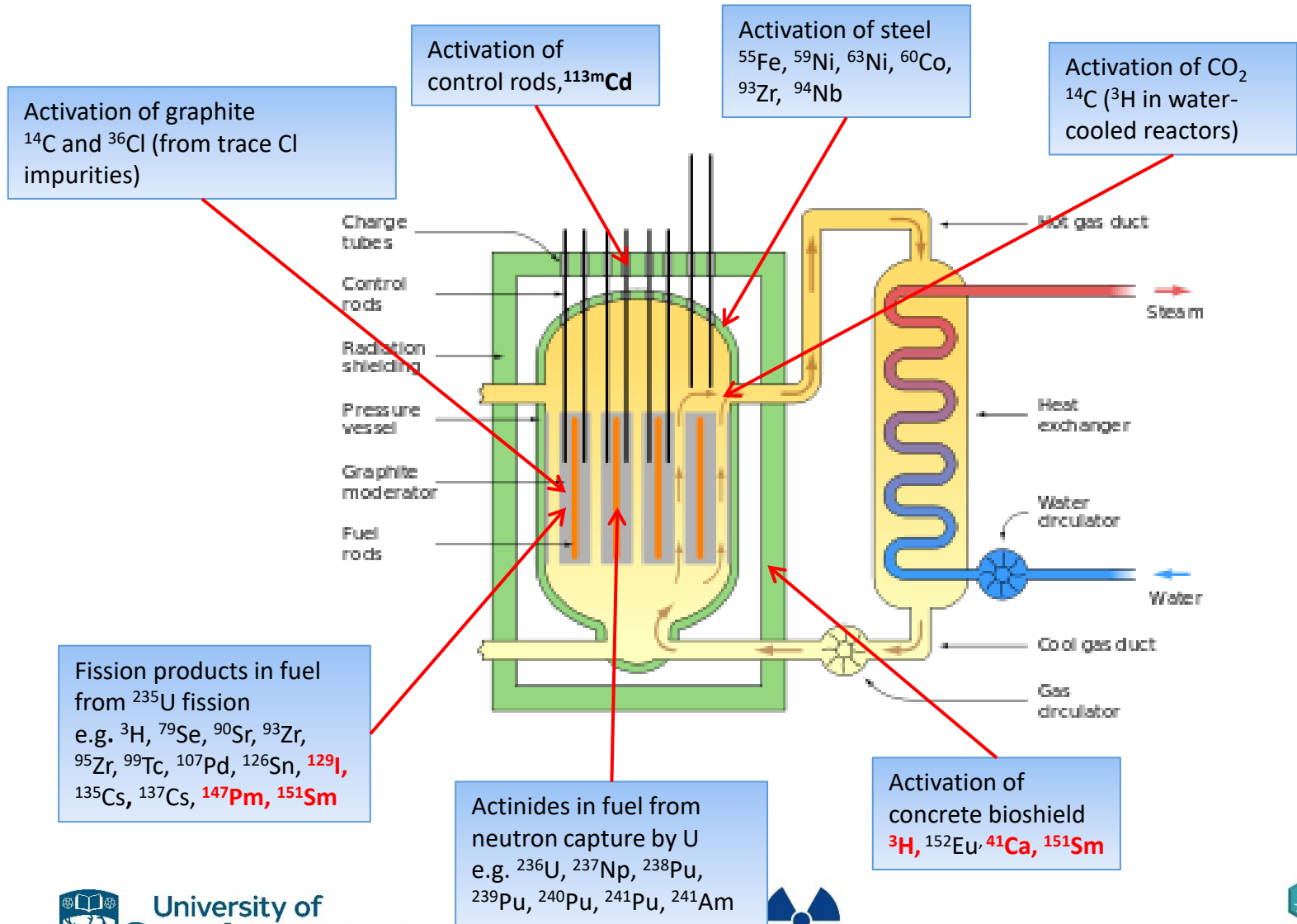


# Extraction chromatographic separations for decommissioning waste analysis

P Warwick, F Rowlands, M James, D Reading,  
P Gaca, B Russell (NPL)



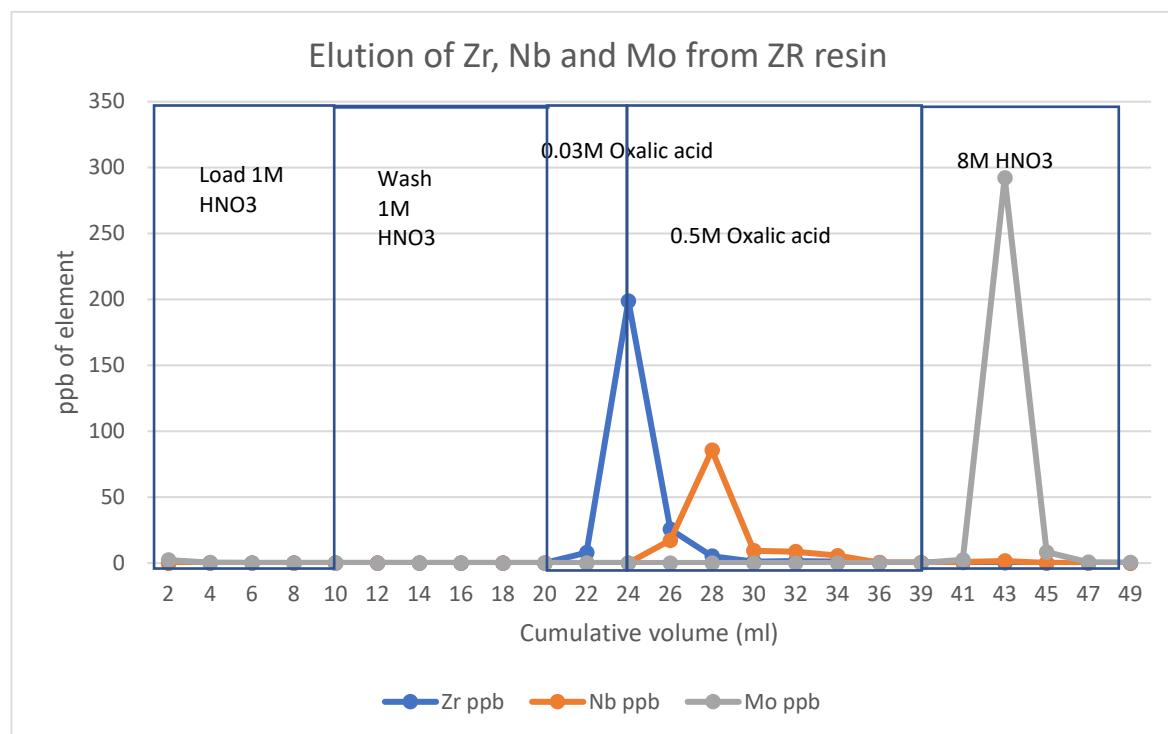
# DTM radionuclides



# Separation

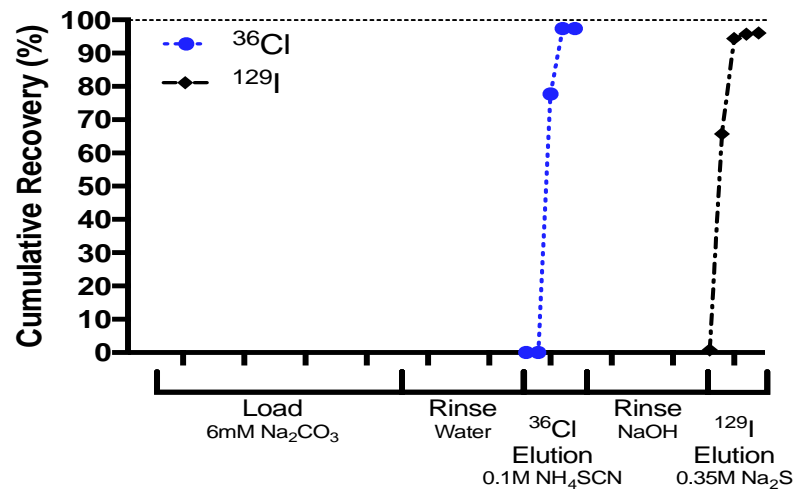
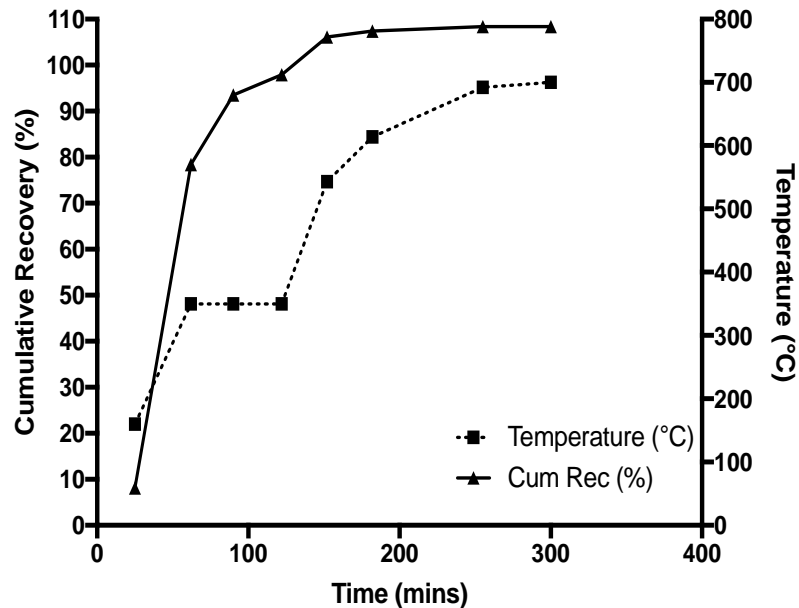
- Rapid separation of radionuclides
- Novel separation schemes for radionuclides not routinely analysed
- Modified separations for mass spectrometry (to account for the different range of interferences)
- Automation

# Zr-93, Mo-93



4 x 0.5 cm  
ZR resin

# I-129



# Production of $^{151}\text{Sm}$

- Fission product (m - 151 isobar = 0.42% yield)
- $^{150}\text{Sm} (n, \gamma) ^{151}\text{Sm}$
- ( $^{150}\text{Sm} = 7.38\%$ ;  $\sigma_{\text{therm}} = 100 \pm 4\text{b}$ )

IAEA database

# Radiometric measurement

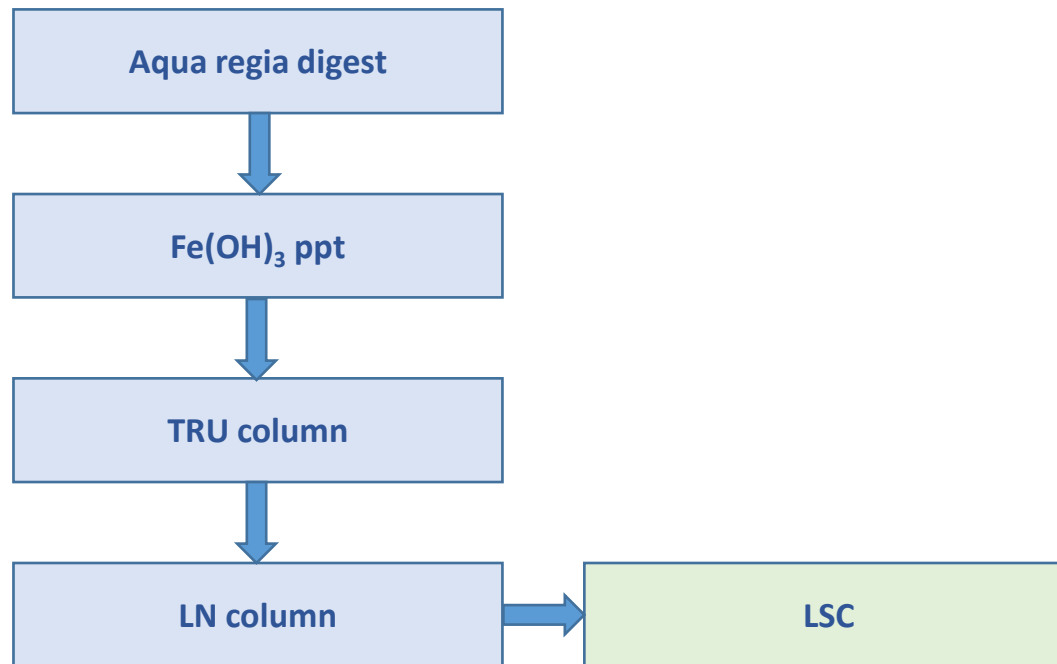
- Half life = 94.7 a
- Pure beta emission
- $E_{\max} = 76.4 \text{ keV (99.07 \%)}; 54.9 \text{ keV (0.93\%)}$
- Liquid scintillation measurement
- Efficiency correction using one of 4 approaches
  - (i) Standardised  $^{151}\text{Sm}$
  - (ii) Proxy radionuclide ( $^{63}\text{Ni}$ )
  - (iii) Ciemat-NIST approach
  - (iv) TDCR
- Interferences notably from  $^{147}\text{Pm}$ ,  $^{152}\text{Eu}$ ,  $^{154}\text{Eu}$  and  $^{241}\text{Am}$ .

# Mass spectrometry

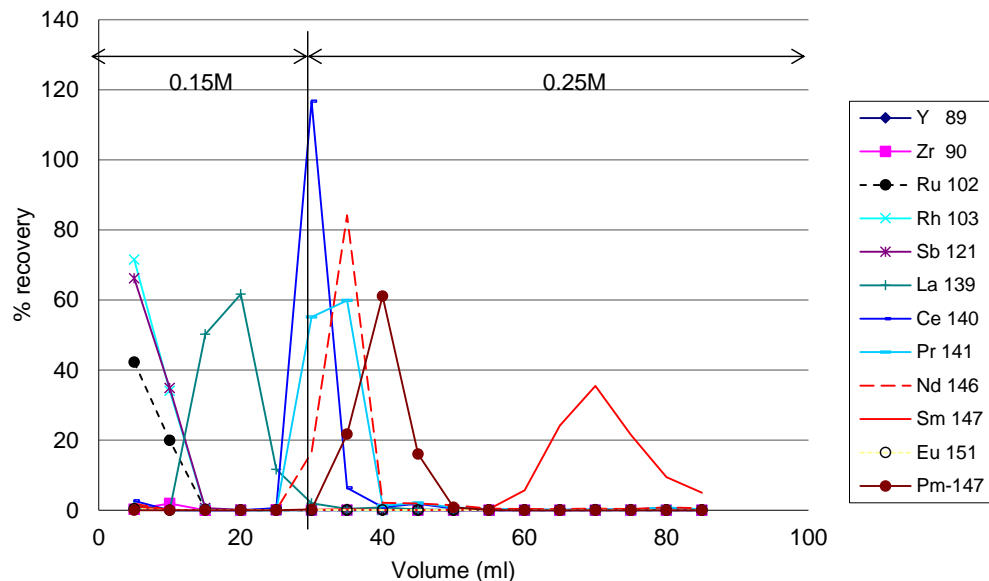
- Specific activity =  $9.25 \times 10^{11}$  Bq/g
- Interferences from  $^{151}\text{Eu}$  (47.81%)



# Original separation scheme



# LN-resin original elution profiles

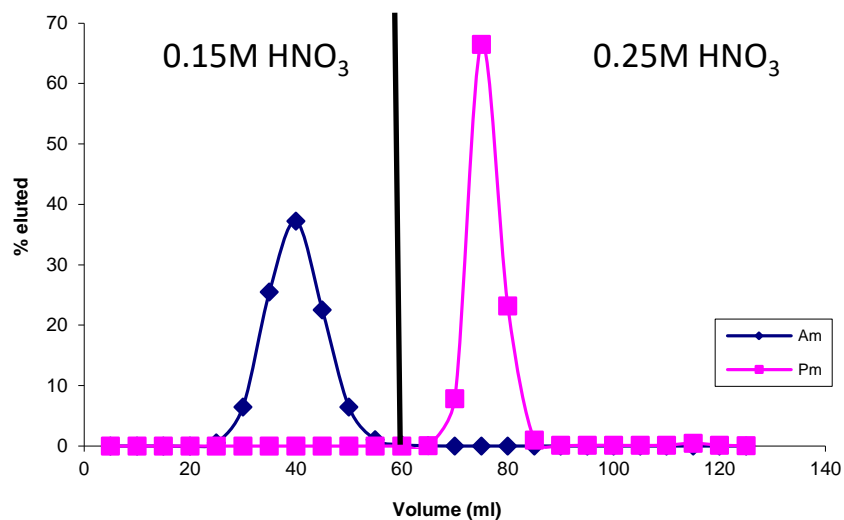


Separation optimised for Pm / Sm separation

Eu elution data inconclusive

5 x 0.5cm LN-Resin column

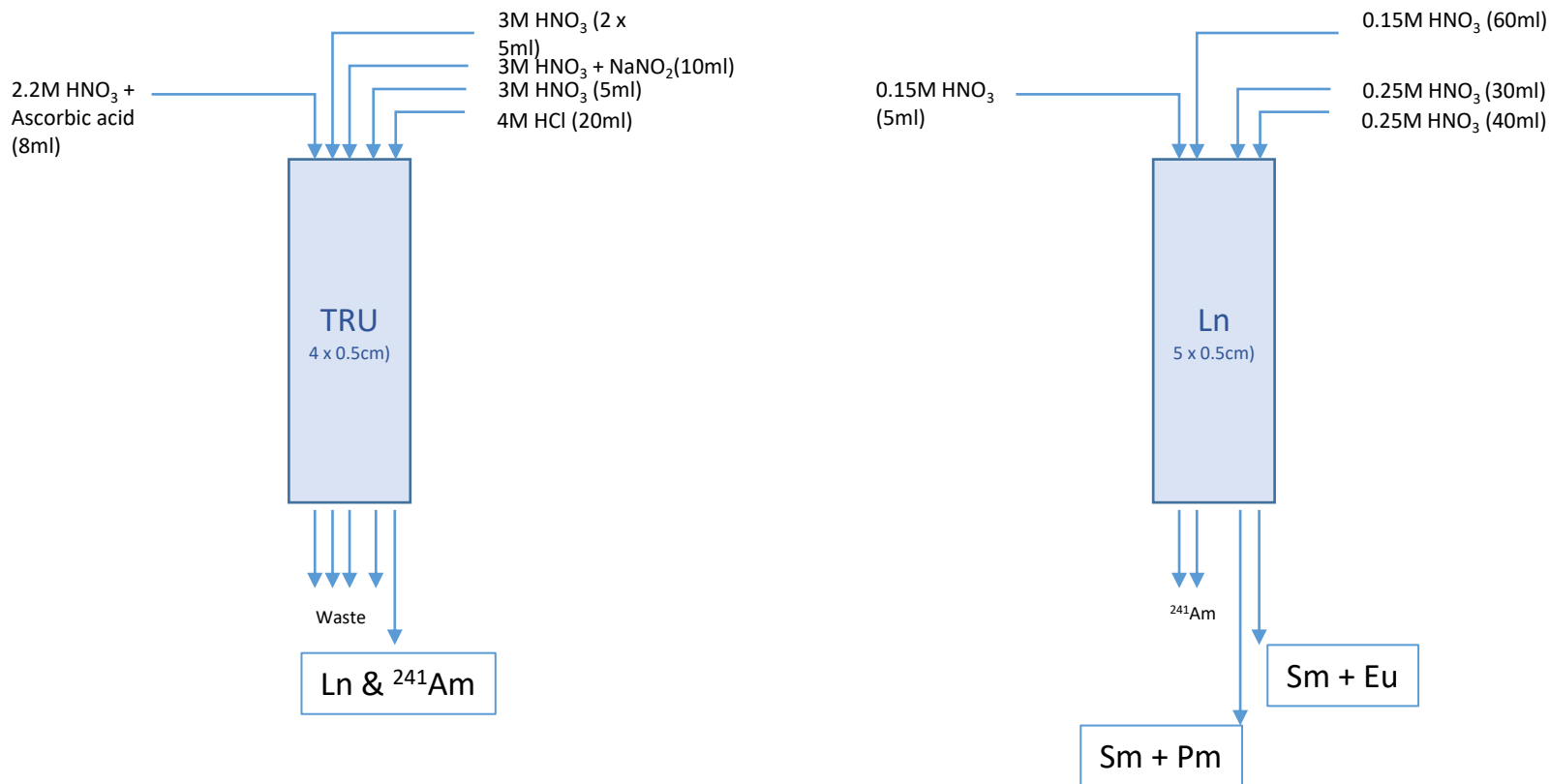
# Am / Pm separation



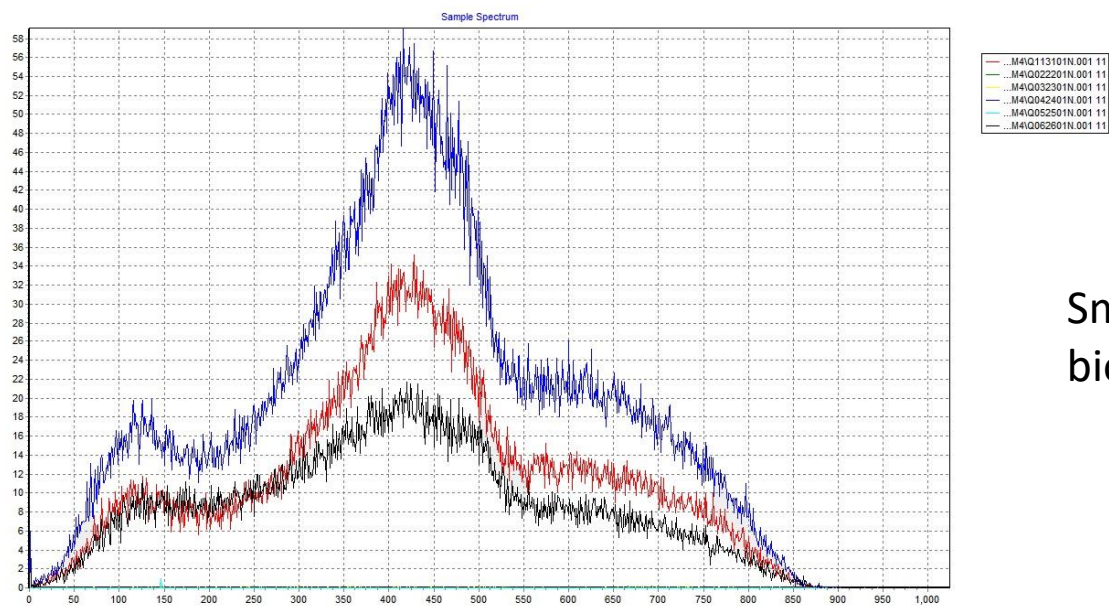
Good separation of Am from Pm (and Sm)

5 x 0.5cm LN-Resin column

# Original separation scheme



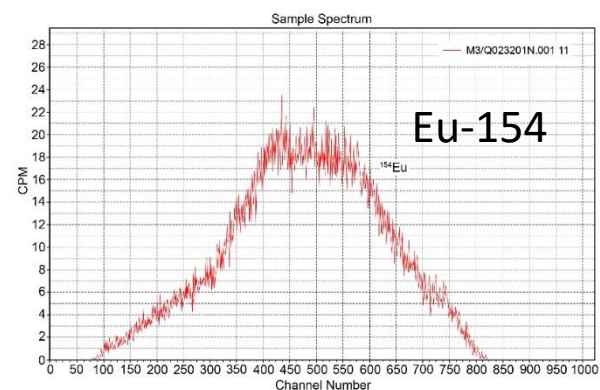
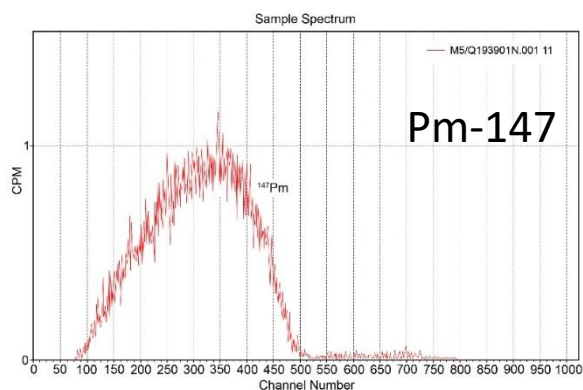
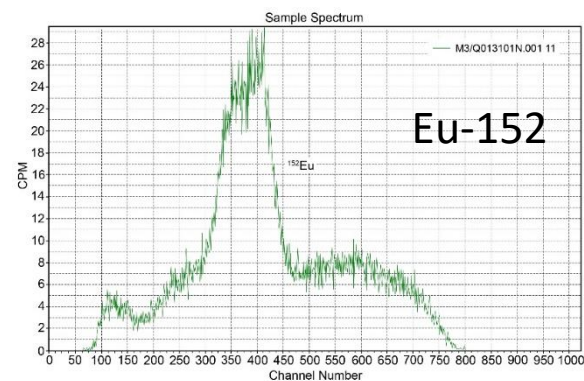
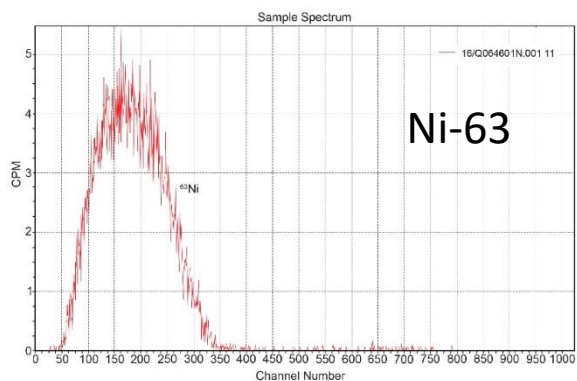
# Sm-151



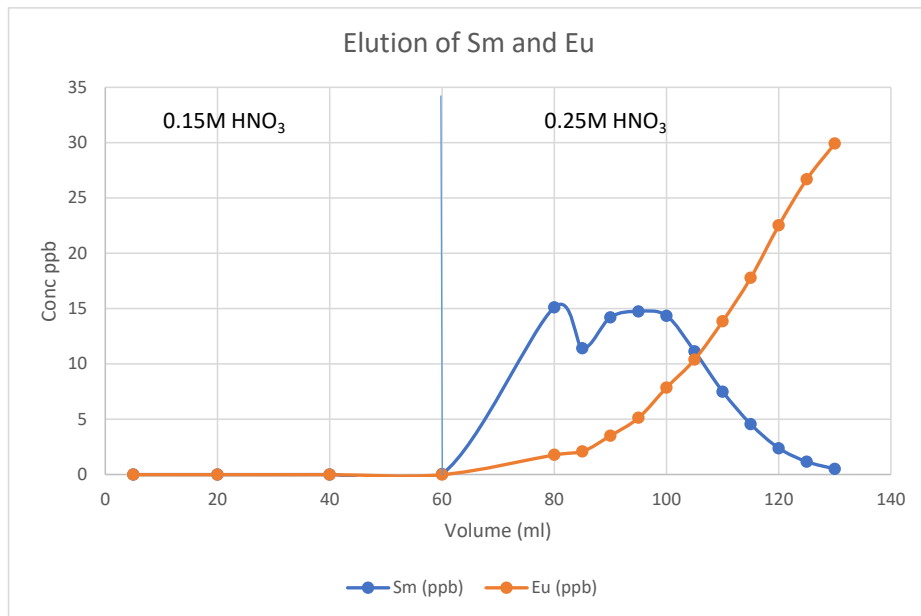
Sm fraction purified from a bioshield concrete core

F Rowlands

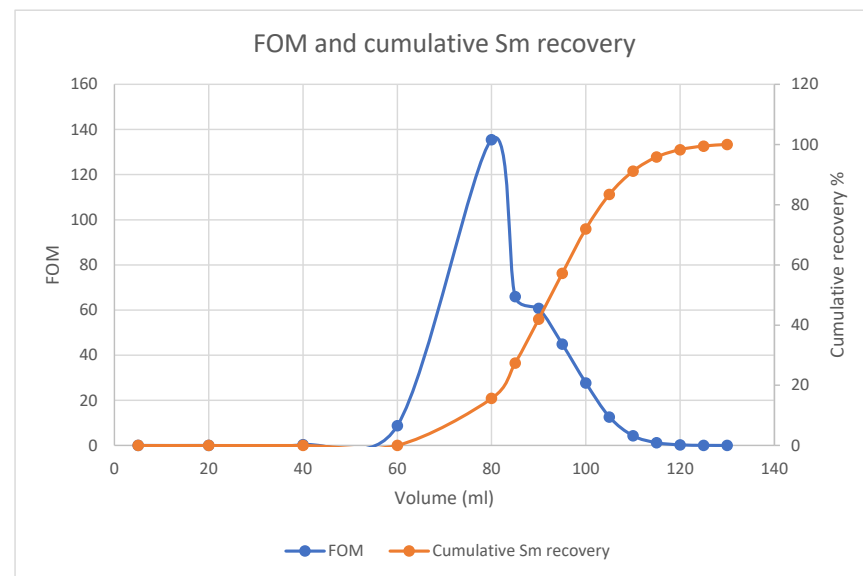
# Reference spectra



# Optimisation of Sm elution to minimise Eu

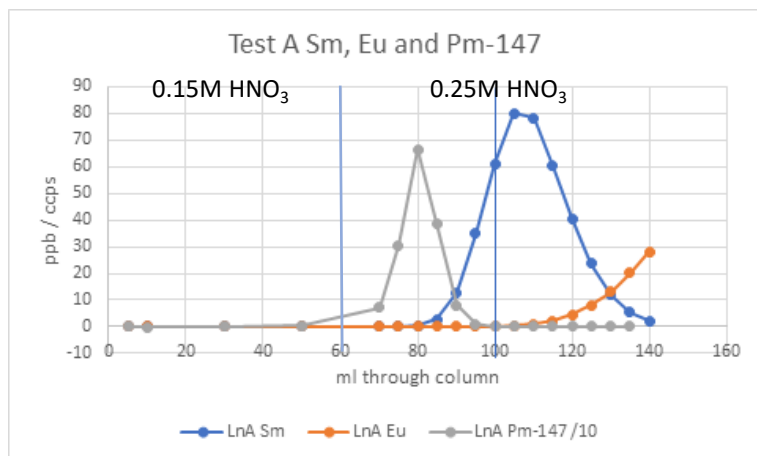


By limiting the volume of 0.25M HNO<sub>3</sub> used to elute the Sm, a better Sm/Eu separation is achieved although Sm recovery is lowered.

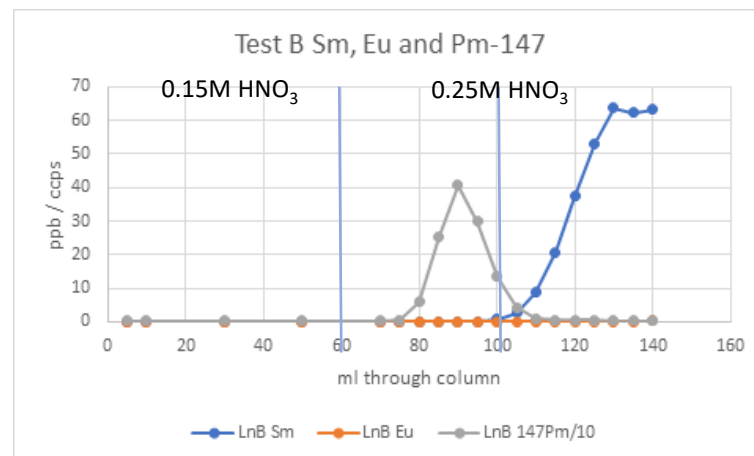


# Separation

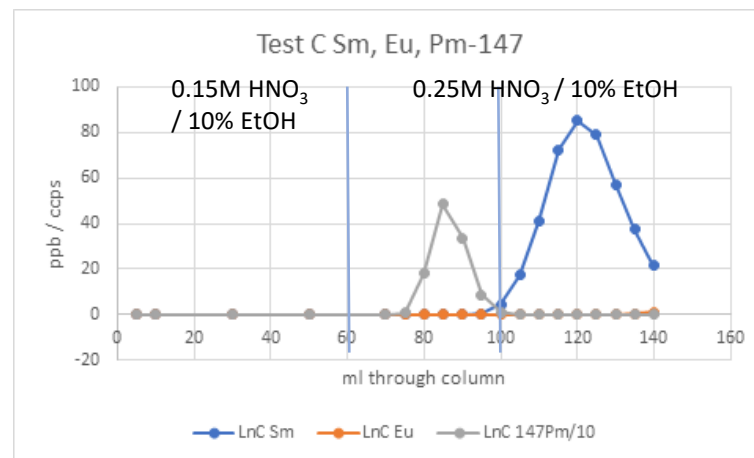
5 x 0.7cm Ln column



9 x 0.5cm Ln column

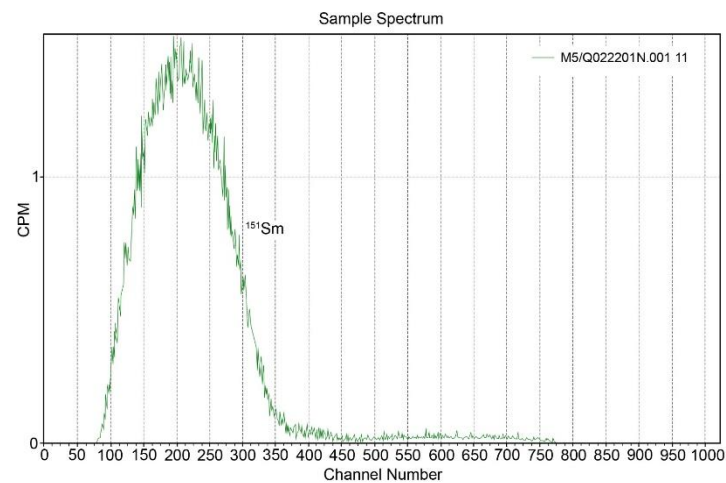
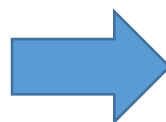
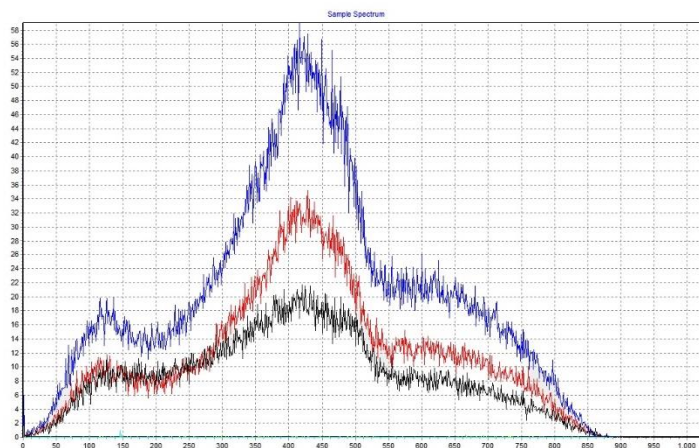


9 x 0.5cm Ln column

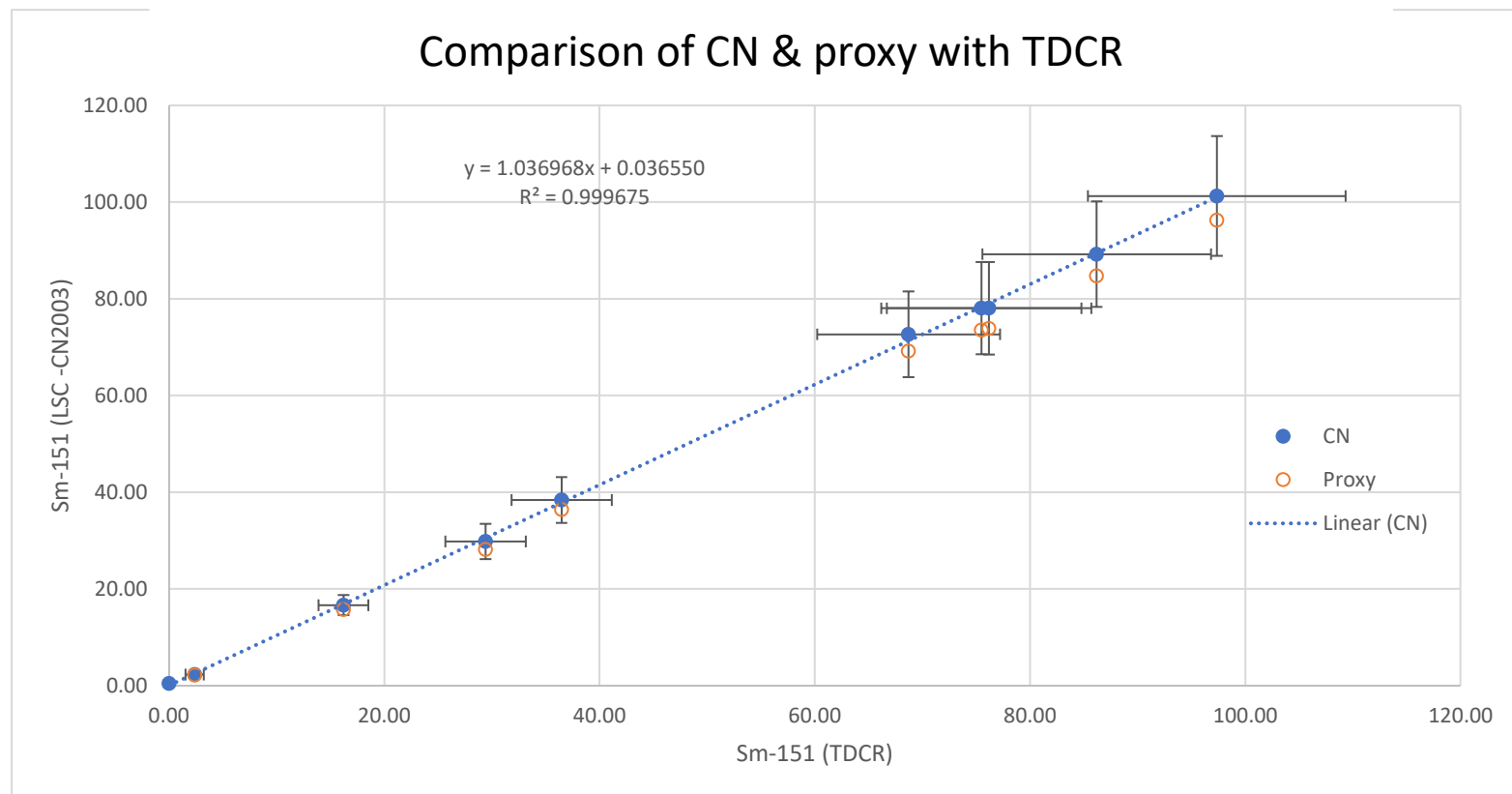




# Sm-151



# Sm-151



# Summary

- Development of novel separation procedures in support of waste characterisation.
- Adaptation of separation techniques for mass spectrometric applications
- Incorporation of resins into rapid screening procedures.
- Development of automated analysis.

# Any questions?

