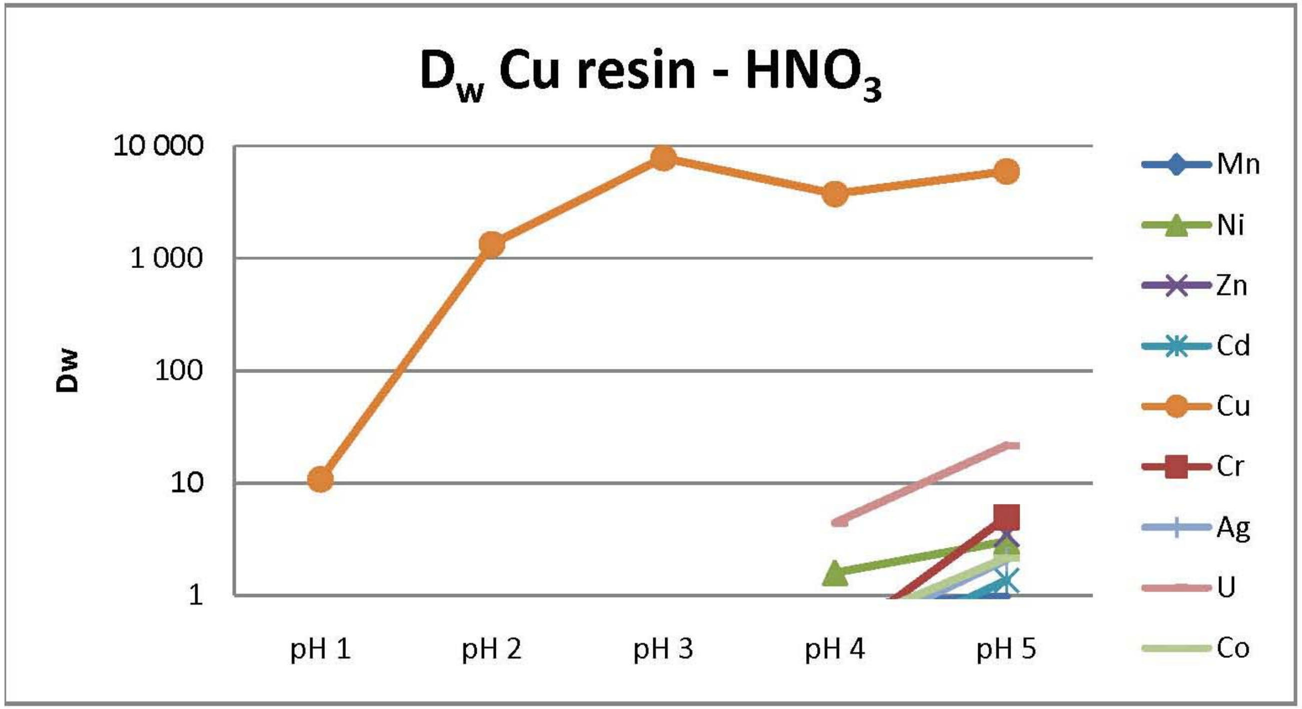


Figure 2: $D_w$ of Cu and selected elements on Cu resin in $HNO_3$, varying pH values, measurement via ICP-MS, each element 10µg/ml

Loading conditions (> pH 2):

- Good selectivity for Cu over all other elements tested

Elution:

- Low Cu $D_w$ at low pH

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# $D_w$ Cu resin in $H_2SO_4$

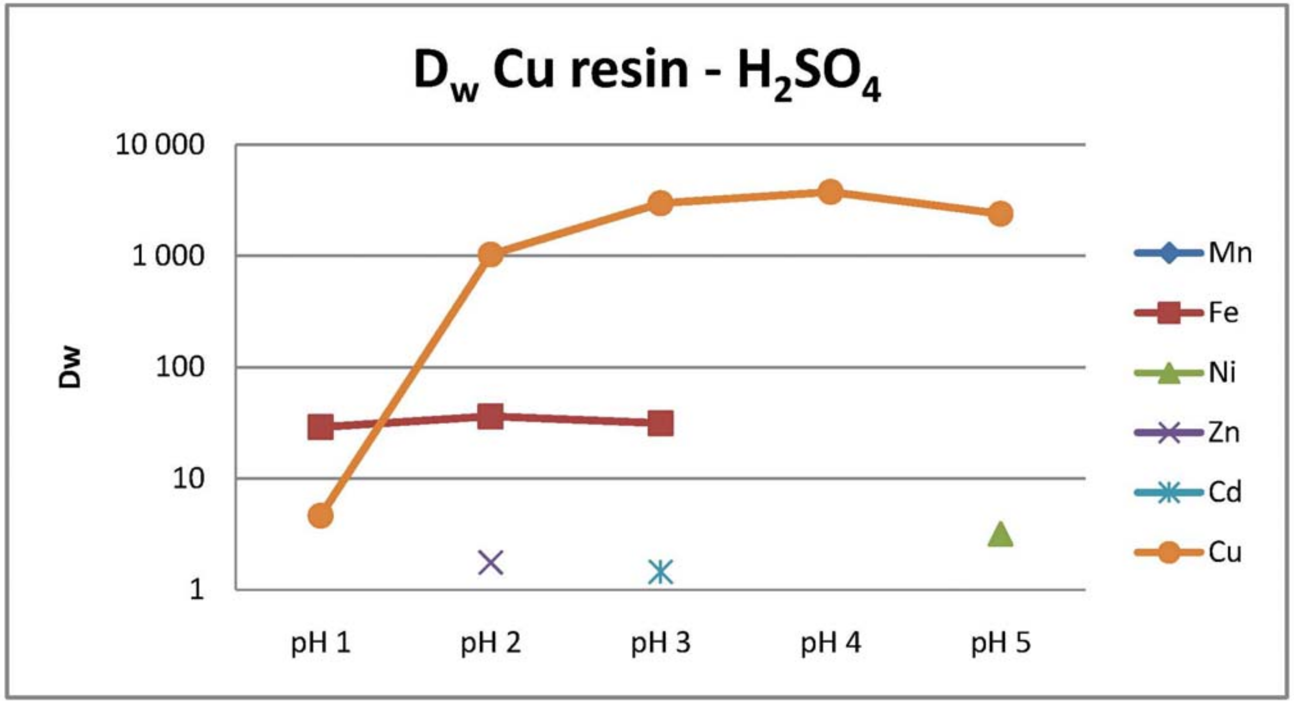


Figure 3: $D_w$ of Cu and selected elements on Cu resin in $H_2SO_4$, varying pH values, measurement via ICP-MS, each element 10µg/ml

Loading conditions (> pH 2):

- Good selectivity for Cu over most other elements tested
- Limited selectivity for Fe

Elution:

- Low Cu $D_w$ at low pH

# $D_w$ Cu – interferences - HCl pH 2

## Ni interference

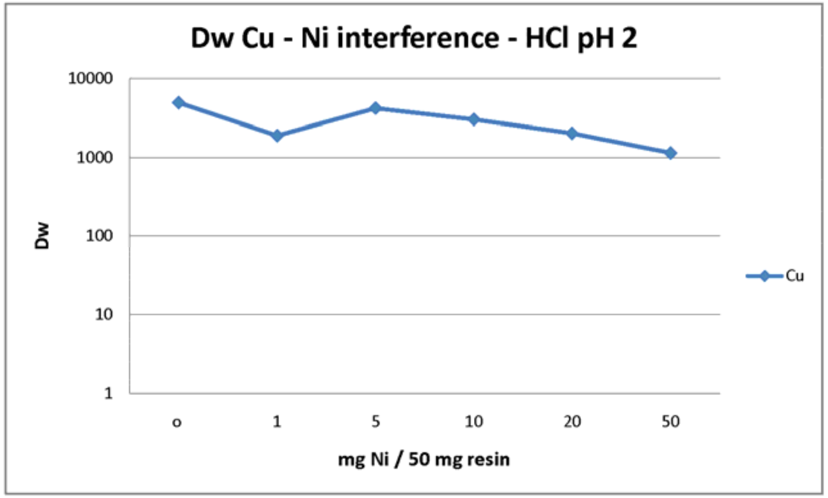


Figure 4: $D_w$ of Cu on Cu resin in HCl (pH 2) in presence of various amounts of Ni

## Zn interference

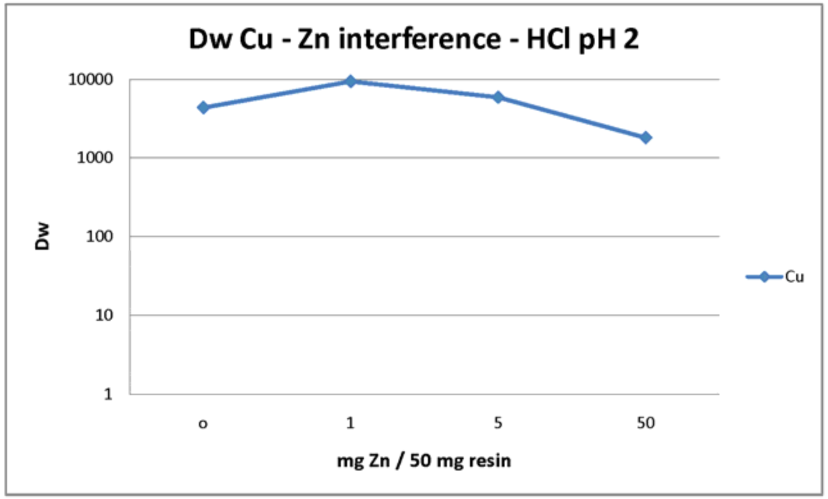


Figure 5: $D_w$ of Cu on Cu resin in HCl (pH 2) in presence of various amounts of Zn

- For high amounts of Ni or Zn $D_w$(Cu) remains > 1000 in pH 2 HCl
- For up to 1 g target material per g resin only negligible interference

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# Conclusions I

- Stable, high Cu $D_w$ values at pH > 2 for all acids
- Good selectivity for Cu over tested elements
- No selectivity for Zn or Ni
- No interference on Cu extraction by elevated amounts of Ni or Zn
  - Tested up to 1g Ni or Zn per g of resin
- HCl system chosen for radiopharmaceutical application
  - Loading solution: HCl pH 2 (or higher)
  - Elution with HCl (elevated concentration)

# First approach

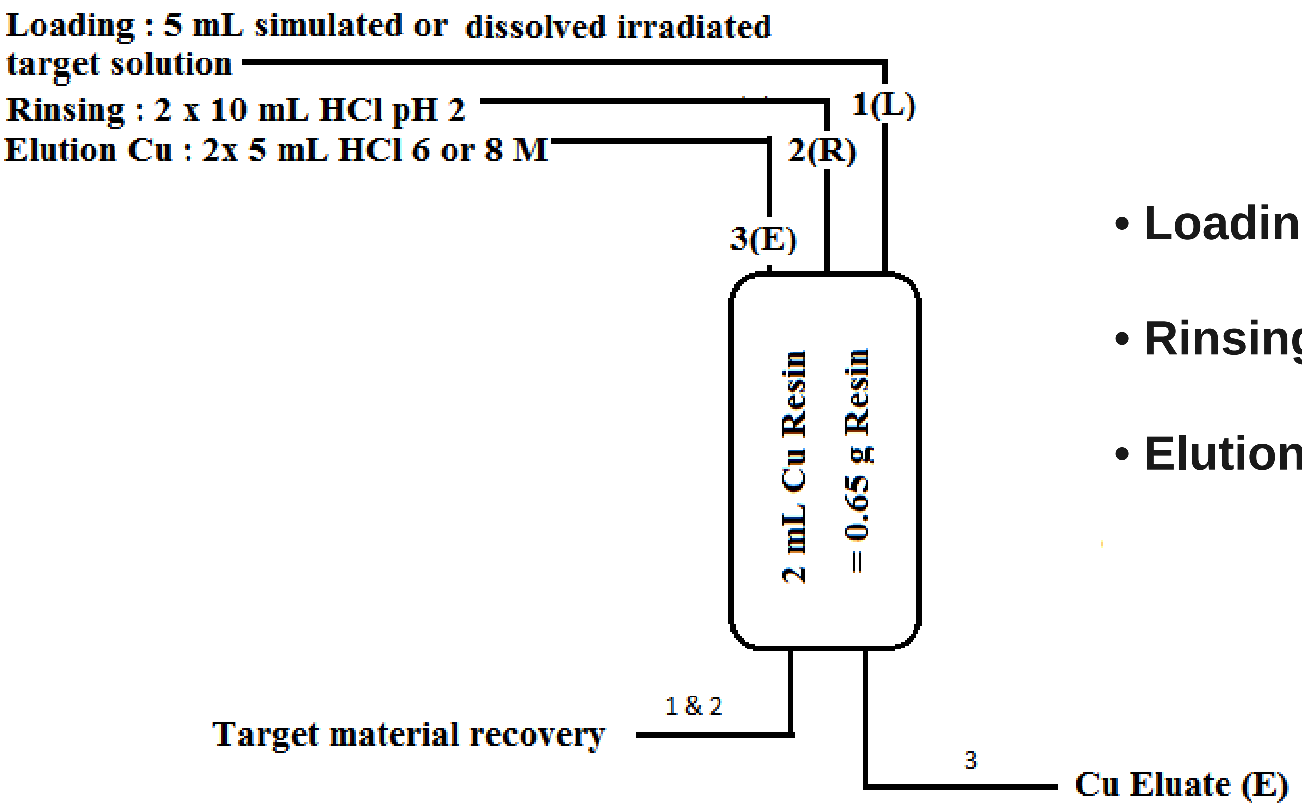
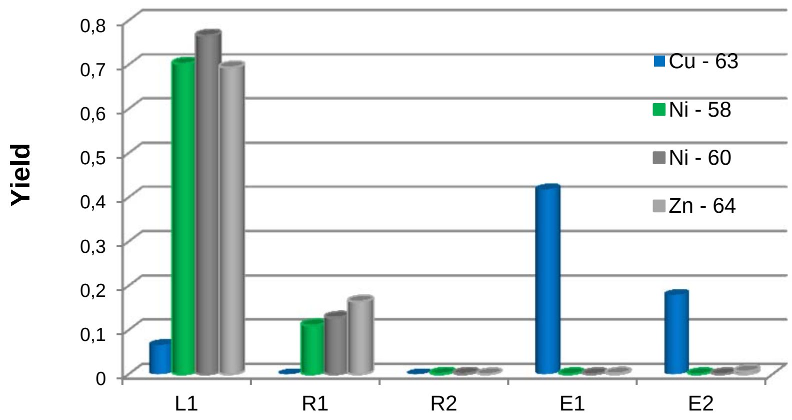



Figure 6: Scheme of elution conditions – first approach

- **Loading : L1**
- **Rinsing : R1 & R2**
- **Elution : E1 & E2**

# Elution study – 2 mL CU resin column

**Elution with 6 M HCl**

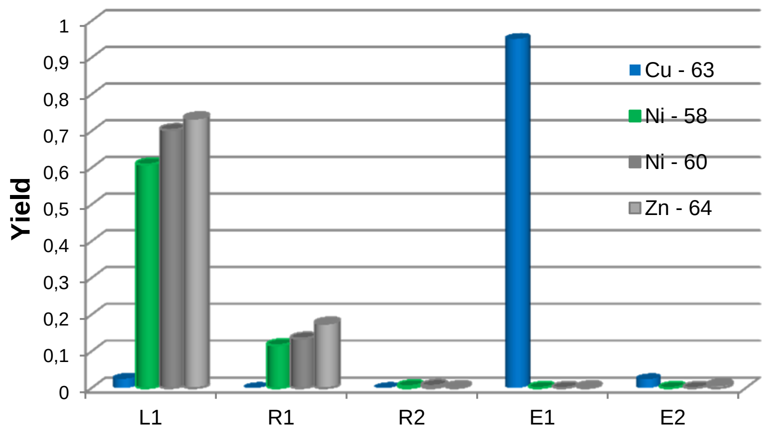



L1 = 5 mL simulated Ni-Target

R1 = 10 mL HCl pH 2
R2 = 10 mL HCl pH 2

E1 = 5mL HCl 6 M
E2 = 5mL HCl 6M

**Elution with 8 M HCl**




L1 = 5 mL simulated Ni-Target

R1 = 10 mL HCl pH 2
R2 = 10 mL HCl pH 2

E1 = 5mL HCl 8 M
E2 = 5mL HCl 8 M

Figure 7/8 : Elution study; simulated Ni targets (200 mg Ni, traces of Zn, Cu), elution with 6M HCl and 8M HCl

- 8M HCl allows for elution in smaller volume
- Clean Cu fraction

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# Elution study - irradiated Ni target

- Irradiation of a Ni foil (10 mg) :
  - (ø =13 mm, 0.025 mm thickness)
  - Cyclotron BC1710 at Forschungs Zentrum Jülich
  - $E_p$ = 15 MeV
  - 1 h; 0.5 mA

- Additionally 170 mg non-irradiated Ni-foil added

# Elution study – irradiated Ni target

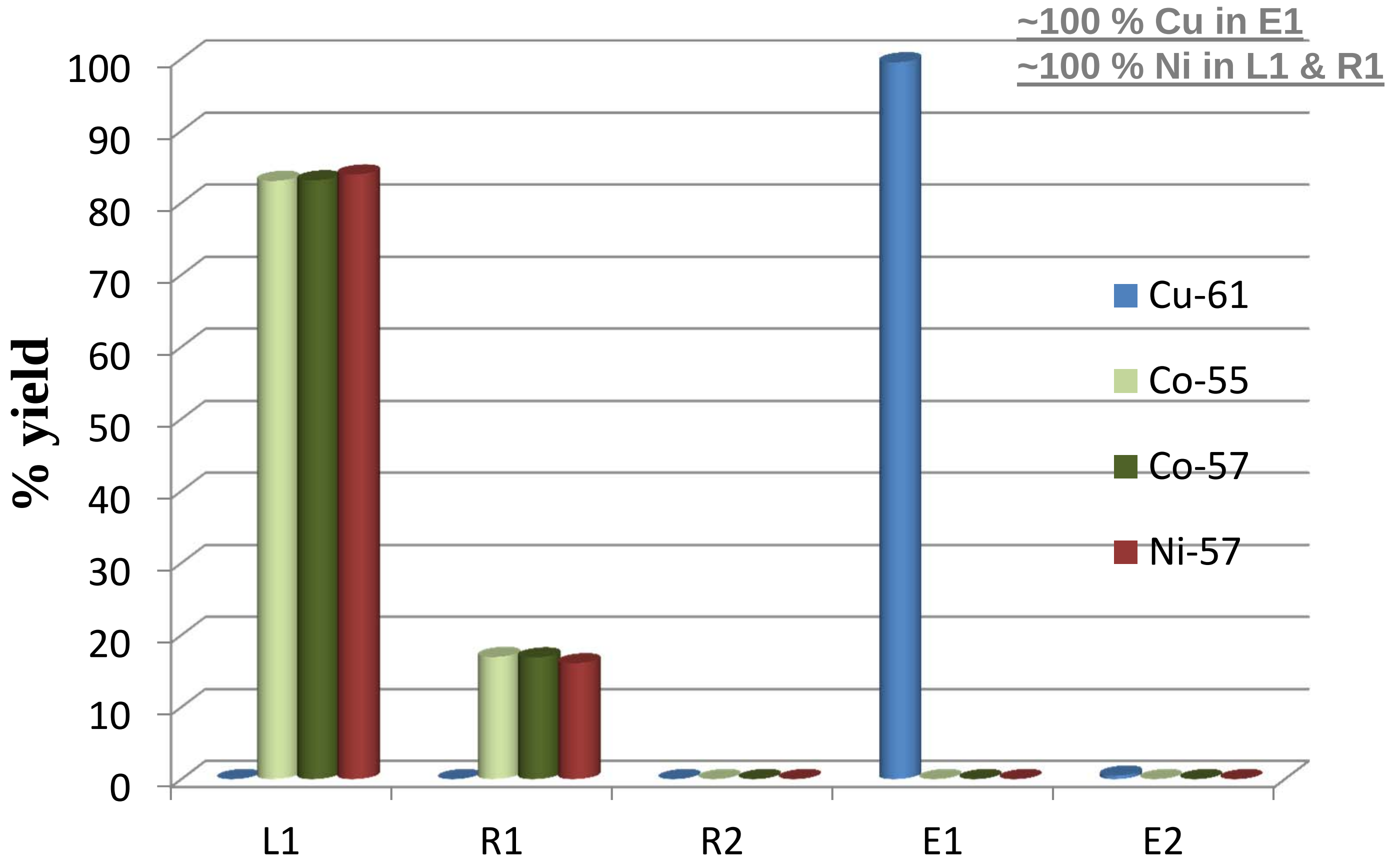


Figure 9 : Elution study, irradiated $Ni_{nat}$ target

**Loading:**

L1 : 5 mL HCl pH 2

**Rinsing**:

R1 : 10 mL HCl pH 2

R2 : 10 mL HCl pH 2

**Elution:**

E1 : 5 mL HCl **8 M**

E2 : 5 mL HCl **8 M**

- Very pure Cu fraction
- Elution and rinsing volumes to be optimized

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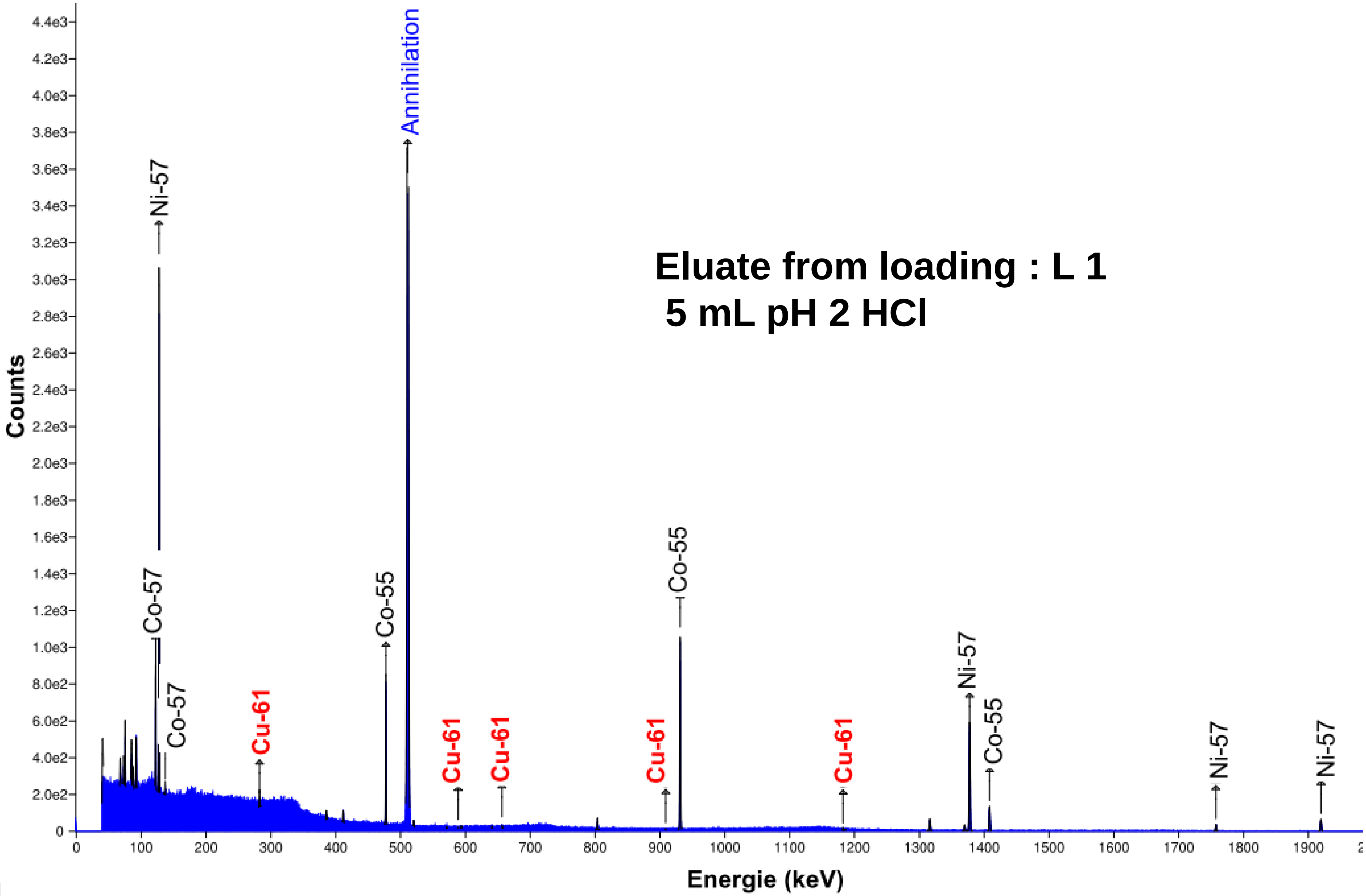
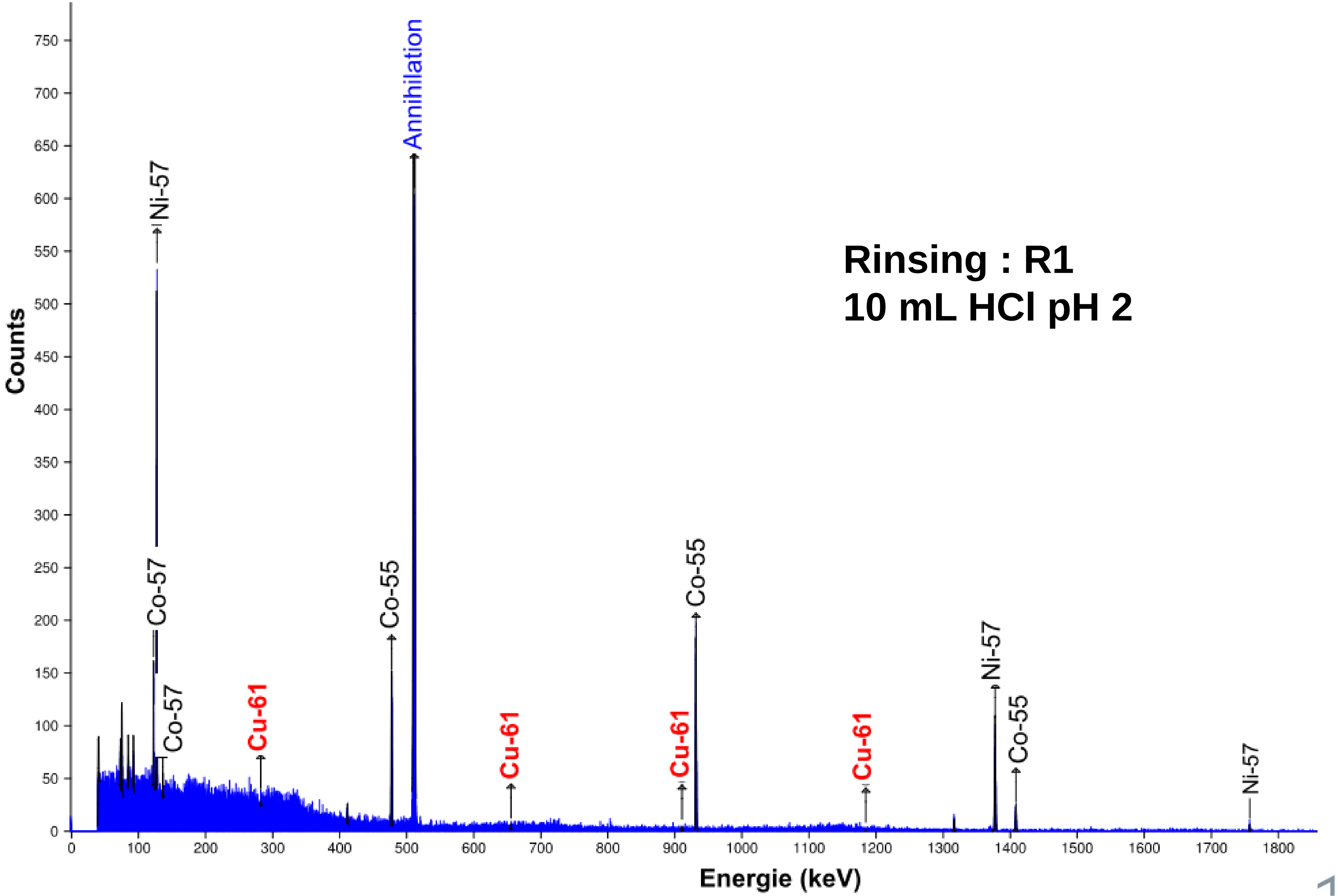


Eluate from loading : L 1
5 mL pH 2 HCl
Counts
Energie (keV)
Annihilation
Ni-57
Co-57
Co-57
Cu-61
Co-55
Cu-61
Cu-61
Cu-61
Co-55
Cu-61
Ni-57
Co-55
Ni-57
Ni-57

**Rinsing : R1**
**10 mL HCl pH 2**

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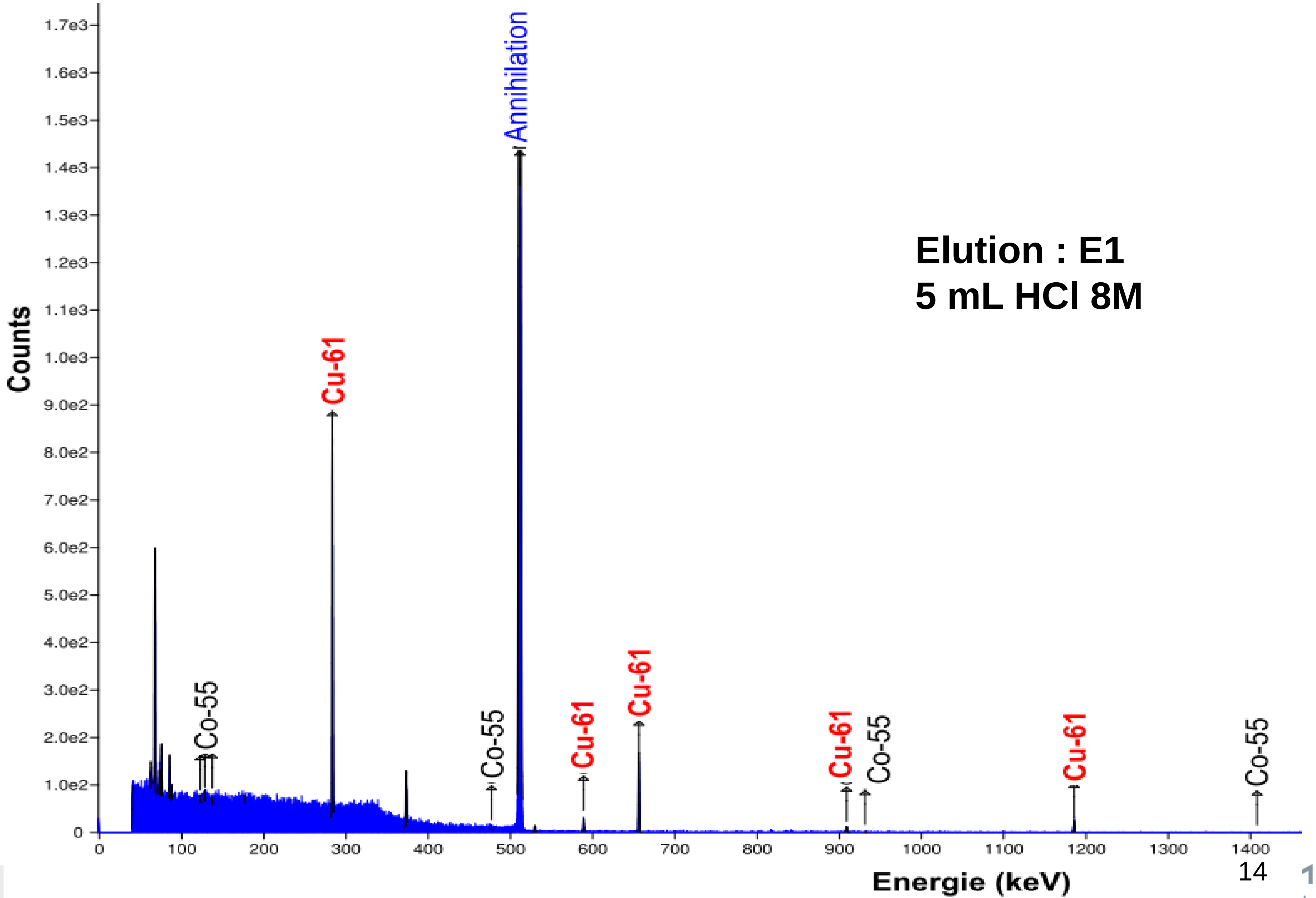
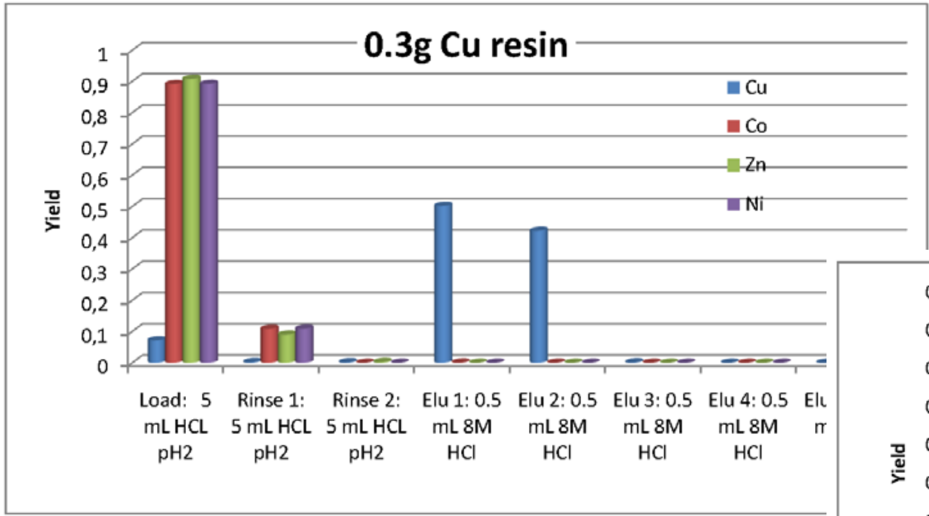

Elution : E1
5 mL HCl 8M
Counts
Energie (keV)
Annihilation
Cu-61
Cu-61
Cu-61
Cu-61
Cu-61
Co-55
Co-55
Co-55
Co-55
1.7e3
1.6e3
1.5e3
1.4e3
1.3e3
1.2e3
1.1e3
1.0e3
9.0e2
8.0e2
7.0e2
6.0e2
5.0e2
4.0e2
3.0e2
2.0e2
1.0e2
0
0
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400

# Optimization of Cu elution volume



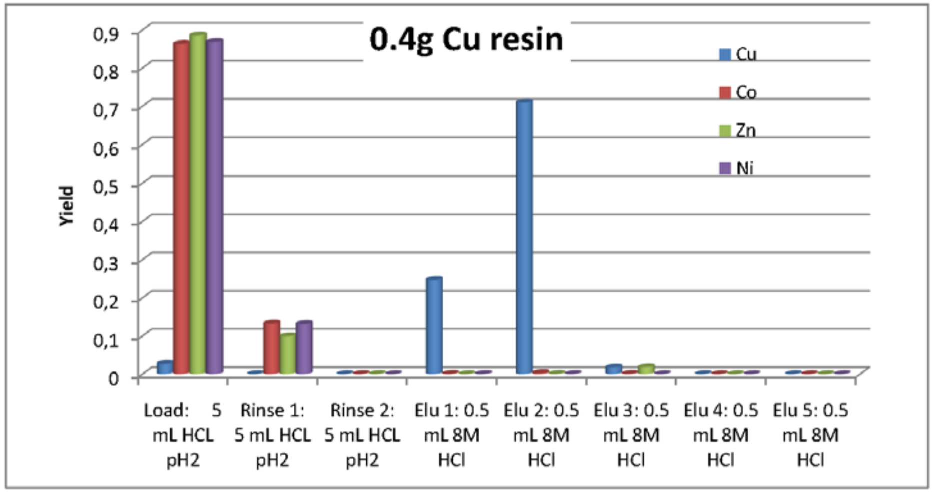




Figure 10/11: Elution study, simulated Ni target, varying Cu resin amounts, gravity flow

- Use of smaller columns allows reducing Cu elution volume
  - Near quantitative Cu recovery in ~1 mL 8M HCl
- No impact on Cu purity

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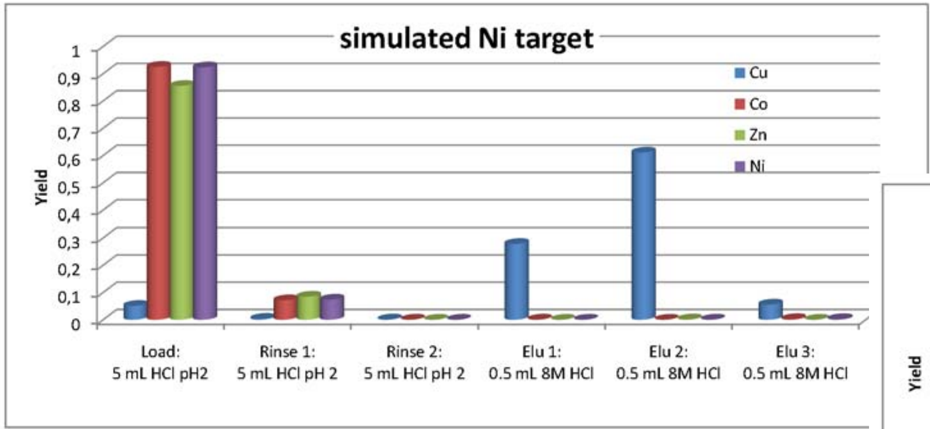
# Optimization of Cu elution volume – vacuum-assisted flow



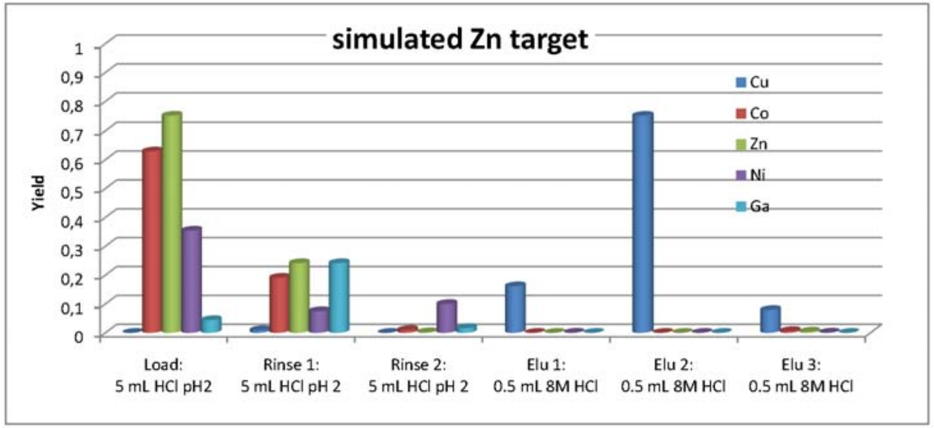




Figure 12/13 : Elution study, simulated Ni and Zn target, 350 mg Cu resin

- Flow rate: 1 mL/min (rinsing up to 6 mL/min)
- 0.35g CU resin columns
- > 90% recovery in 1 mL 8M HCl, quantitative recovery in 1.5 mL
- Simulated Ni and Zn targets (200 mg target material)
- No impact on purity

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# Optimized method

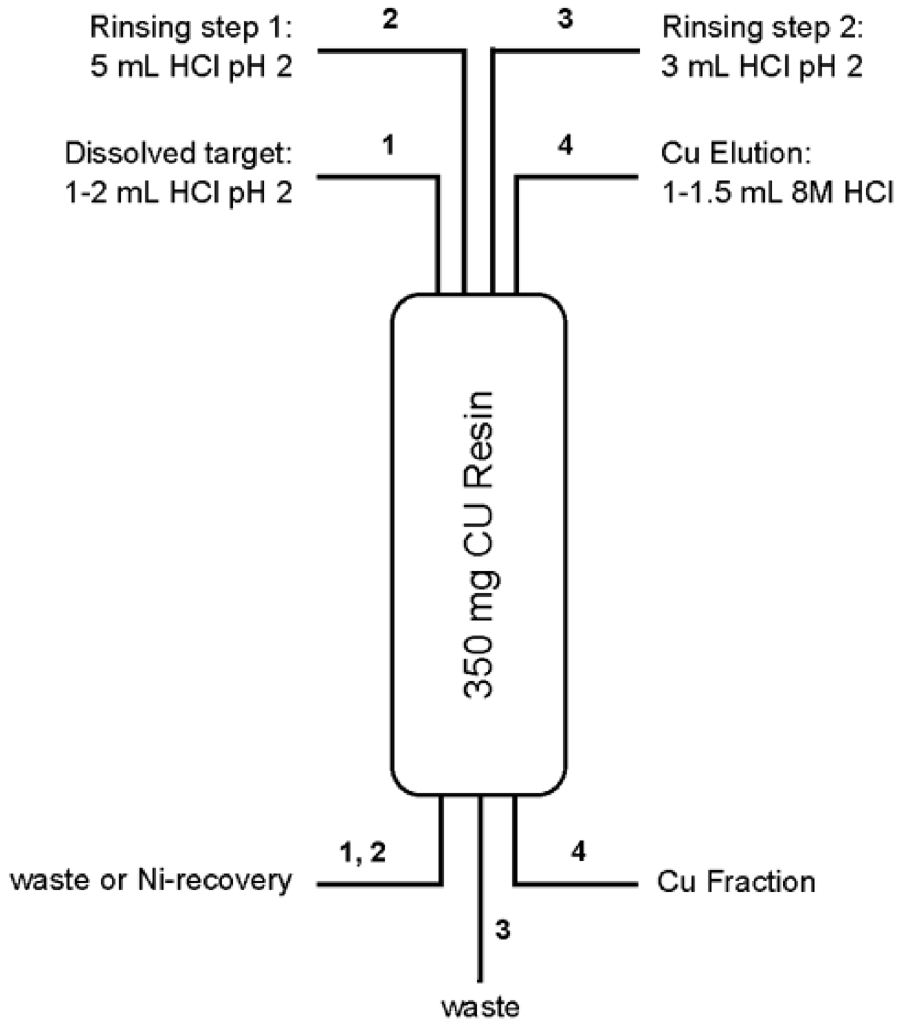


Figure 14: Scheme of elution conditions – optimized method

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# Decontamination factors $D_f$

- Flow rate: 1 mL/min (rinsing 6 mL/min)
- 0.35 g columns
- Loading solution: Ni, Zn, Co, Ga and Au in 5 mL HCl pH 2
- Separation following optimized method
- ICP-MS measurement
- Calculation of deconfactors $D_f$ for Cu fractions
  - Fraction E1 (0.5 mL 8M HCl):
    - $D_f$: Ni, Co & Zn > 20 000
    - $D_f$: Au & Ga > 10 000
  - Fraction E2 (0.5 mL 8M HCl):
    - $D_f$ : Ni > 20 000, Co > 40 000, Zn > 70 000, Au > 50 000, Ga > 10 000

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# Conclusion II

- 350 mg columns allow for Cu elution in small volume
- Vacuum assisted flow
- Rapid separation
- Quantitative recovery of Cu in 1 – 1,5 mL 8M HCl
  - Cu yield > 90% in 1 mL 8M HCl
  - 97,6% ± 2,3% (k = 1, N=25) in 1.5 mL 8M HCl
- Pure Cu fraction
  - $D_f$ (ICP-MS)
  - $\gamma$-spectrometry
- Obtained Cu suitable for labelling (ARRONAX)
- Ni recovered in small volume of 8M HCl
  - 10 – 13 mL load and rinse
  - Further purification for reuse e.g. via diect load on AIX

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# Conversion of Cu eluate

- Aim: recovery of Cu in small volume of dilute HCl, water or NaCl solution
- Anion exchange resins (AIX) shows necessary selectivity
- Cu eluate (1 – 1,5 mL 8M HCl) from Cu resin column directly loaded onto small AIX column
- Rinse with 8M HCl
- Elution with deion. water

# Elution study - AIX

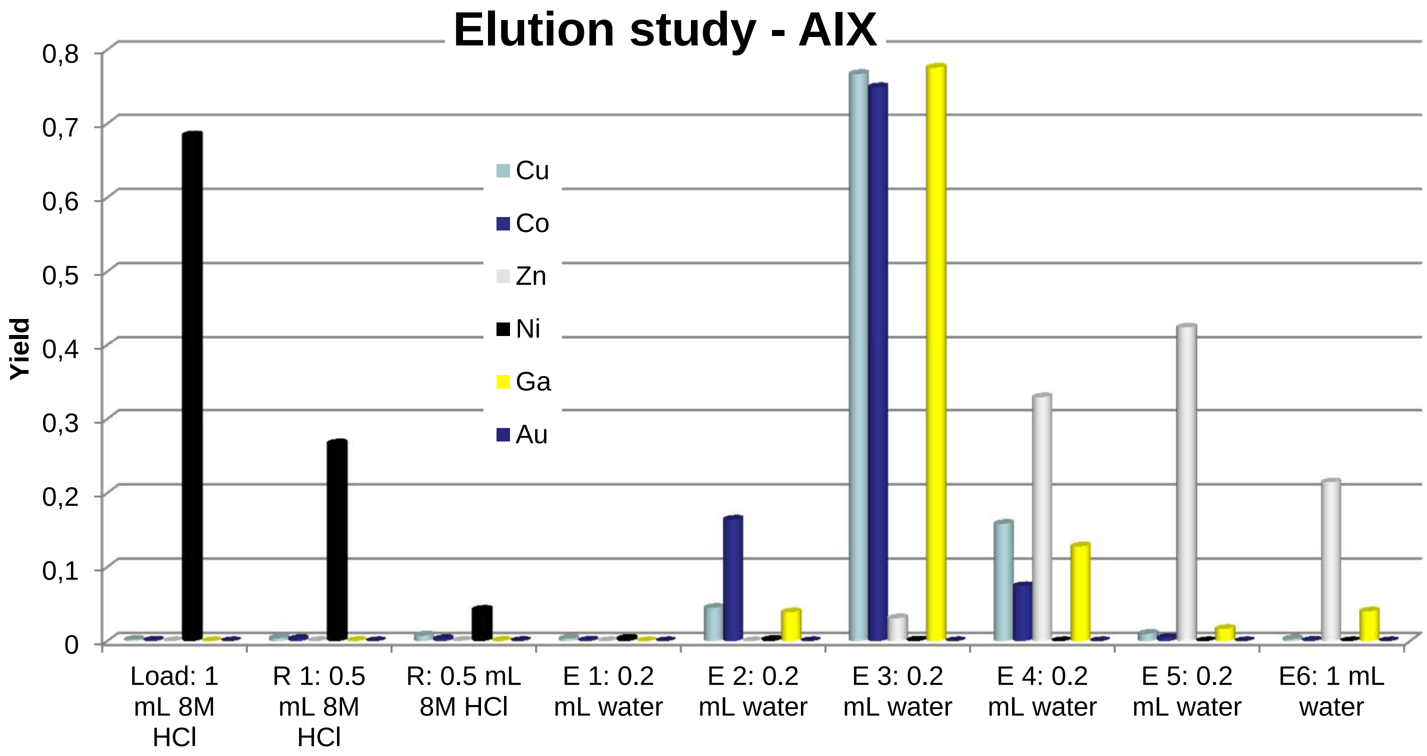


Figure 15: Elution study, AIX, various elements, 400 mg A8 Resin (200 – 400 mesh, Eichrom Technologies)

- 400 mg AIX (A8)
- Cu elution in 0.6 – 0.8 mL water
- Anion exchange conversion step gives additional decontamination from Ni, Zn, Au

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# Cu eluate conversion step

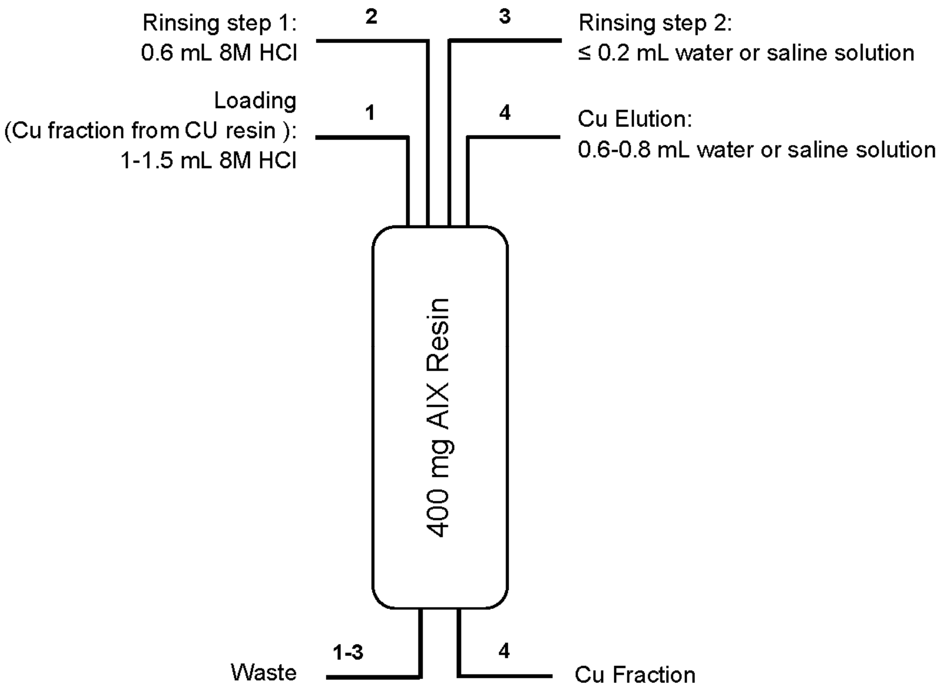


Fig 16: Conversion step using anion exchange, 400 mg A8 resin (Eichrom Technologies), 200 – 400 mesh

- Direct load of Cu eluate onto anion exchange resin / elution with water or saline solution
- Further purification from matrix elements and organics
- Cu yield in 0.7 mL water: 93,8 ± 6,4% (k = 1, N=25)

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# Full method

- Vacuum-assisted flow
- CU resin (350 mg)
  - Load from 1 - 2 mL HCl pH2
  - Rinse with 5 mL and 3 mL HCl pH 2
  - Load and rinse contain ~100% Ni ($\rightarrow$ Ni-64 recovery)
  - Cu elution in 1 – 1.5 mL 8M HCl
- Cu yield > 95%; high decontamination factors
- Conversion on AIX (400 mg)
  - Load from 1 – 1.5 mL 8M HCl
  - Rinse with 0.6 mL 8M HCl and < 0.2 mL water
  - Cu elution in 0.6 – 0.8 mL water (or saline solution)
- Cu yield > 90%; add. decontamination (Ni, Zn, Au, organics)
- Overall separation time: <10 minutes

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# Other applications

- Removal of trace Cu-64 before Ni-64 plating
- Cu concentration and purification for analytical purpose (e.g. Cu in sea water)

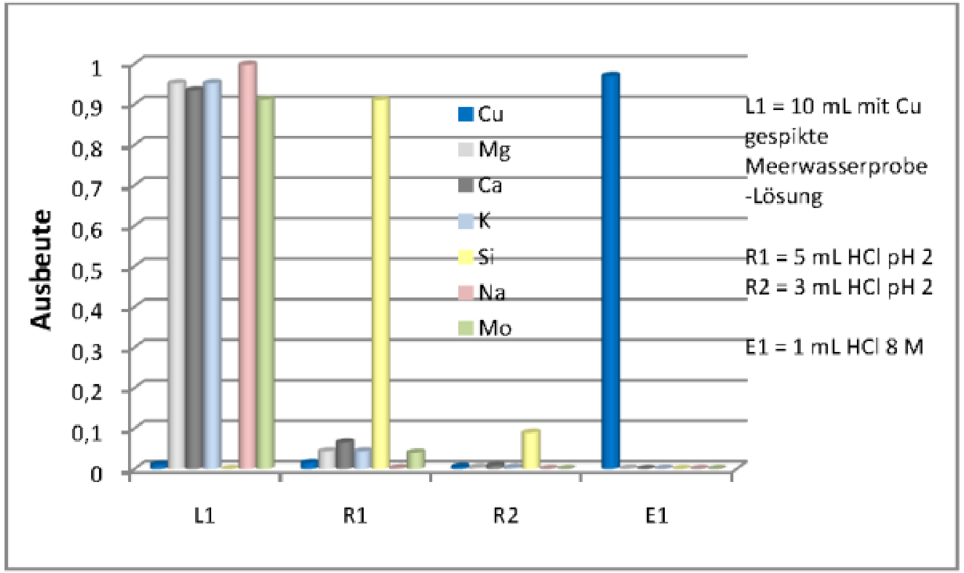


Fig 17: Elution study, 10 mL spiked sea water, 350 mg CU resin column, vacuum assisted flow

- ➢ 10 mL sea water (pH 2.3)
- ➢ Cu yield > 95% in 1 mL 8M HCl
- ➢ Pure Cu fraction

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