ON-GOING PROJECTS
R&D AND TECHNICAL SUPPORT

« BITS'N PIECES »

S. Happel

TrisKem Users Group Meeting

14th of September 2010

Chester (UK)

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Outline

Results of some on-going projects

- Ga-68 purification
- Zr separation
- Sn separation

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Ga purification

- Context: purification of Ga-68 obtained from Ge-68/Ga-68 generator
- Too high levels of Fe and Zn in Ga-68 fraction

- Literature:
  - Cation exchange
  - Anion exchange
  - UTEVA (McAlister/Horwitz)

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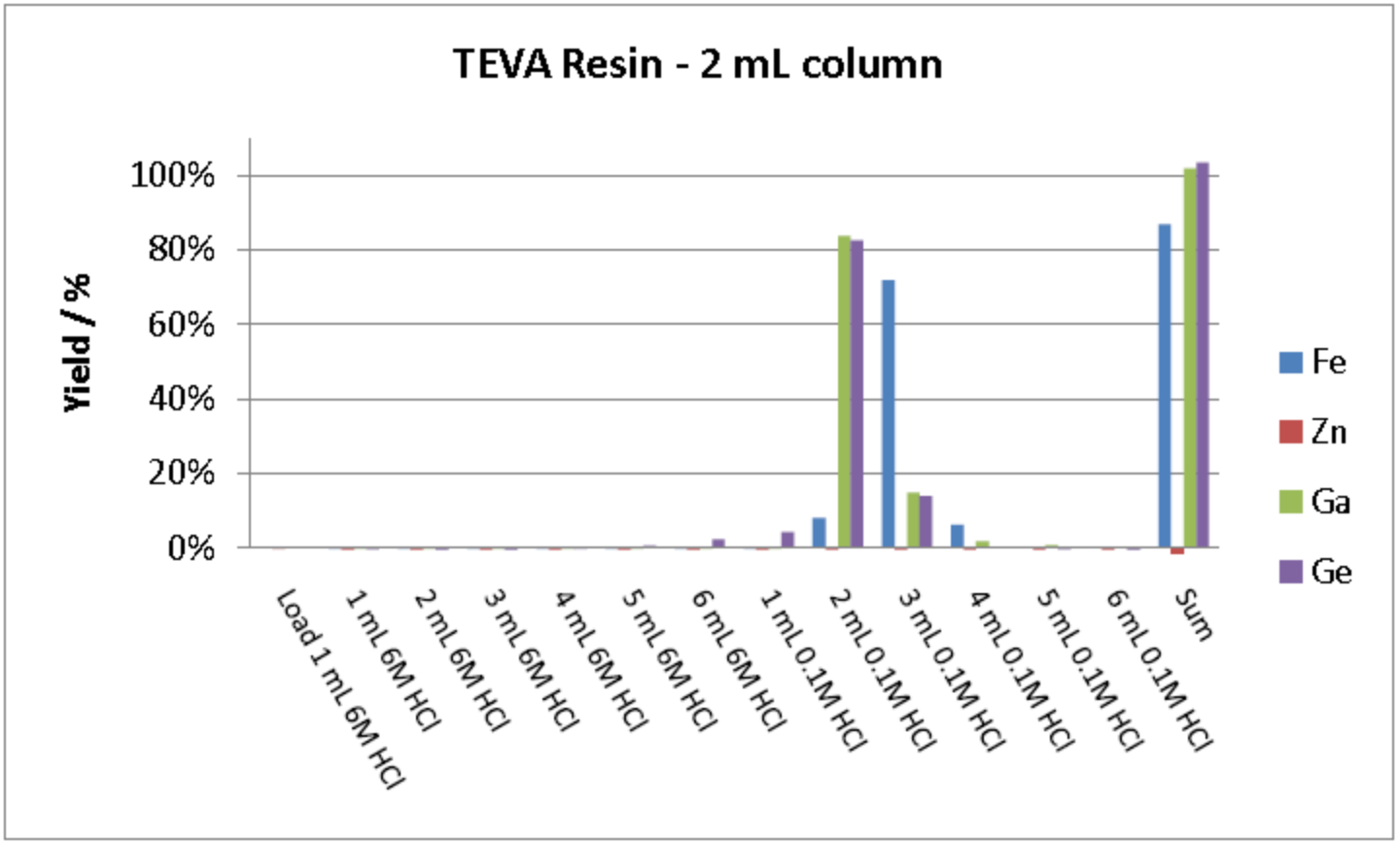
Ga purification



Elution study 2 mL TEVA column; 6M HCl, Elution condition: 0.1M HCl

- **Good separation from Zn (stays on column)**
- **No separation from Ge, little separation from Fe**
  - **Fe separation improvement would lead to Ga loss**

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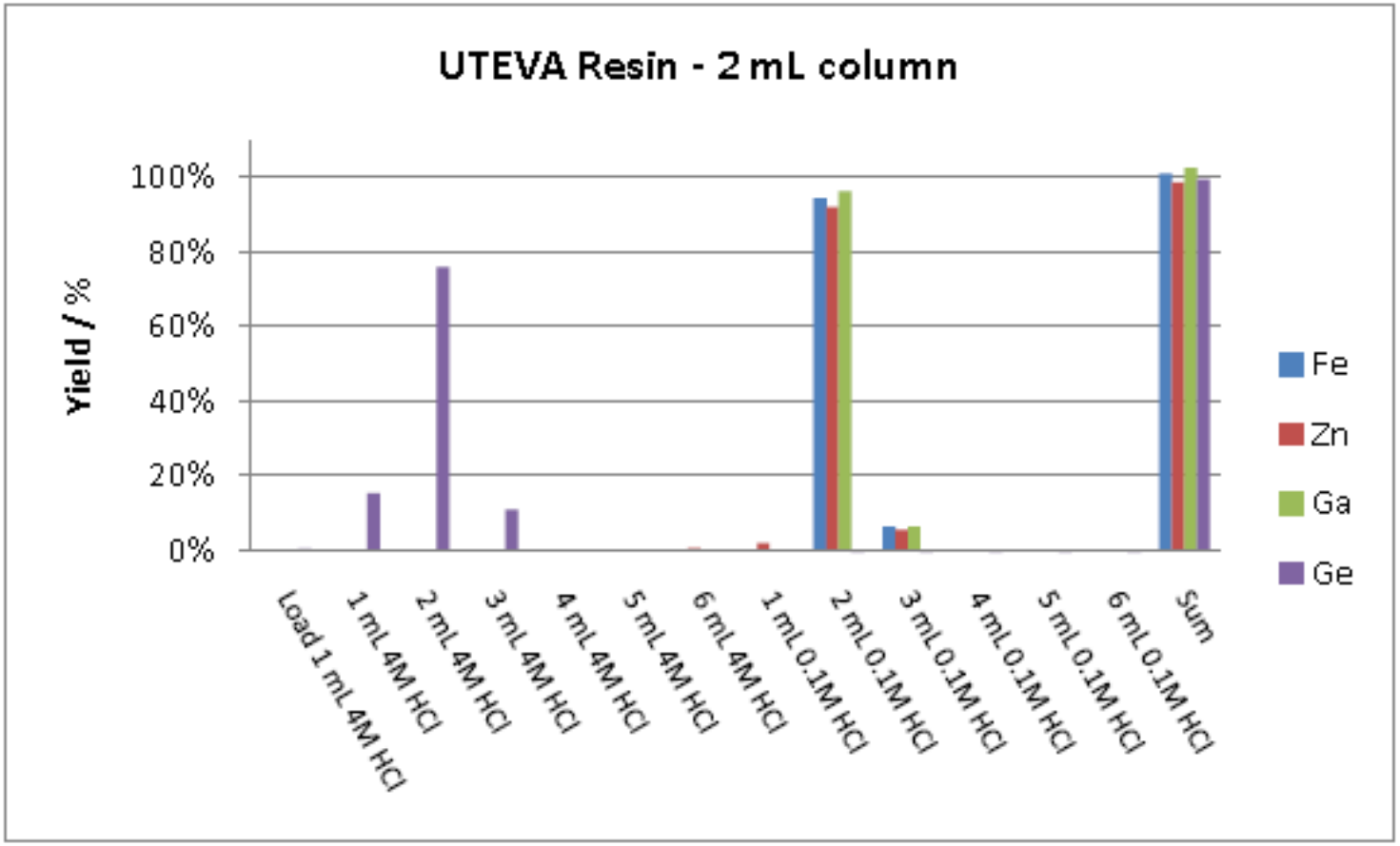
Ga purification



Elution study 2 mL UTEVA column; 4M HCl, Elution condition: 0.1M HCl

- **Good separation from Ge**
- **No separation from Zn and Fe**

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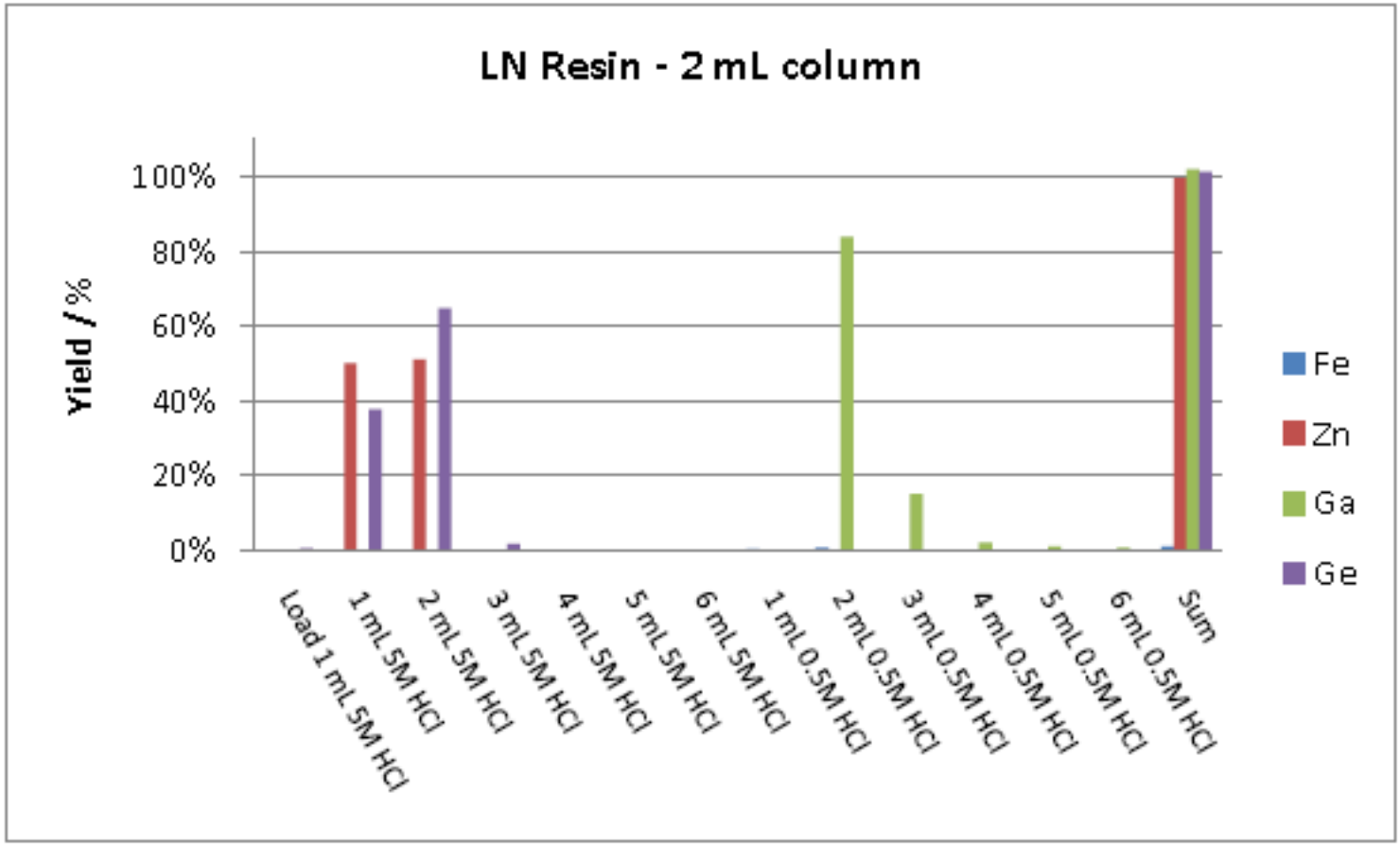
Ga purification



Elution study 2 mL LN column; 5M HCl, Elution condition: 0.5M HCl

- **Good separation from Ge, Fe (remains on resin) and Zn**
- **> 80% Ga in 1 mL 0.5M HCl**
- **Quantitative elution in 2 mL 0.5M HCl**

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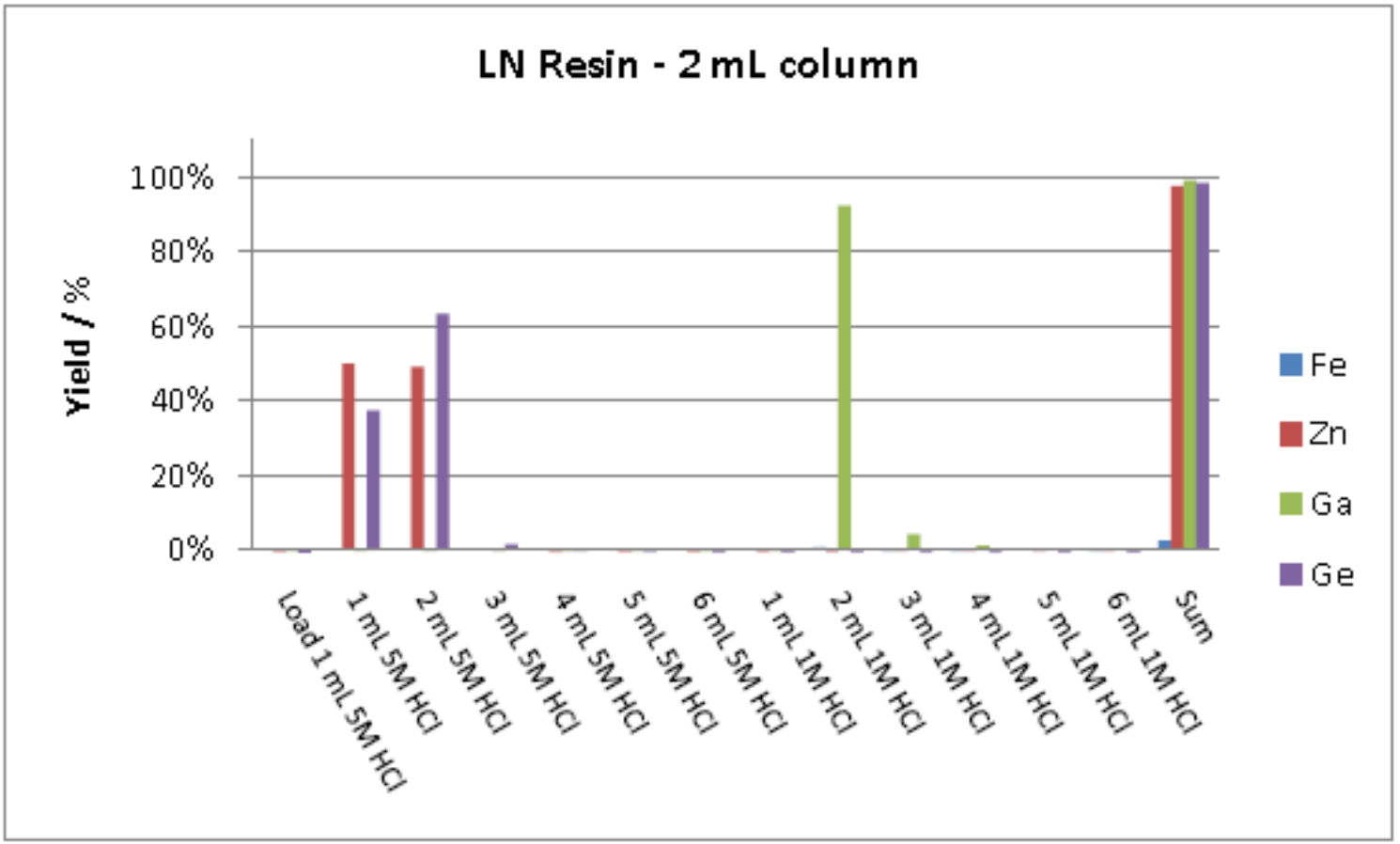
Ga purification



Elution study 2 mL LN column; 5M HCl, Elution condition: 1M HCl

- **Good separation from Ge, Fe and Zn**
- **> 95% Ga in 1 mL 1M HCl**
- **Quantitative elution in 2 mL 1M HCl**

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Zr-93

- Long-lived radionuclide
- Decommissioning and radioactive waste
- LSC or ICP-MS ($\rightarrow$ Nb-93!)
- Combination of literature methods
  - Vajda et.al. (UTEVA)
  - Le Fèvre/Pin (UTEVA)
  - Bombard et. al. (TEVA)
  - NAS (AIX)

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Zr separation via UTEVA/TEVA

- 1st separation on UTEVA
  - Advantage: no selectivity for Fe
- Method following Vajda et. al.
  - Modified elution
  - Important for loading solution: quantitative F^- removal / masking!
- Load: 5mL 9M HNO_3 / 100 µL H_2O_2
  - Multi-element solutions approx. 100µg per element
- Rinse: - 5 mL 9M HNO_3 / 50 µL H_2O_2
  - 2 x 5 mL 9M HNO_3
  - 5 mL 5M HNO_3
- Elution: 4 x 5 mL 0.01M HNO_3 / 0.1M oxalic acid
- All fractions analysed by ICP-MS

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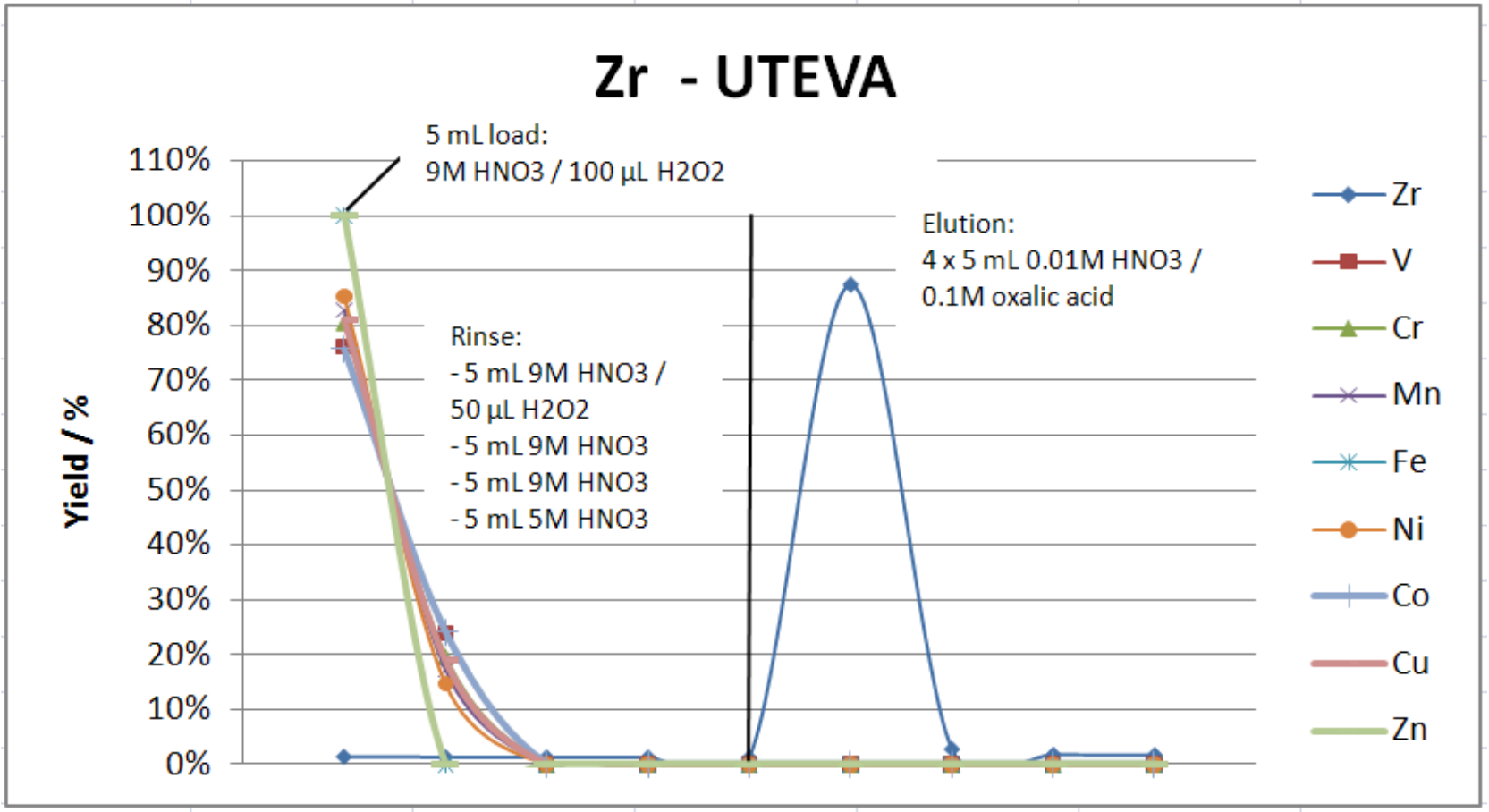
Zr separation on UTEVA



- **Zr elution works fine**
  - **Near quantitative recovery in 5 mL 0.01M HNO_3 / 0.1M oxalic acid**

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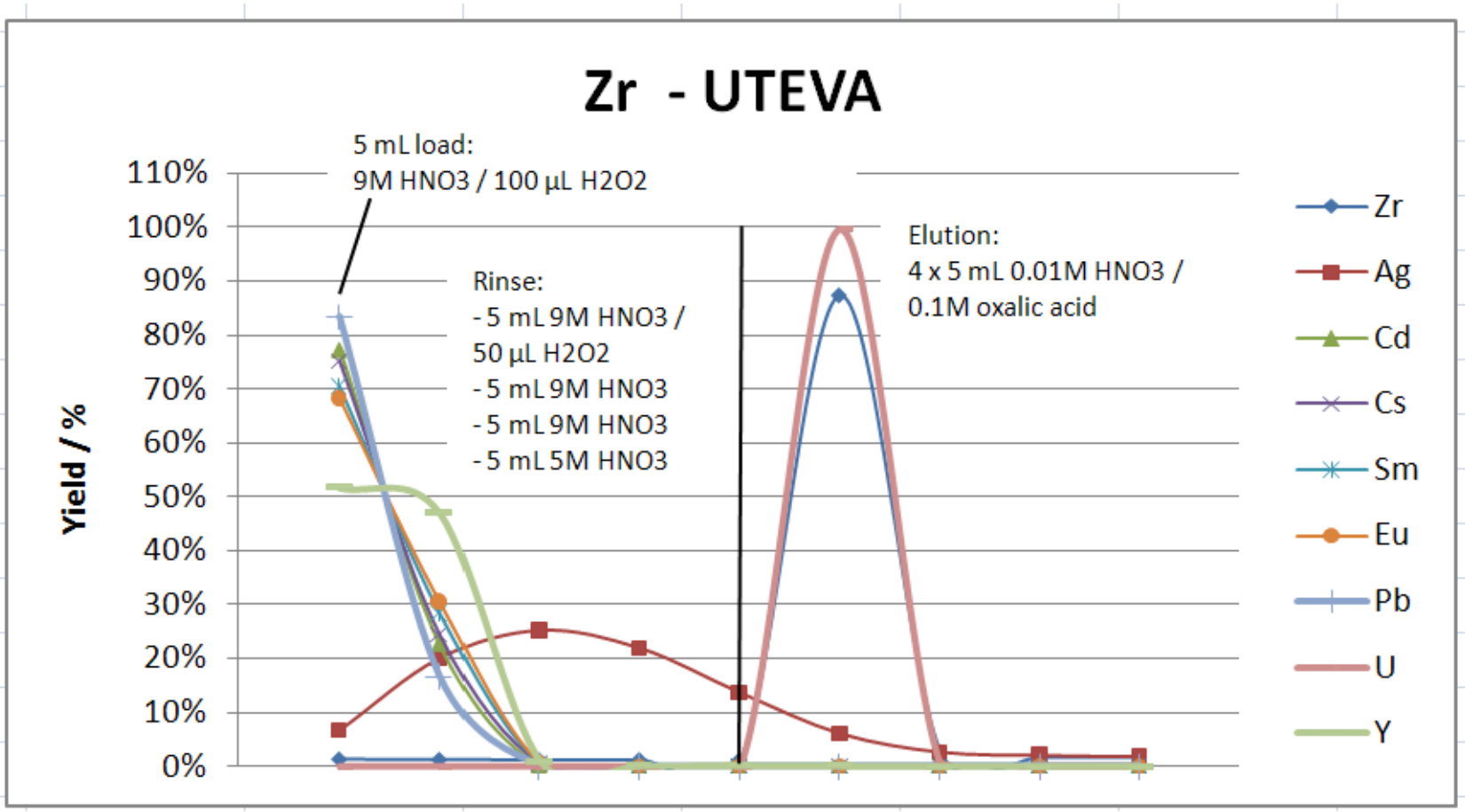
Zr separation on UTEVA



- No Zr / U separation
- Ag separation not quantitative

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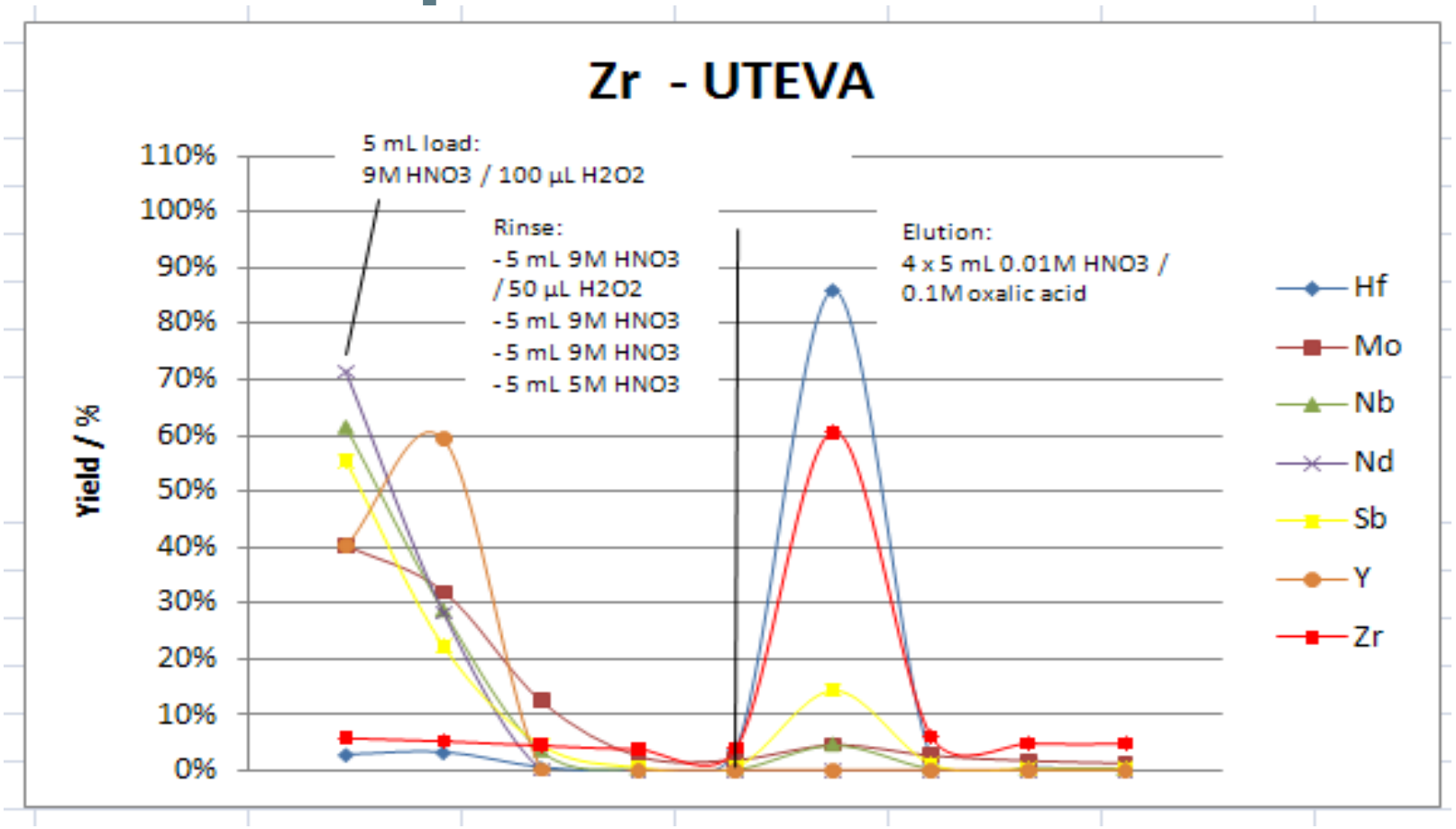
Zr separation on UTEVA



- No Zr / Hf separation
  - Not conform with literature….
- Sb(III) separation to be confirmed
- Good Nb separation

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Zr separation on TEVA – UTEVA eluate

- 2nd separation on TEVA
  - Advantage: different selectivity than UTEVA
- Method following Bombard et. al.
  - Modified elution
- Column preconditioning: 8M $LiNO_3$ / 0.01M HNO_3
- Load: 3mL 9M HCl
  - UTEVA eluate from first test evaporated and converted to 9M HCl
- Rinse: 2 x 1 mL 9M HCl
- Elution: 3 x 5 mL 0.05M HCl
- All fractions analysed by ICP-MS
- Additionally applied to multi-element solution

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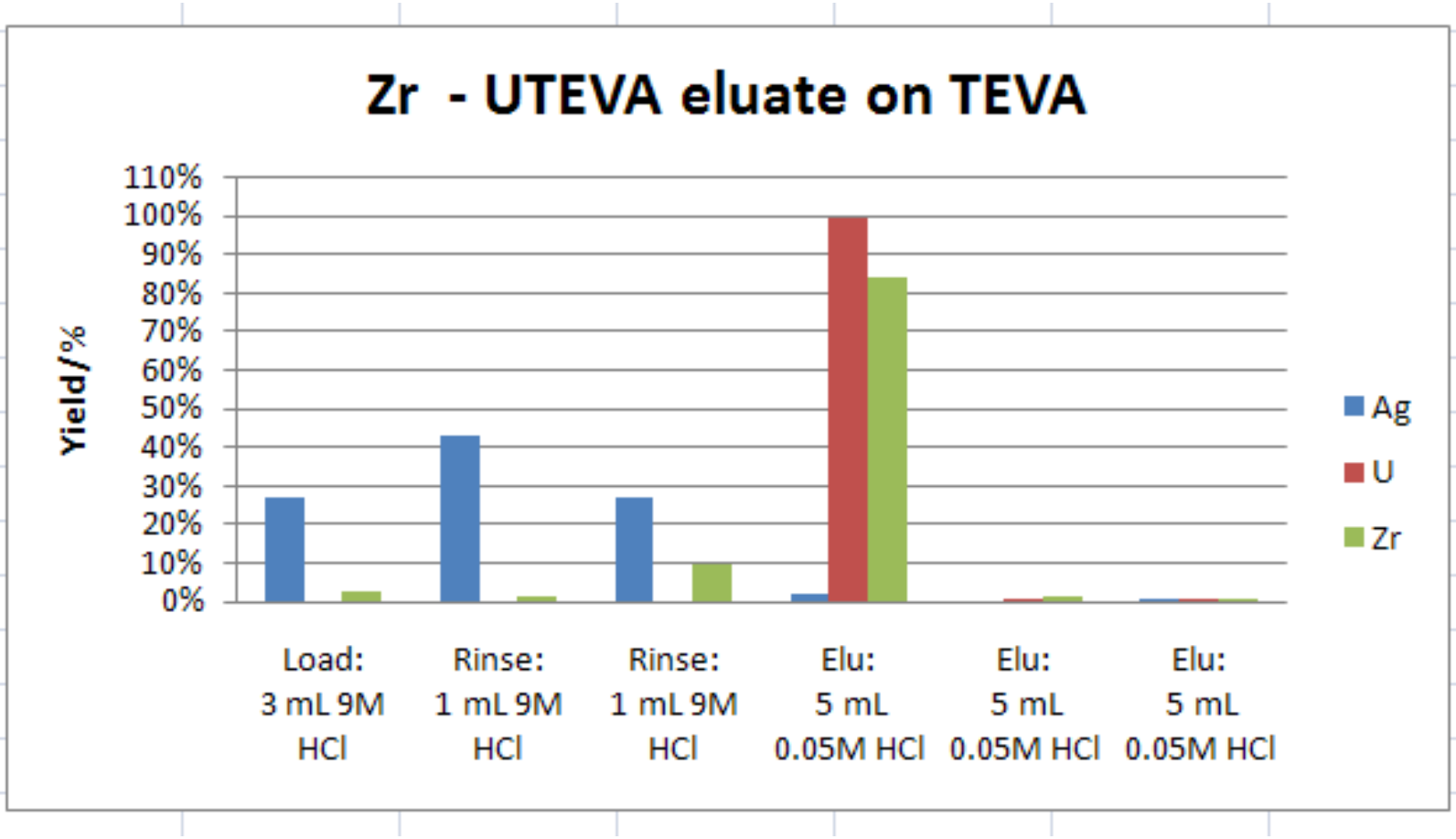
Zr separation on TEVA



- Good separation from Ag
- No separation from U
  - U/Zr separation already on UTEVA
  - Modification of Zr elution from TEVA

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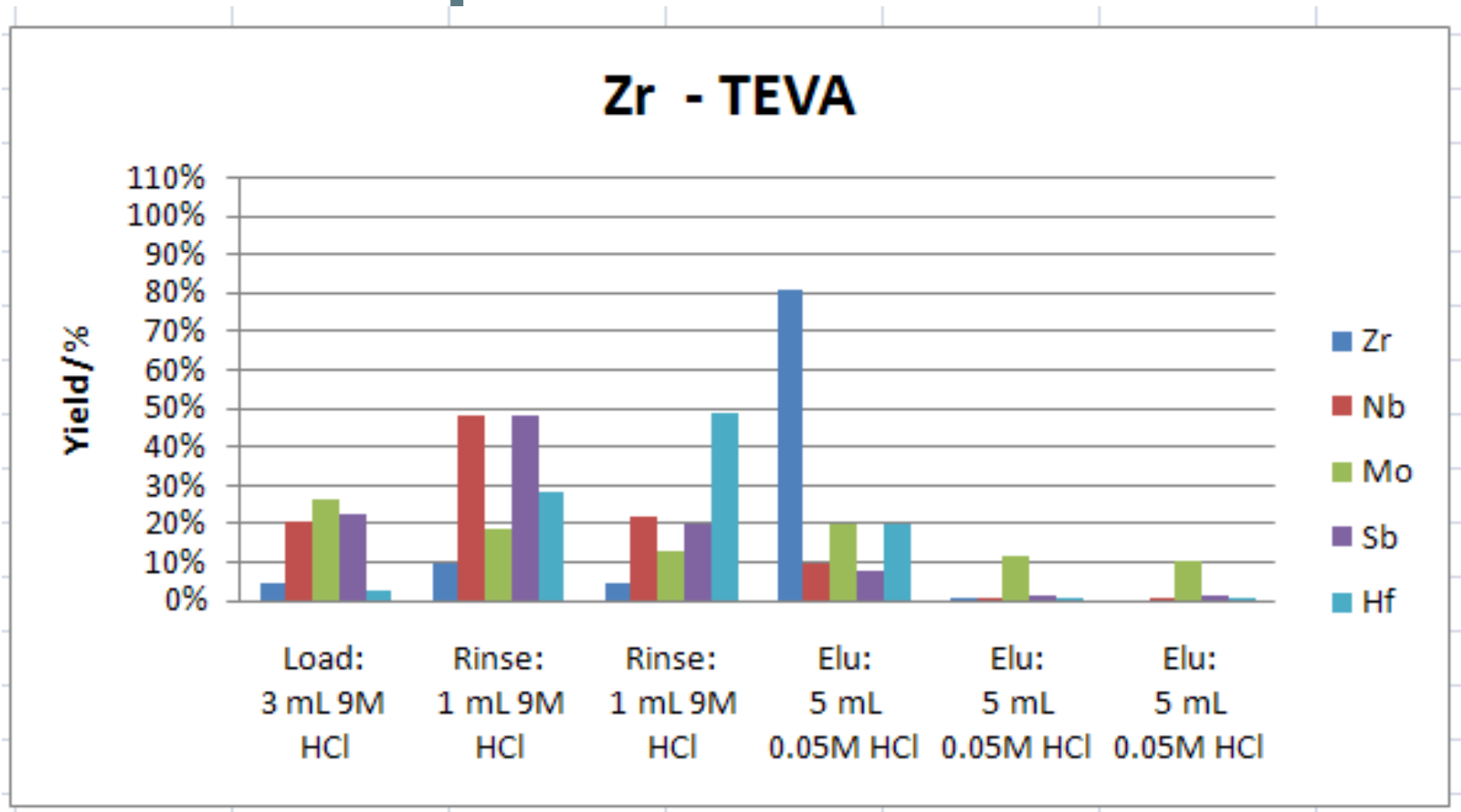
Zr separation on TEVA



- Separation of Nb, Mo, Sb and Hf from Zr problematic under chosen conditions
  - Hf and Sb separation should be improved by increase of 9M HCl rinsing step volume
  - Mo behavior not conform with literature – control of oxidation state

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Zr separation on UTEVA – modified elution

- No Zr/U separation with 0.01M HNO_3 / 0.1M oxalic acid
- Horwitz et al. show that Zr can be eluted with 2M HNO_3 / 0.02 M oxalic acid while U stays on UTEVA

- Load: 5mL 9M HNO_3 / 100 µL H_2O_2
  - Multi-element solutions approx 100µg per element
- Rinse: - 5 mL 9M HNO_3 / 50 µL H_2O_2
  - 2 x 5 mL 9M HNO_3
  - 5 mL 5M HNO_3
- Elution: 4 x 5 mL 2M HNO_3 / 0.03M oxalic acid

- All fractions analysed by ICP-MS

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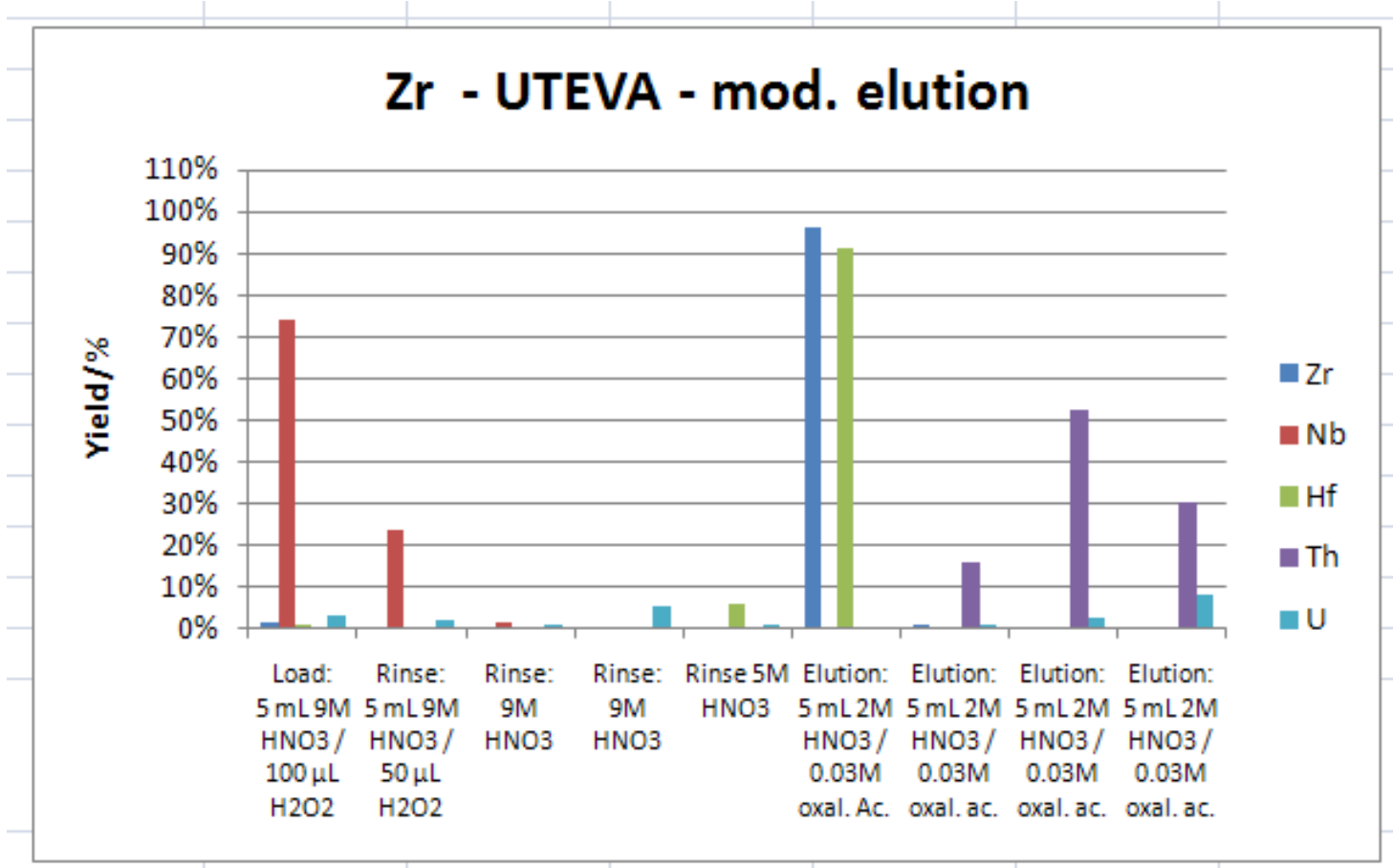
Zr separation via UTEVA/TEVA



- Good U/Zr separation
- Still no Zr/Hf separation
- Th separation delicate
- Zr elution in 5 mL 2M HNO_3 / 0.03M oxalic acid

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Zr separation on TEVA – modified elution

- NAS describes anion exchange method based on
  - Loading from 9M HCl
  - Zr Elution in 7M HCl
- Column preconditioning: 8M $LiNO_3$ / 0.01M HNO_3
- Load: 3mL 9M HCl
  - Multi-element solutions approx 100µg per element
- Rinse: 2 x 1 mL 9M HCl
- Elution: 4 x 5 mL 7M HCl
- All fractions analysed by ICP-MS

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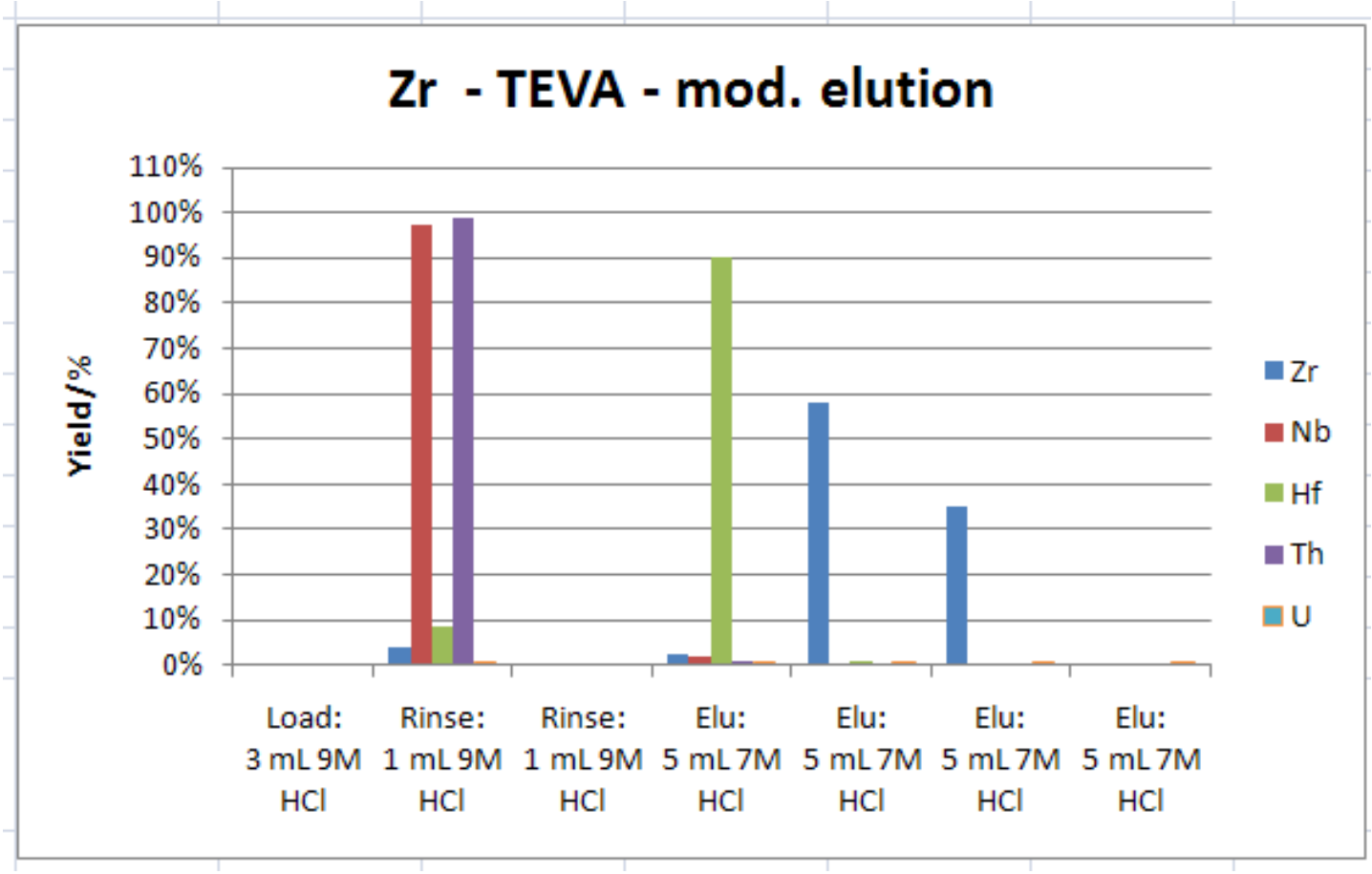
Zr separation on TEVA



- Th and U separation very good
- Interesting Zr/Hf elution behaviour
- Zr recovered in 10 mL 7M HCl
- To be confirmed…

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Conclusion

- Combining UTEVA and TEVA seems promising
- Method still under work, needs to be improved
- Influence of matrix and HF to be evaluated
- Separation on UTEVA:
  - Improvement of Zr/Hf separation
  - Confirmation of U and Th separation from Zr
  - Confirmation of Nb/Zr separation
  - Influence of HF and matrix…
- Separation on TEVA
  - Preconditioning with 9M HCl
  - Increased volume of 9M HCl rinsing step
  - Confirmation for Mo and Hf elution behaviour
  - Zr Elution via 0.05M HCl or 7M HCl

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Sn separation

- Context: Sn-121m in decommissioning samples
- High levels of matrix elements (incl. Ca and Fe)

- Literature:
  - Ikaza/Yata: TBP resin
  - Dissertation M. Langer: TRU resin
    - Load: 5mL 8M HCl (Fe and Sn retained, Co, Cd and Ni pass)
    - Rinse: 25 mL 8M HCl (removes remaining Co, Cd and Ni)
    - Rinse: 2.5M HCl (Fe elution)
    - Sn Elution with 20 mL 0.1M HCl

Sn separation – 1st Elution study

- Preparation of TBP resin

- 2 mL TBP resin column
- Preconditioned with 5 mL 2M HCl
- 5 mL sample solution containing (V, Cr, Mn, Fe, Ni, Co, Cu, Zn, Y, Ag, Cd, Sn, Sb, Cs, Ba, Nd, Sm, Eu, Pb and U - each approx 50µg) in 2M HCl
- Sample loaded onto TBP resin column – eluate collected
- Rinsing with 15 mL 2M HCl (collected in 1 mL steps)
- Elution with 20 mL 0.1M HCl (collected in 1 mL steps)

- All fractions measured by ICP-MS

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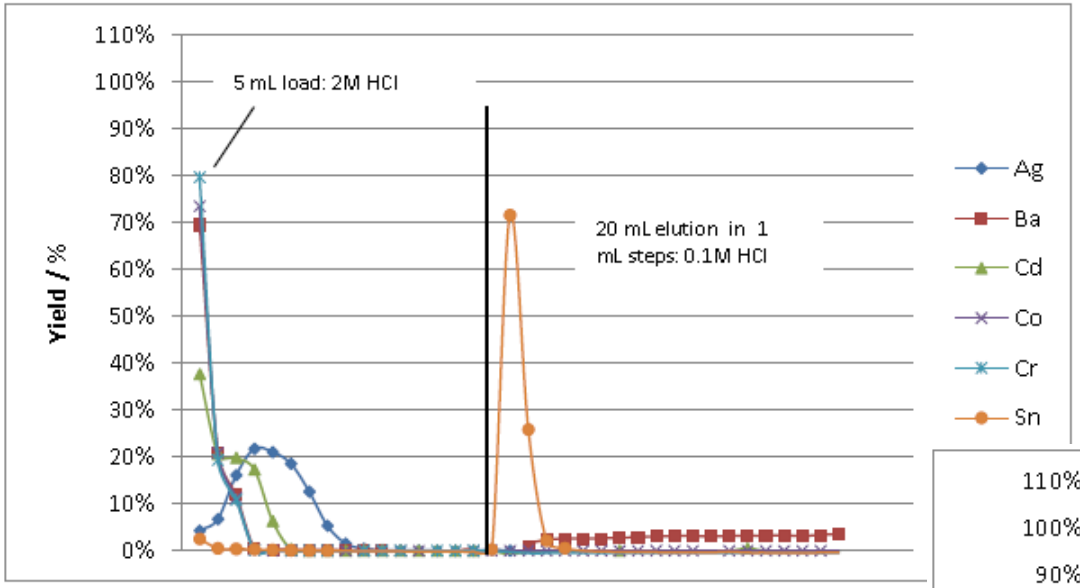
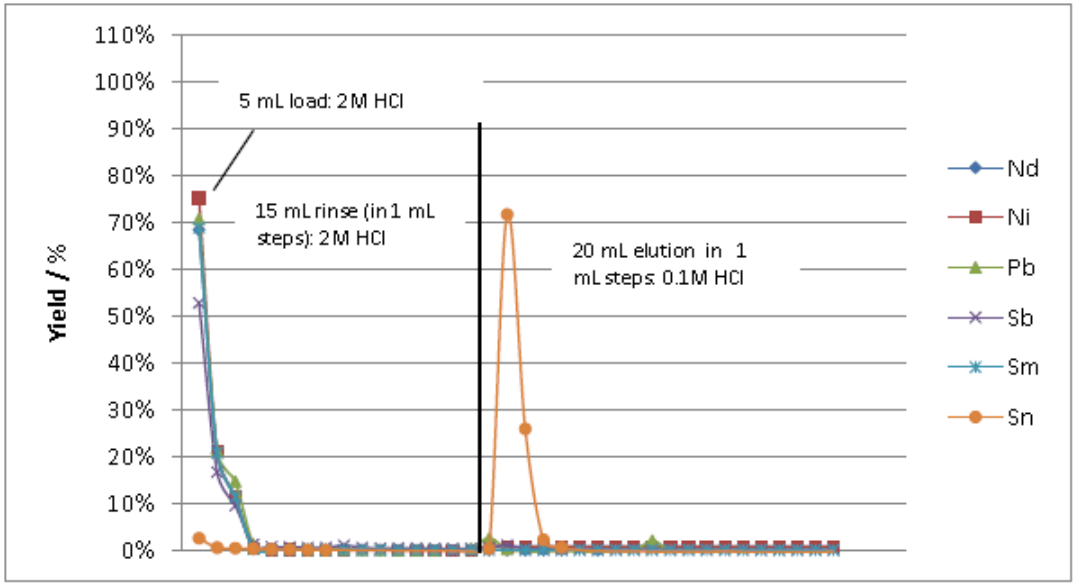
Results – 1st Elution study





- **Overall good selectivity for Sn, elution in 5 mL 0.1M HCl possible**

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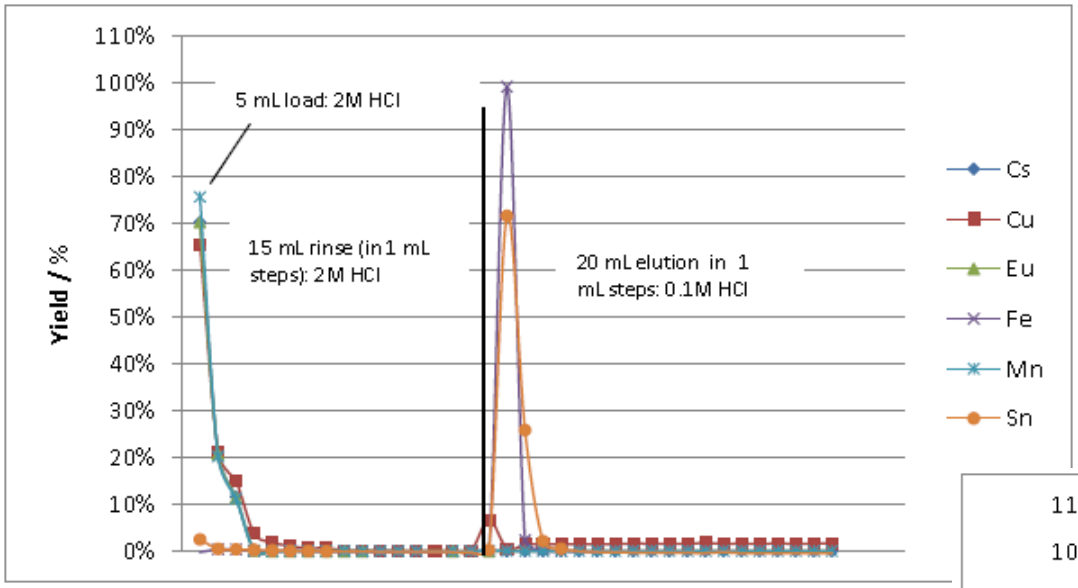
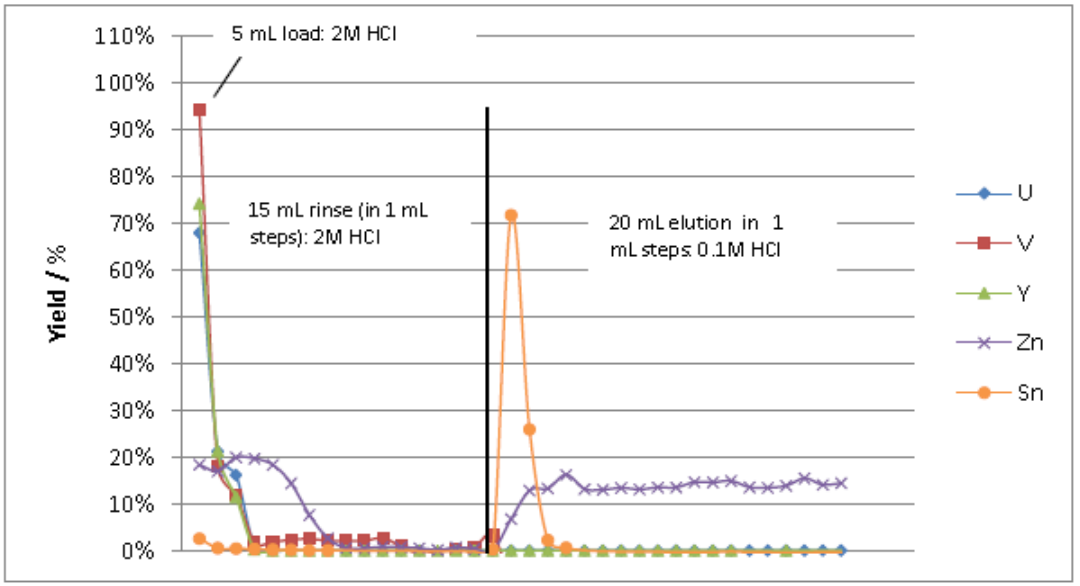
Results – 1st Elution study





- No Fe/Sn separation
- Zn separation to be improved….

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Sn - Fe / Zn separation

- Ghersini/Braun: In 0.1M H_2SO_4 TBP has very low selectivity for Fe and Zn, and high selectivity for Sn
- Potential method:
  - 2 mL column TBP resin
  - Preconditioned with 5 mL 2M HCl
  - 5 mL sample loading solution 2M HCl
  - Rinsing with 2 x 5 mL 2M HCl
  - Rinsing with 5 mL 0.1M H_2SO_4
  - Elution with 5 mL 0.1M HCl
- Test currently under way
- For iron/metal samples: load from 0.1M H_2SO_4, followed by rinse with 2M HCl to avoid saturation of column with Fe?
- Further testing of method
- Testing of TRU resin as described by M Langer

Very special thanks!!

- R. Streng
- C. Dirks
- A. Zulauf

at University Marburg

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