

Cs separations for candidate TK300 resin

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Analysis Requirements

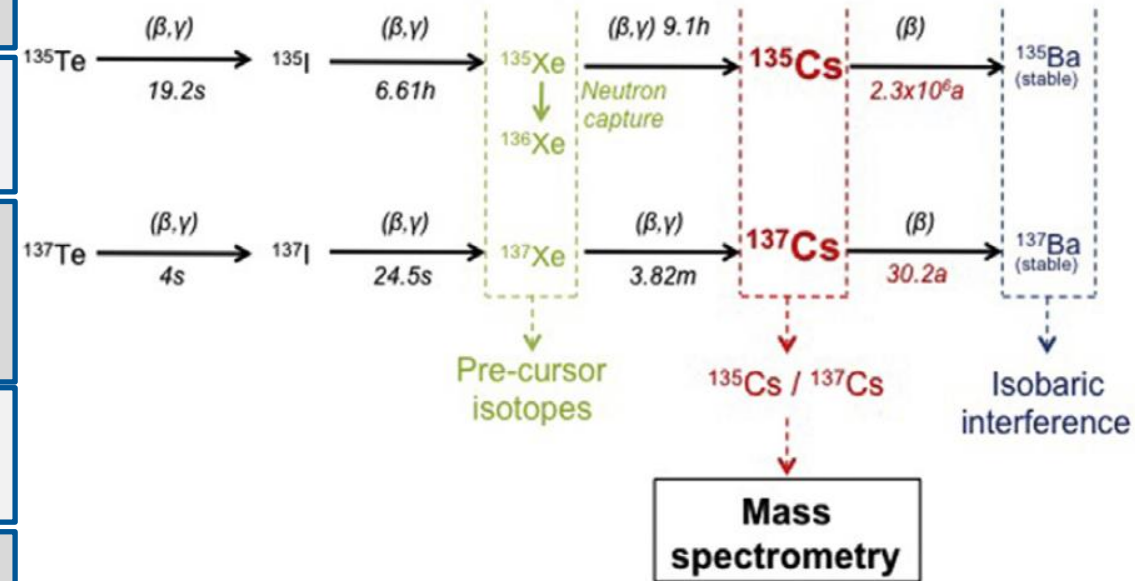
High yield (6.58 %) fission product

Nuclear Decommissioning

Environmental Monitoring (Routine and Radiological Incident)

Long-term Waste Monitoring e.g. Geological Disposal

Nuclear Forensics ($^{135}\text{Cs}/^{137}\text{Cs}$ ratios)



TK300

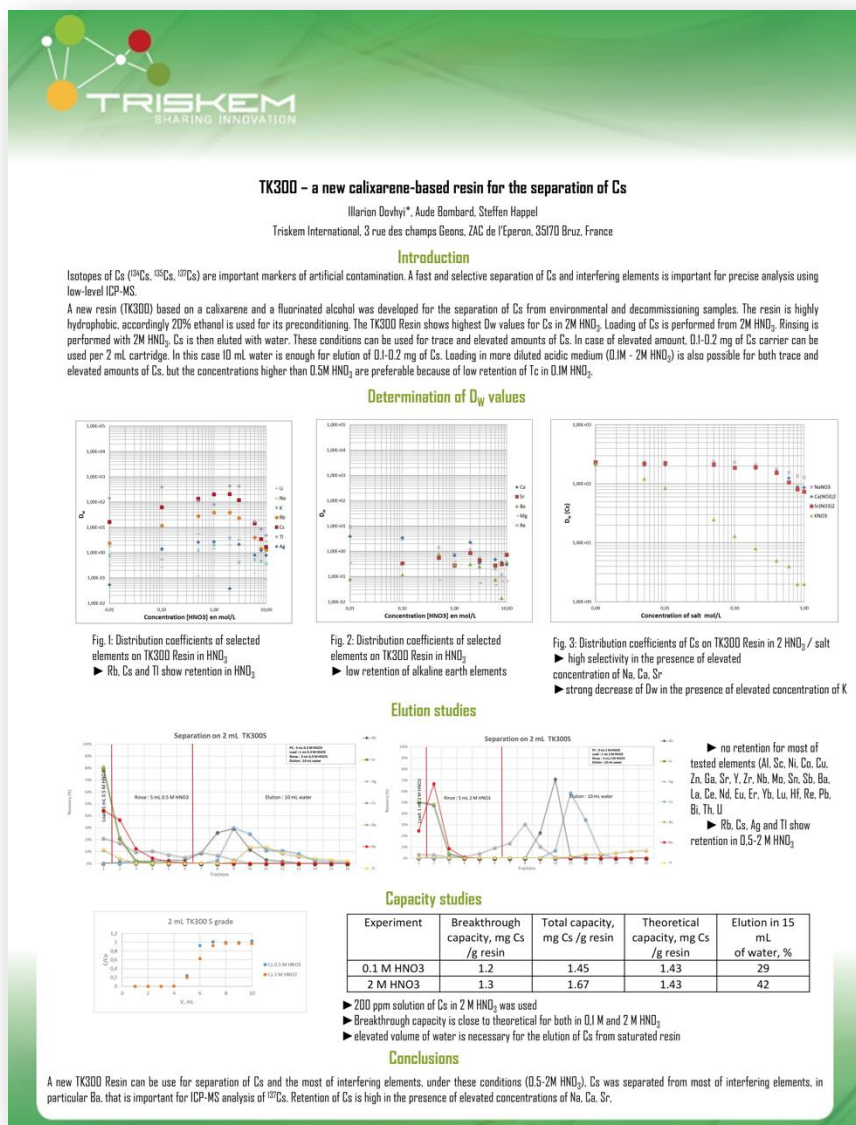
- New macrocycle-based resin for selective separation of Cs
 - Based on calixarene and a fluorinated alcohol
 - Resin is hydrophobic



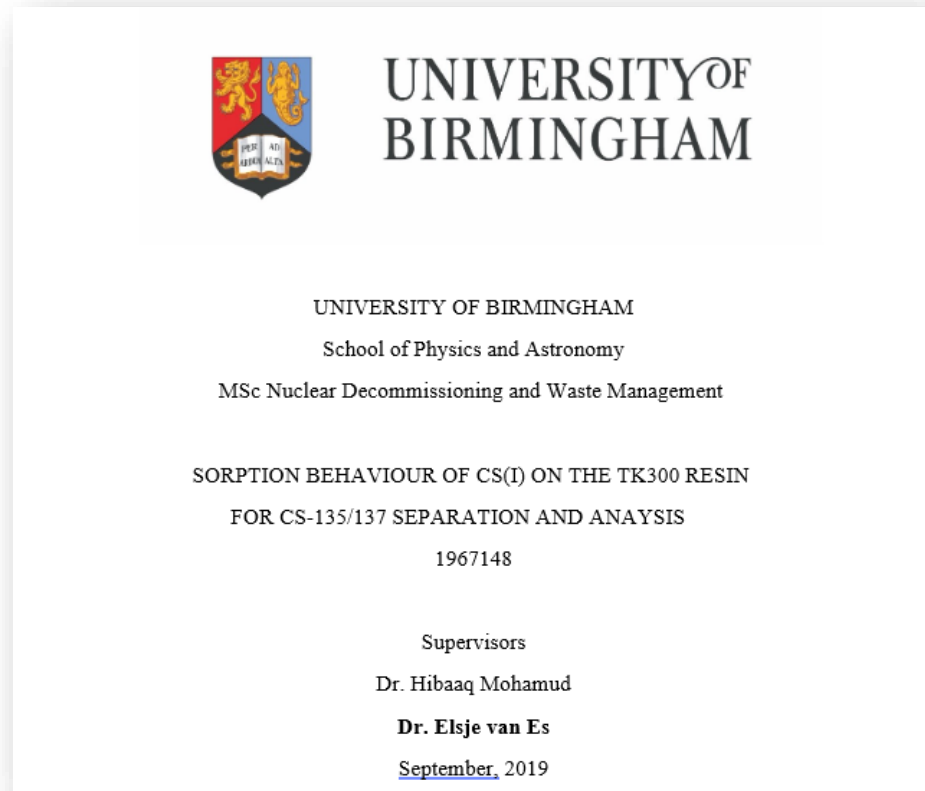
Cs Resin comparison

Property	TK300	AMP-PAN	KNiF-PAN
Active phase	Crown ether extractant (organic extraction chromatography)	Ammonium molybdophosphate (AMP) in PAN matrix	Potassium nickel hexacyanoferrate(II) in PAN matrix
Typical matrix	Environmental waters, digests, radiochemical separations	Acidic waste streams, seawater, high-salt matrices	Natural waters, seawater, urine, milk
Acid tolerance	Good	Excellent, even at relatively high acid concentrations	Moderate to good
Radiation stability	Good	Excellent	Good
Flow characteristics	Fast	Fast	Fast
Capacity	~1.45 mg Cs/g	~33 mg Cs g ⁻¹ (dry resin)	Generally high for trace Cs scavenging
Elution	Can be stripped using appropriate eluents	Difficult, requires concentrated NH ₄ Cl/NH ₄ NO ₃ or destruction of AMP	Often used as a scavenger; elution can be challenging
Best use	Analytical separations where Cs recovery is required	Cs removal/concentration from highly acidic or radioactive solutions	Large-volume environmental sample pre-concentration

- From Triskem research
 - Optimal Cs uptake at 2M HNO₃ with good uptake from 0.5M - 2M HNO₃.
 - Rinsing has been tested with different concentrations of HNO₃.
 - Water can be used as elution medium.
 - Theoretical capacity Cs is about 1.45 mg Cs/g.



Previous work on TK300 at NPL

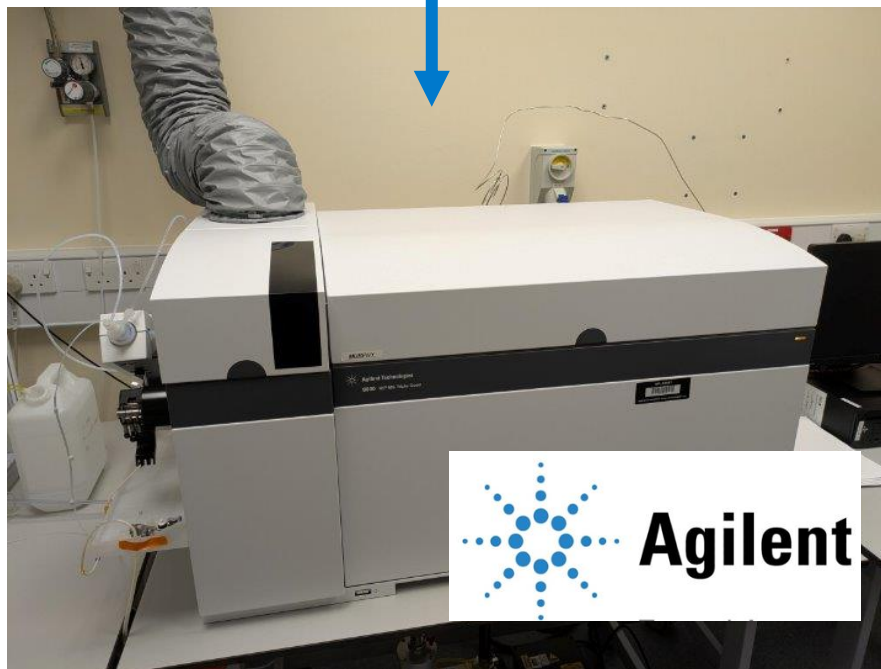


- Different TK300 resin but similar chemistry
- Used as a guide for recent experiments
- Main findings:
 - HNO_3 provides significantly better conditions for Cs retention than HCl
- Cs and Rb are strongly retained at low HNO_3 concentrations
- Most transition metals, lanthanides and actinides exhibited very low K_d values

Facilities at NPL



Vacuum box



HIDEX Q-ARE
Automated Separator

Agilent 8800/8900
ICP-MS

Experimental

Determine the K_d for various analytes of interest via ICP-MS analysis



Identify potential separation conditions e.g. loading, washing and elution



Column separation - determine an elution profile and volume of acid needed for each separation step

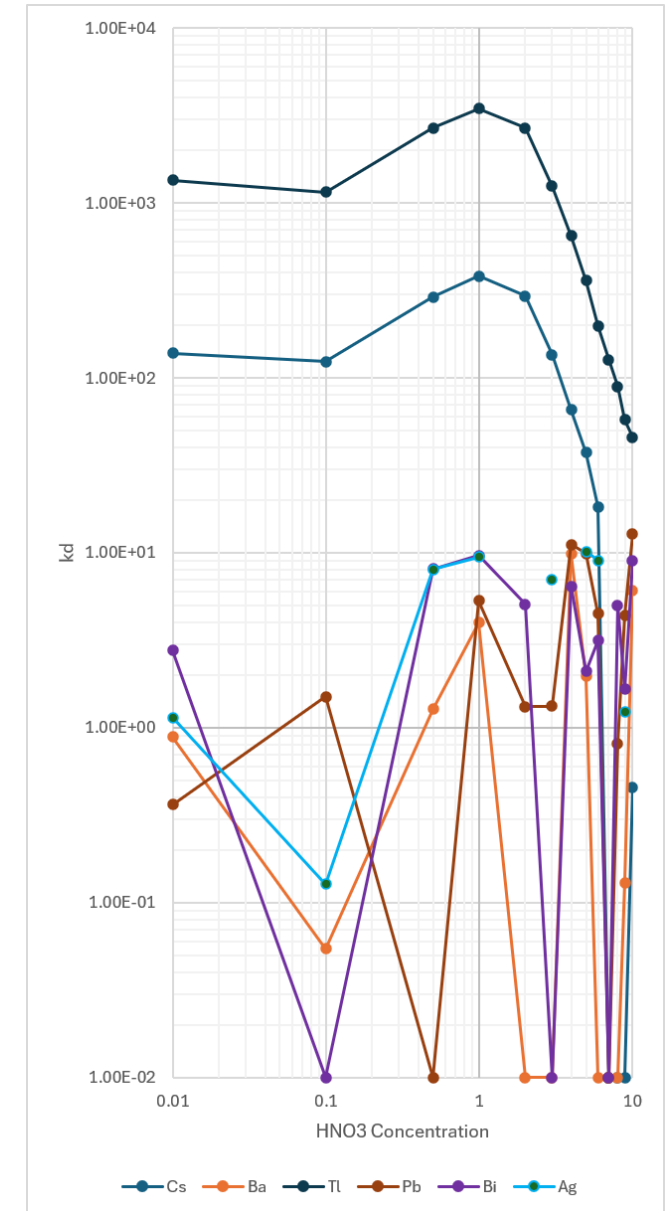
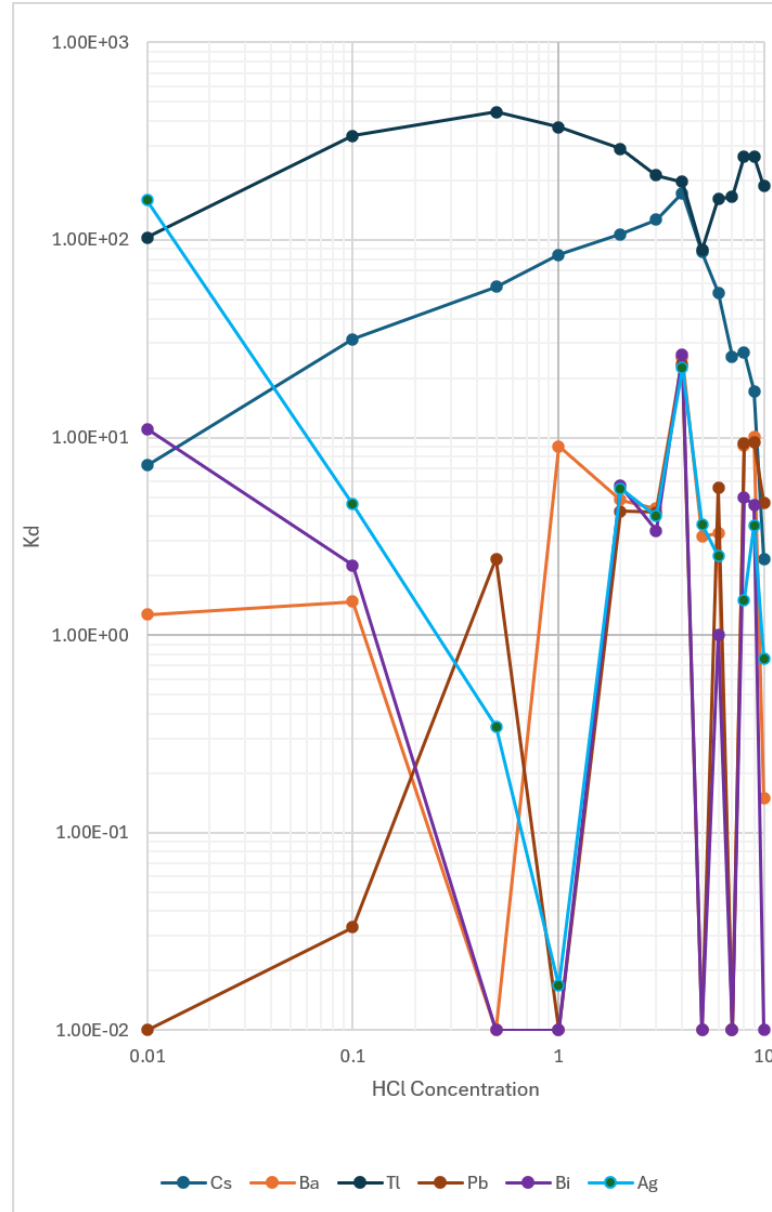


Write up an optimised column separation procedure

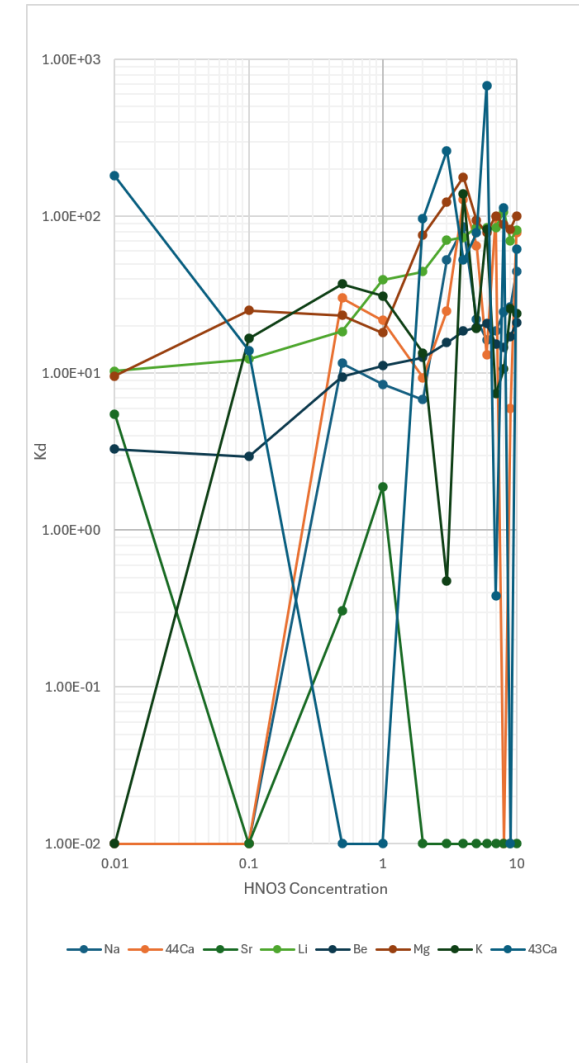
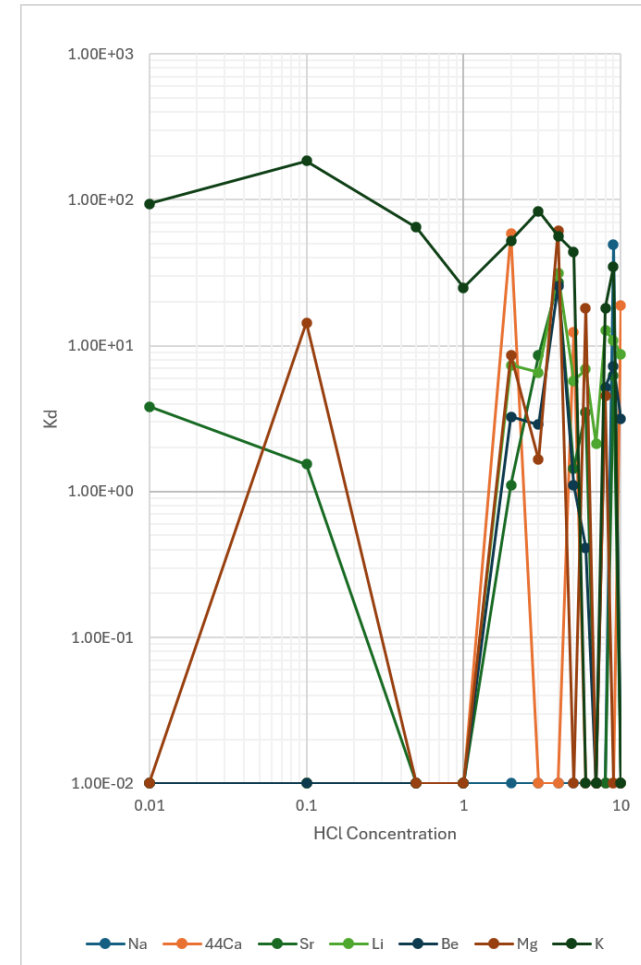
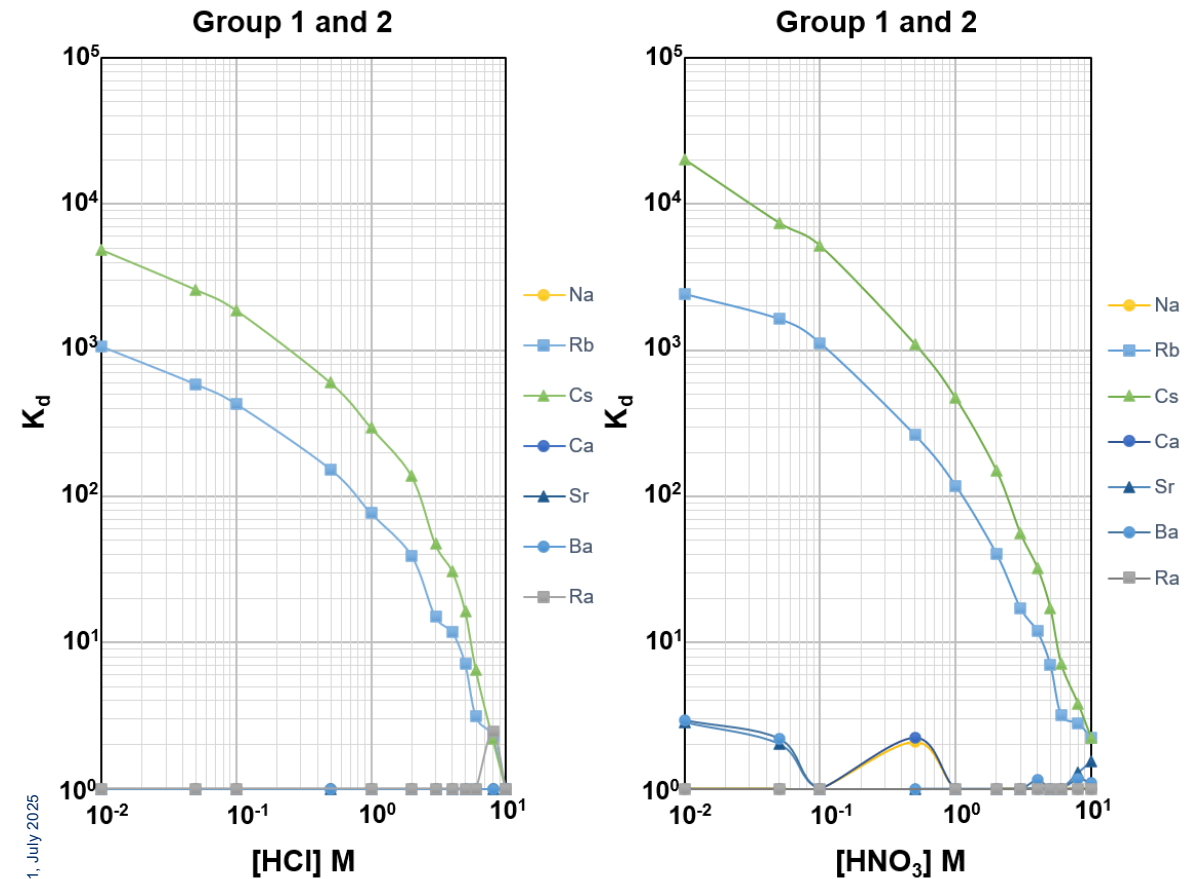
Distribution coefficient tests

- 0.1g TK300 resin dissolved in 0.01M, 0.1M, 0.5M, 1M-10M HNO₃ and HCl acid molarities
- Spiked with Cs and other elements of interest
- Took a t0
- Filtered through vacuum box

$$K_d = \left(\frac{[CPS_i - CPS_f]}{CPS_f} \right) \left(\frac{V}{m} \right)$$

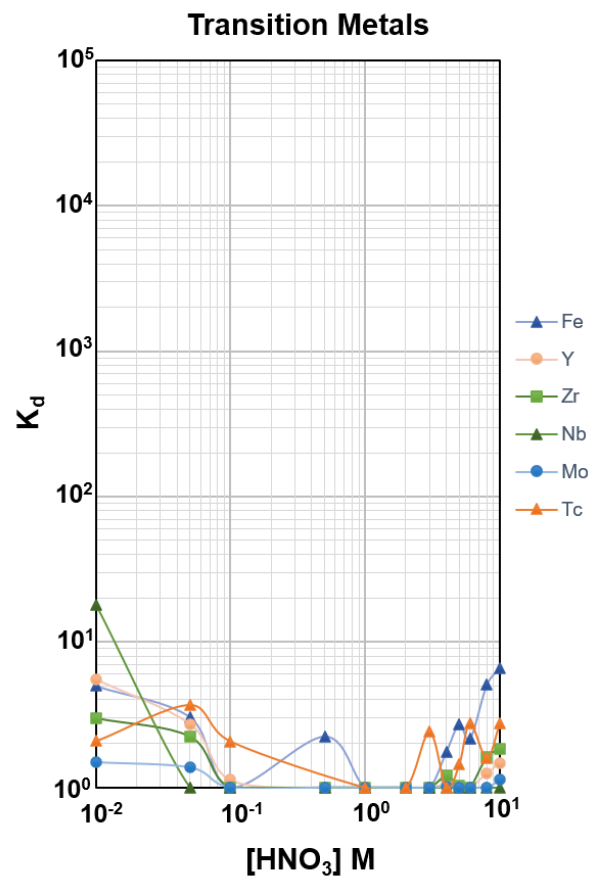
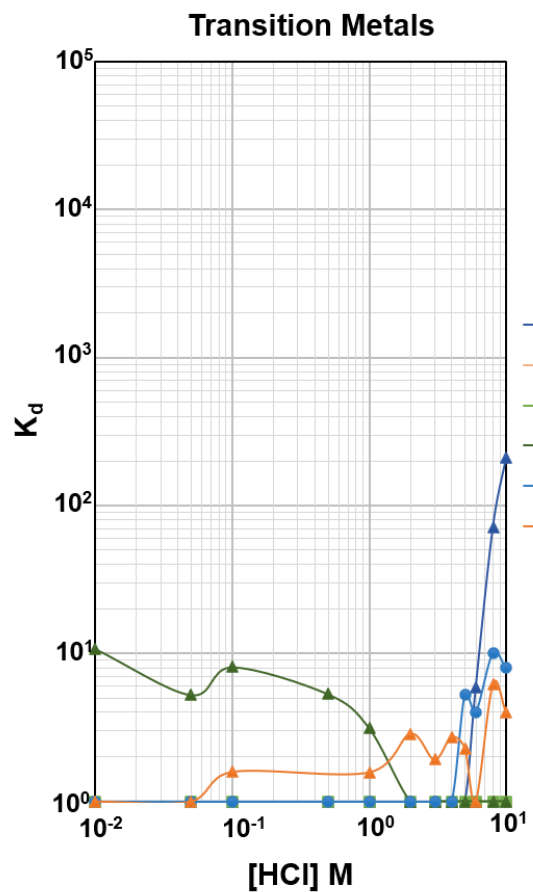


Group 1 and 2

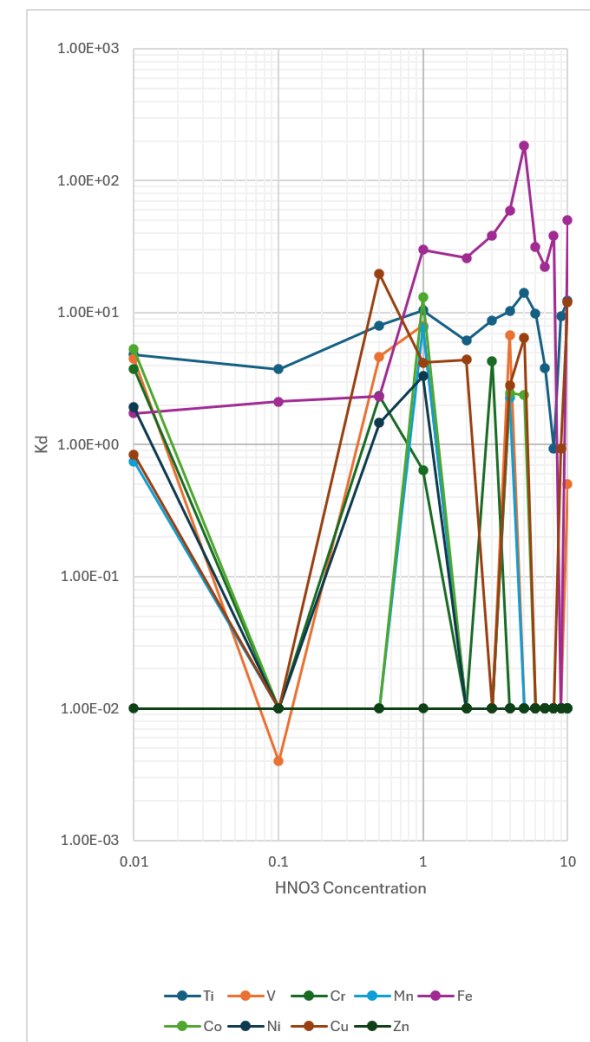
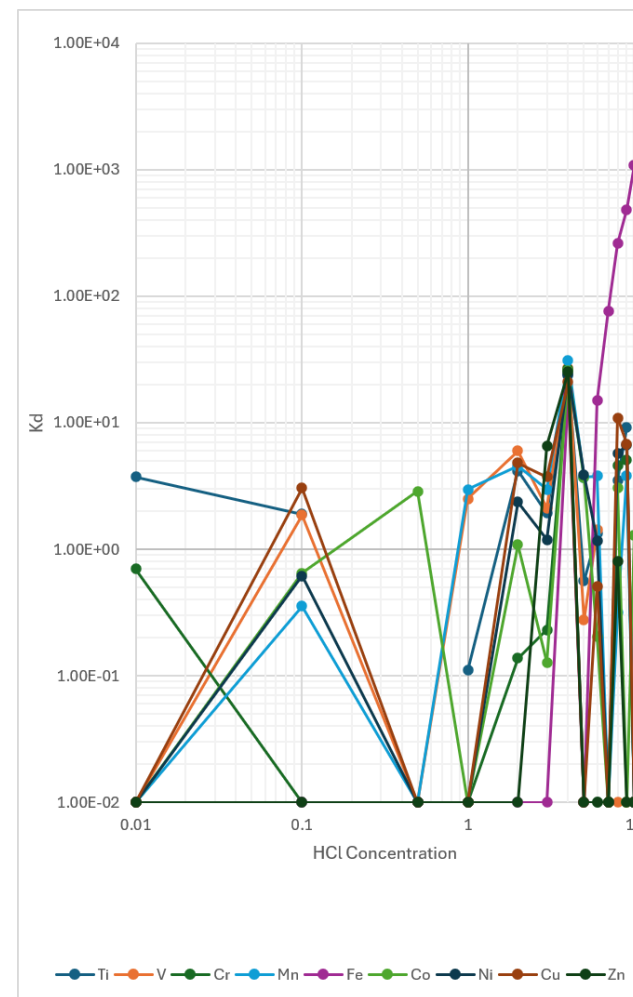


2019 work from EvE thesis

Transition metals

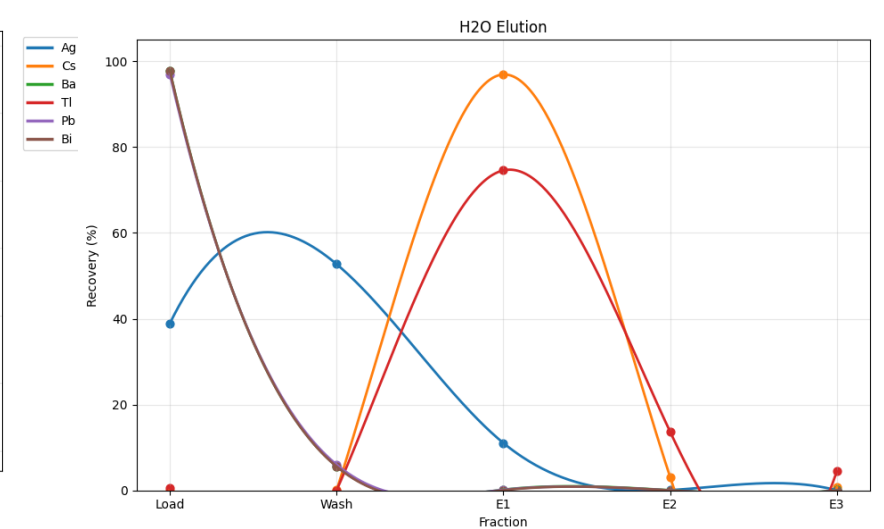
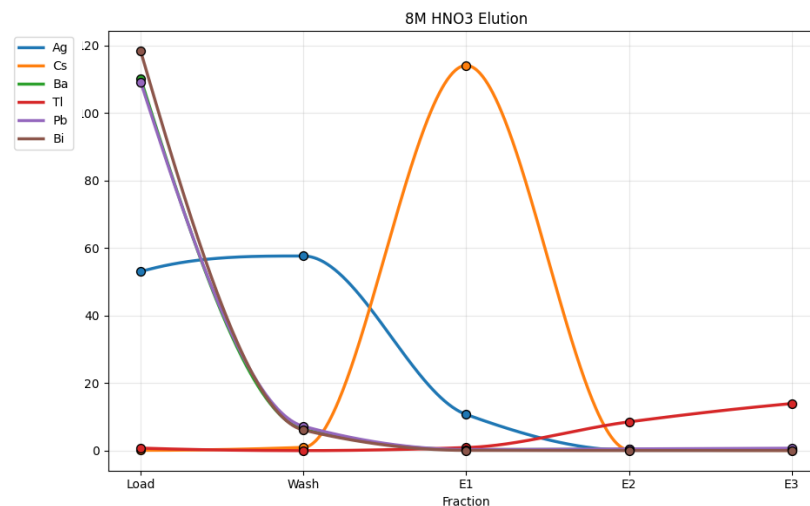
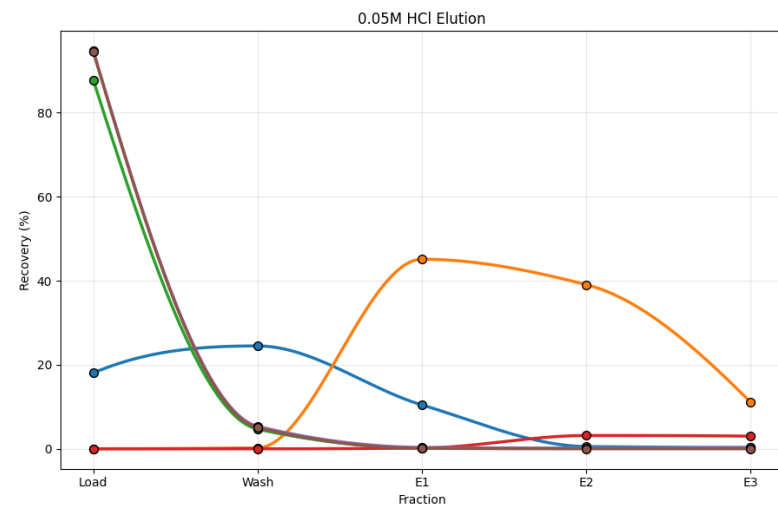
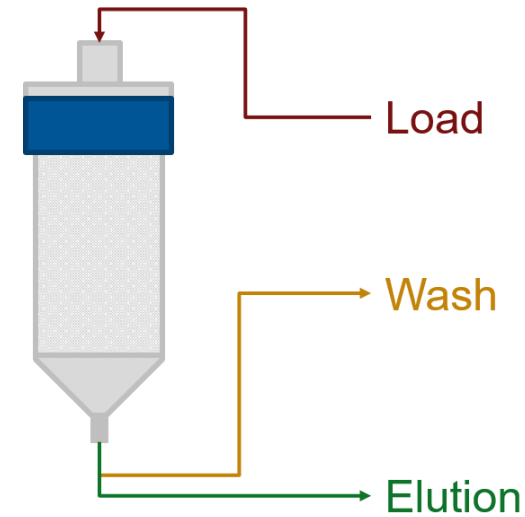


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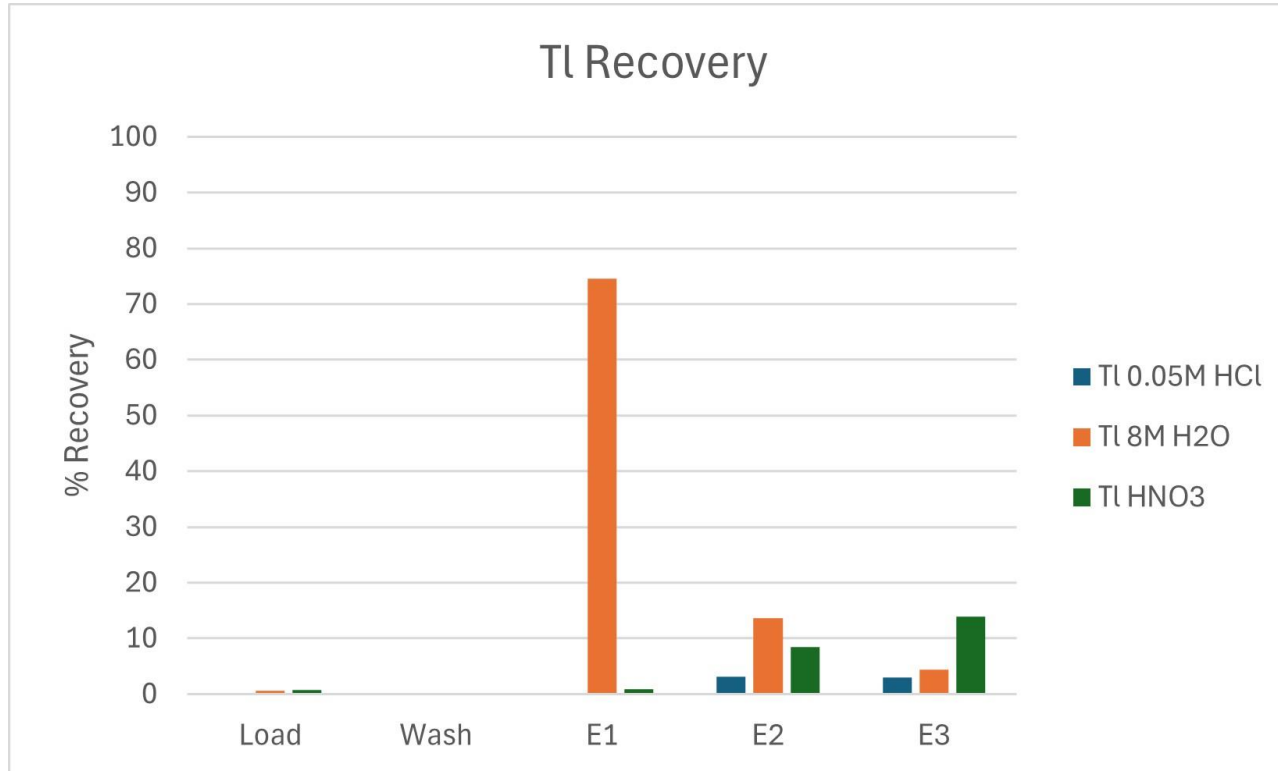


Elution profiles

- Automated separator
- Conditions
 - Load in 10ml 1M HNO₃
 - Wash with 10ml 1M HNO₃
 - Elution with 3 x 10ml of elution condition
- For elements with $K_d > 10^2$
- Tl and Ag show some retention to column
 - Tl eluted out using H₂O



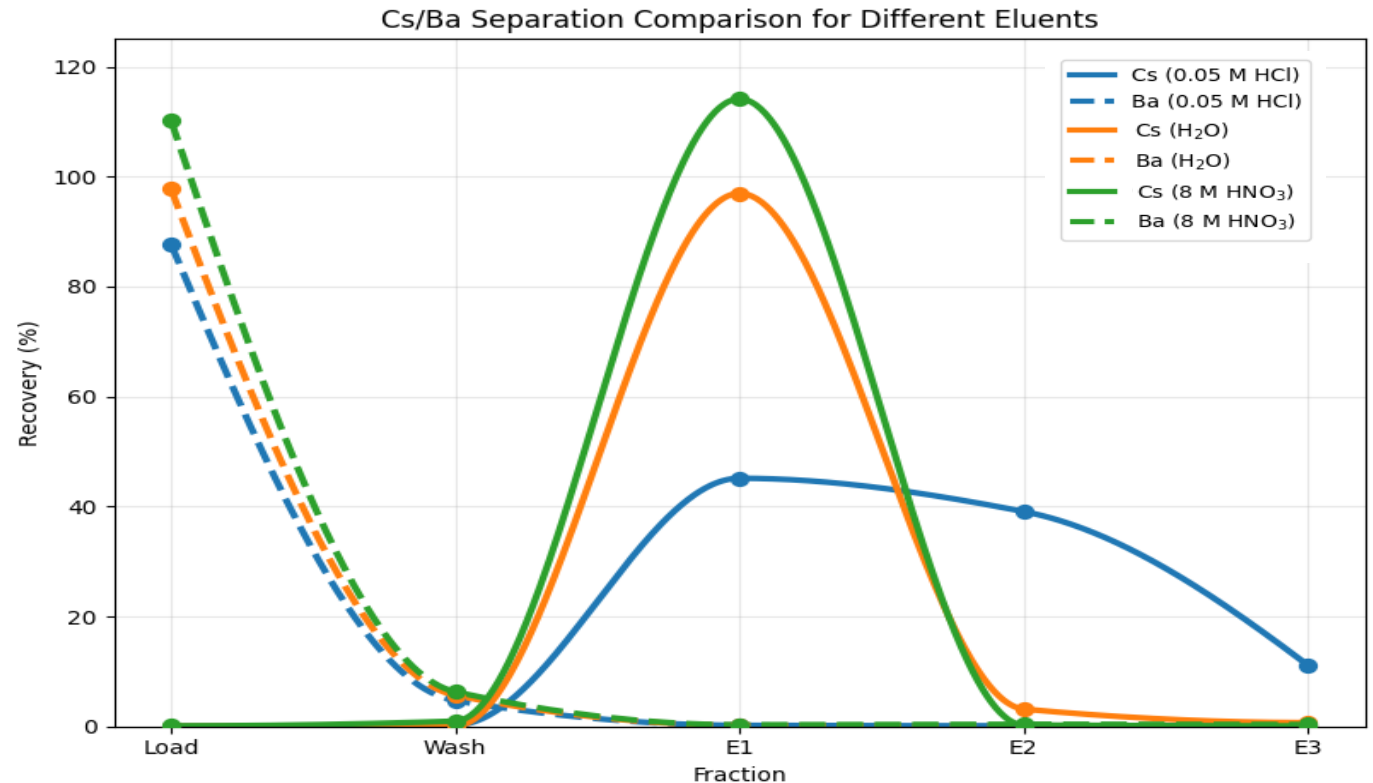
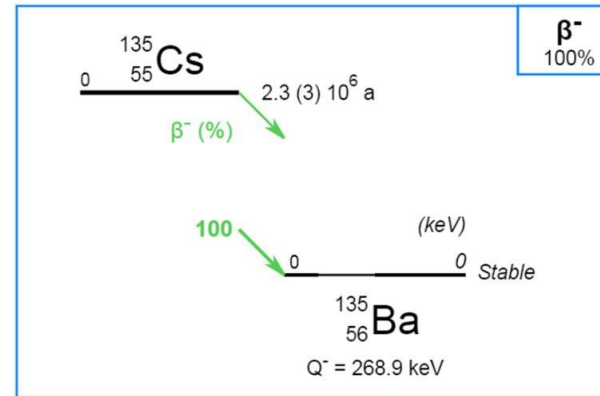
Cs/Tl recovery



- Tl only fully elutes off in H₂O
- Not an isobaric interference so not of concern
- May reduce Cs recovery from column
- Could it happen with Rb?

Cs/Ba separation

- Comparison of Cs/Ba from previous elution profile
- Ba consistently coming off in Load - clean separation
- Good support for mass spec measurements of Cs-135/137 isotopic ratios



^{99}Tc : Analysis Requirements

High yield (6.06 %) fission product

Nuclear Decommissioning

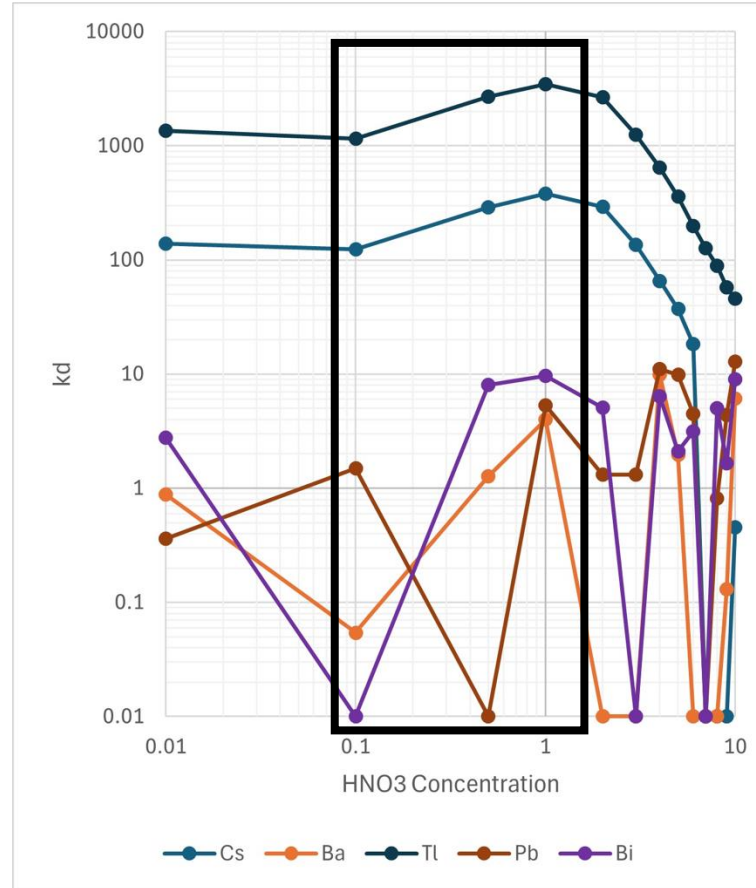
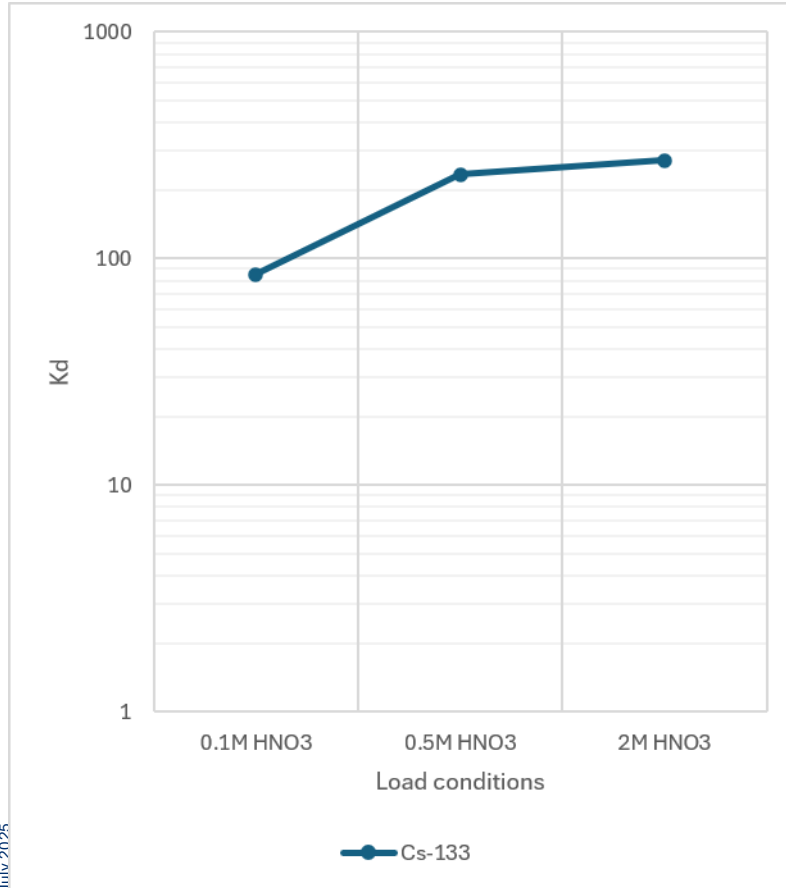
Environmental Monitoring

Radiopharmacy and nuclear medicine ($^{99\text{m}}\text{Tc}$)



Mediso AnyScan SCP (SPECT-CT-PET) system in the NPL nuclear medicine imaging laboratory

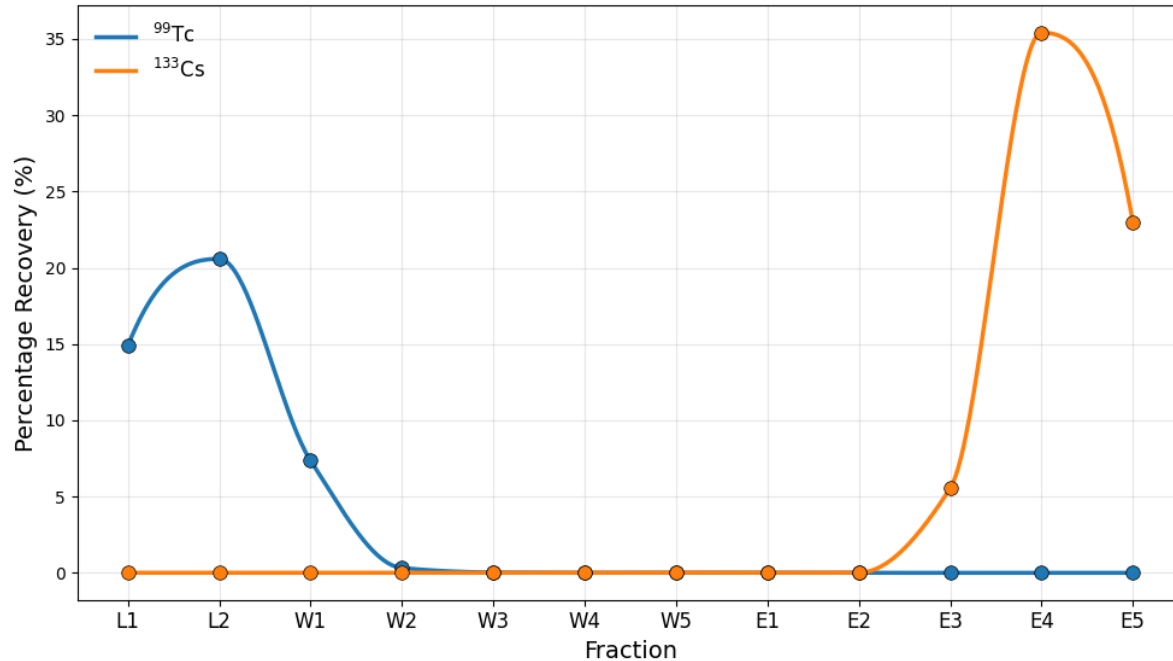
Distribution coefficient tests for Tc-99



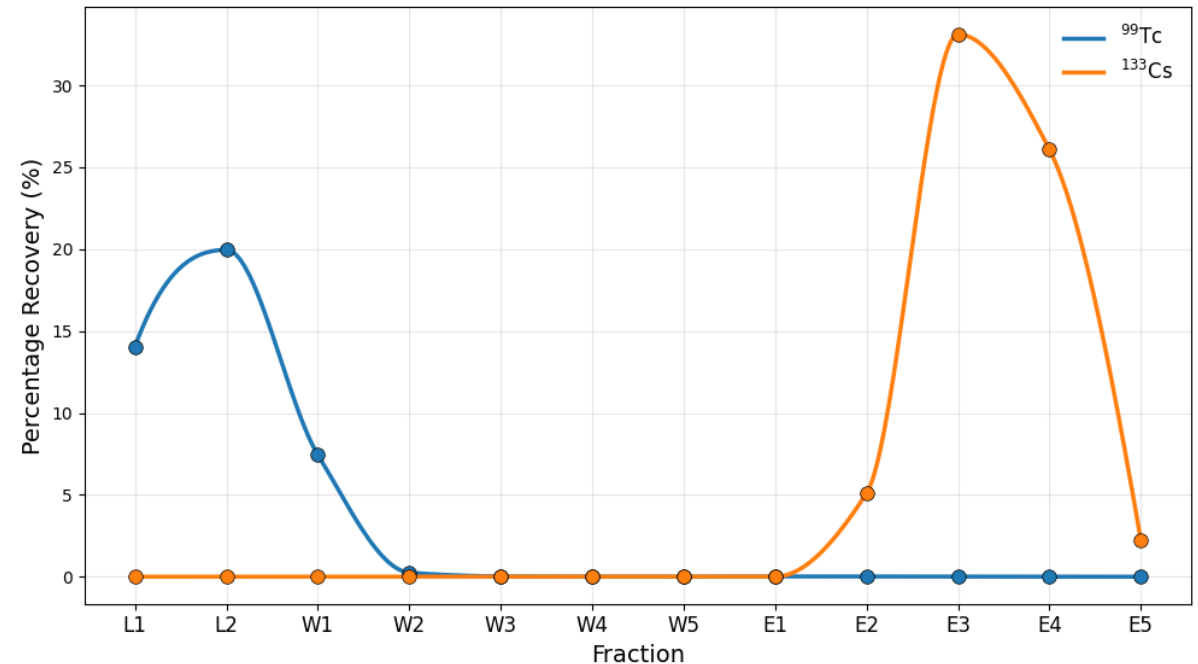
- 0.1g TK300 resin in 10ml 0.1M, 0.5M and 2M HNO₃
- Left for 24 hours and vacuum filtered before mass spec analysis
- Similar K_d values
- K_d all <0 for Tc-99

Column conditioning tests

Column Conditioned with 2 M HNO₃



Column Conditioned with Ethanol



- Vacuum box separations
- 2 x 5ml 2M loads, 5 x 2ml 2M HNO₃ washes, 5 x 2ml DI H₂O elutions
- Better Cs-133 recovery conditioning TK300 with ethanol followed by 2M HNO₃
- Clear separation between elements

Conclusions

- TK300 promising candidate resin for Cs isotope separations
- Determined conditions for effective separation with highest percentage recovery
- Able to successfully separate Ba and other interferences from Cs with high percentage recoveries



Endless tests left to try!



Cs/Tl/Rb separations



Active Cs isotopic ratios (Cs- 134/135/137) – need N₂O gas



Different Cs concentrations in 2M HNO₃ load - micro or macro can impact the chemical yield



Use of automated separator/ICP-MS coupling for real-time online measurements



Adding Ba, Mo and Ru to Cs sample for NPL mass spectrometry interests

Thank you

Any questions?