

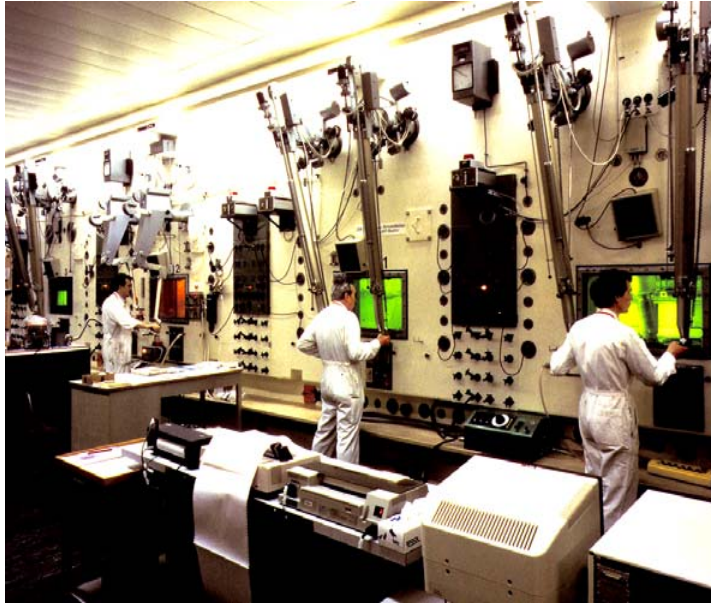
The analytical infrastructure in the Hotlaboratory of PSI

Ines Günther-Leopold

Didier Gavillet

Laboratory for Materials Behaviour





Analytical groups within the Laboratory for Materials Behaviour

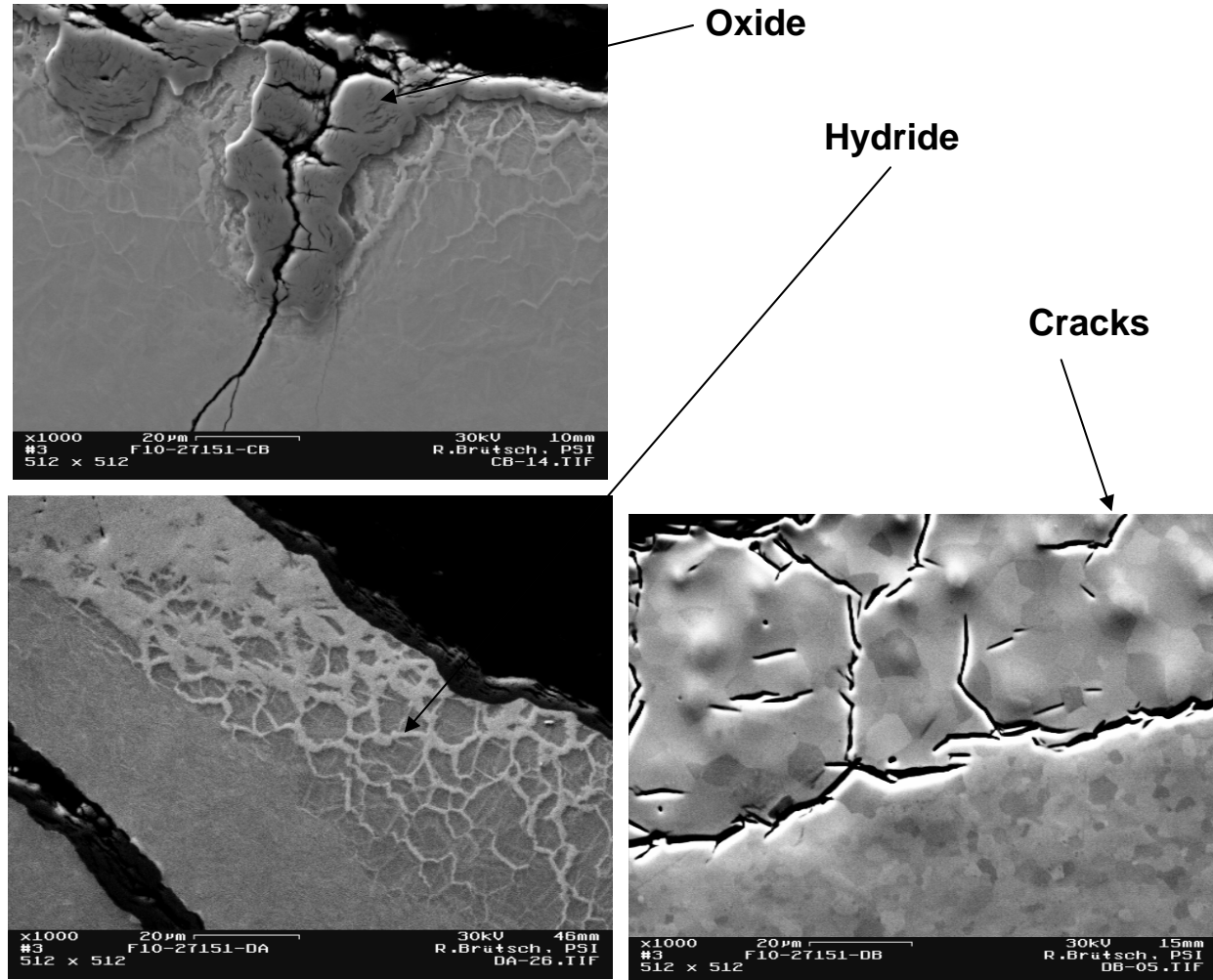
- Surface and Solid State Analysis
- Isotope and Wet Chemical Analysis

Surface and Solid State Analysis (Didier Gavillet)

- SEM (Scanning Electron Microscopy)
- EPMA (Electron Probe Microanalysis)
- SIMS (Secondary Ion Mass Spectrometry)

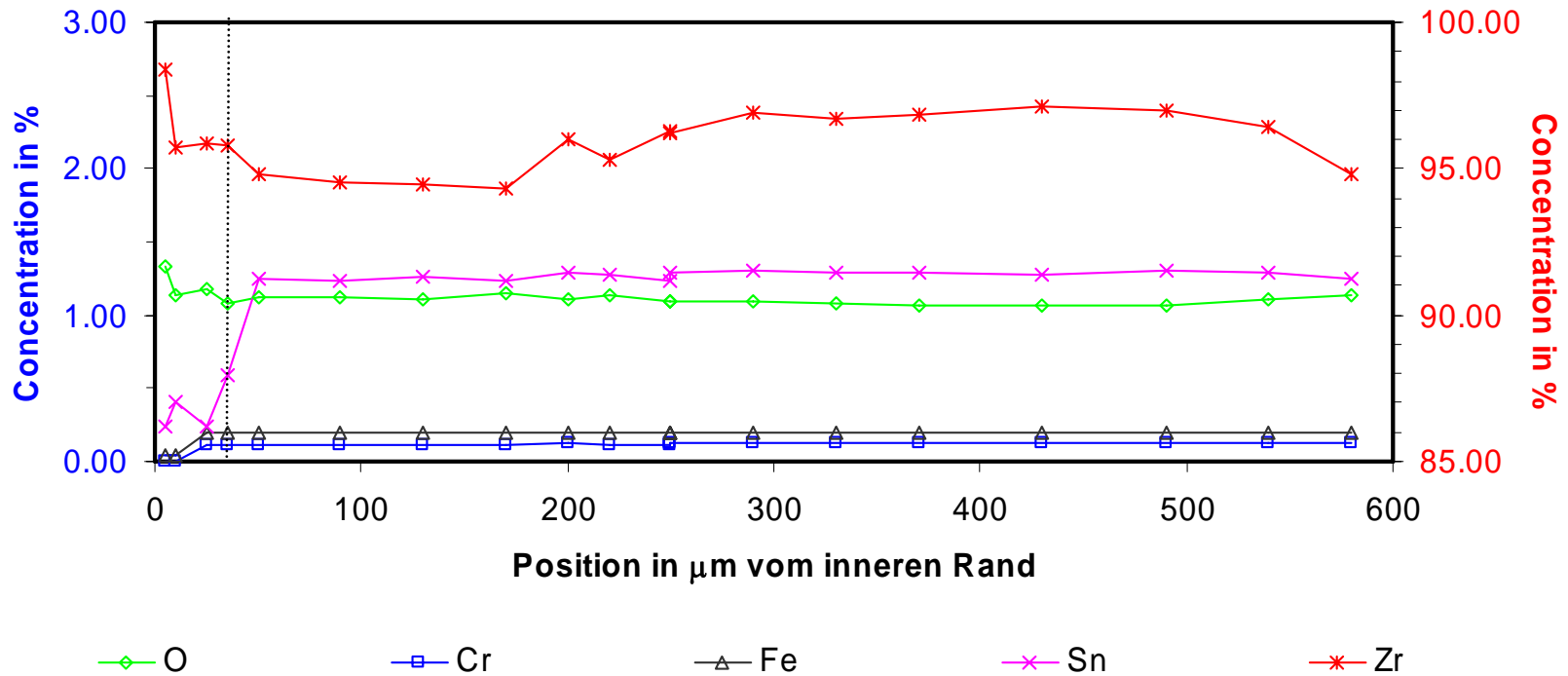
SEM analysis of polished surfaces

- Irradiated Zircaloy with large hydrogen content
- BSE observations
- Possible if the dose rate of the specimen is lower than 1 mSv/h at 1 cm.
- EDX analysis also possible for specimen dose rate lower than 0.5 mSv/h at 1 cm.

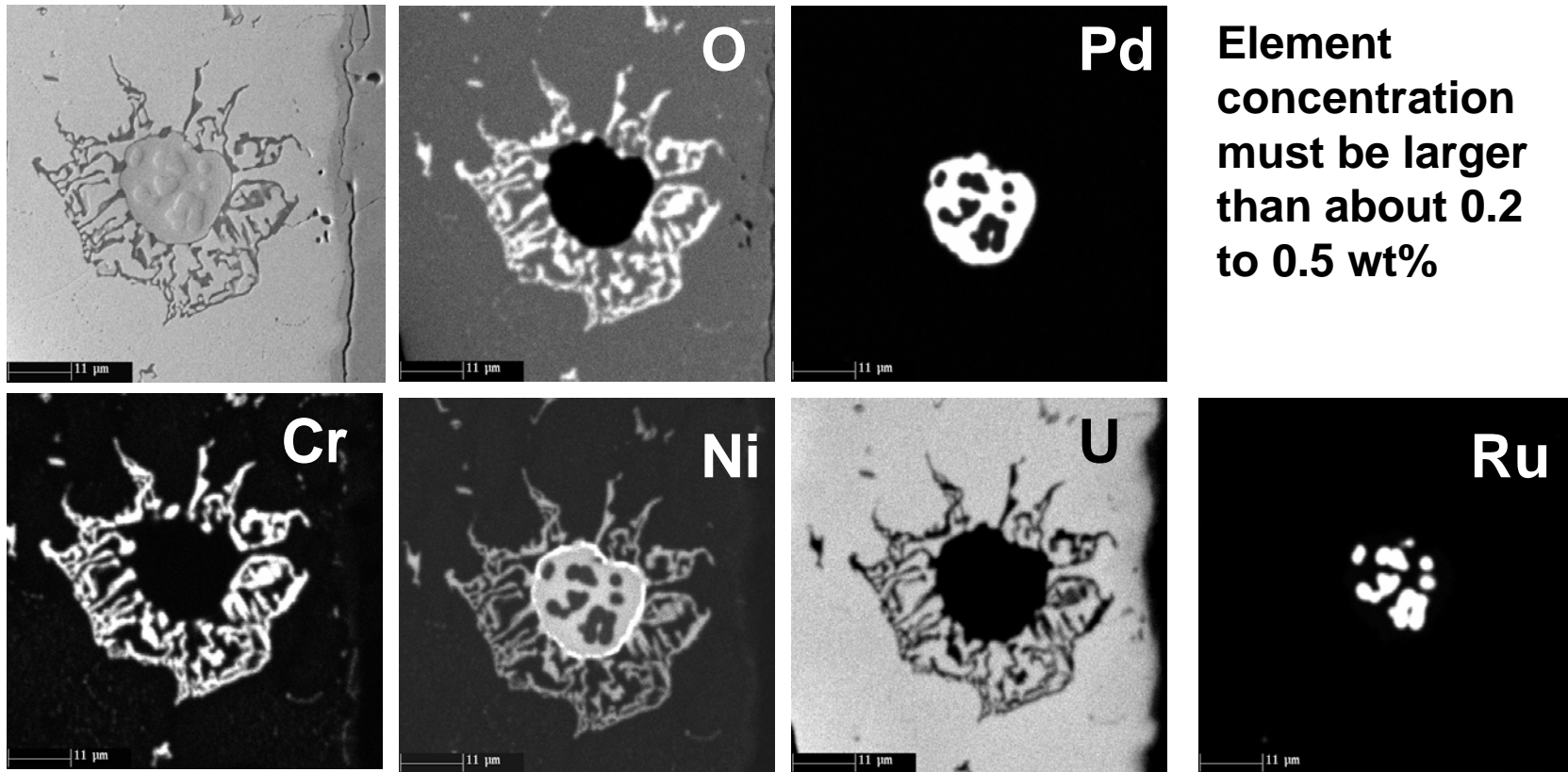


Quantitative EPMA-Analysis

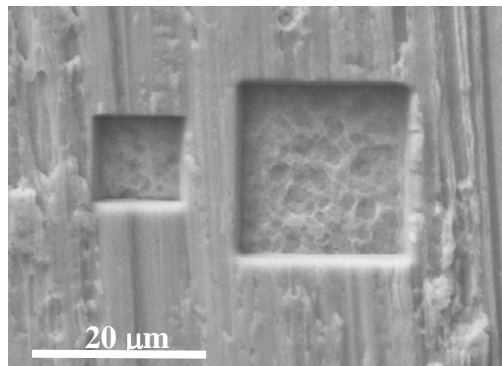
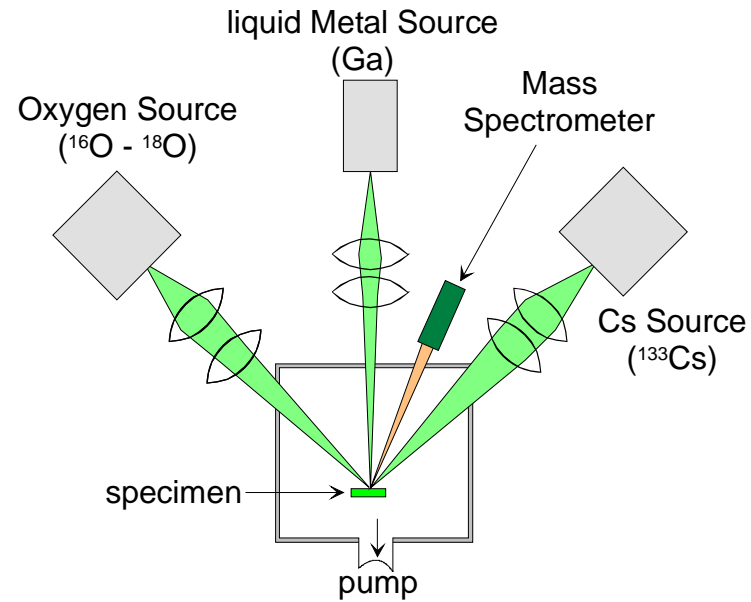
Line scan through an heavily hydrated Zircaloy tube



EPMA – Complex element distribution in melted $\text{UO}_2 / \text{ZrO}_2 / \text{Steel}$ material

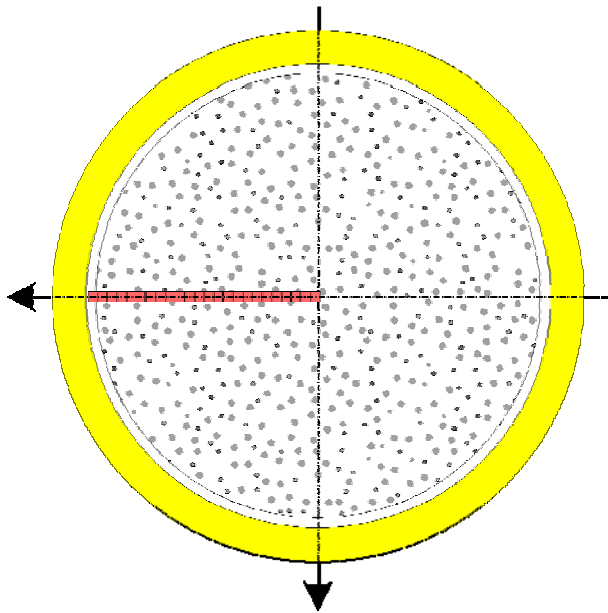


SIMS measurement principle

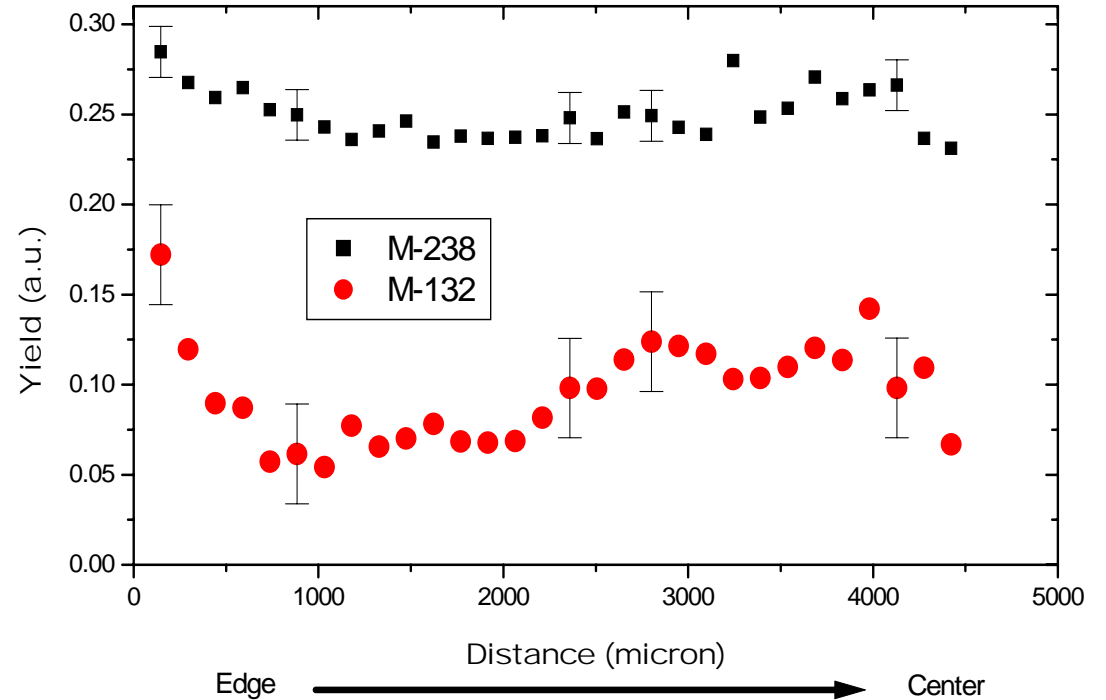


SIMS – Xe measurement in high burnup fuel

Line scan through the pellet radius

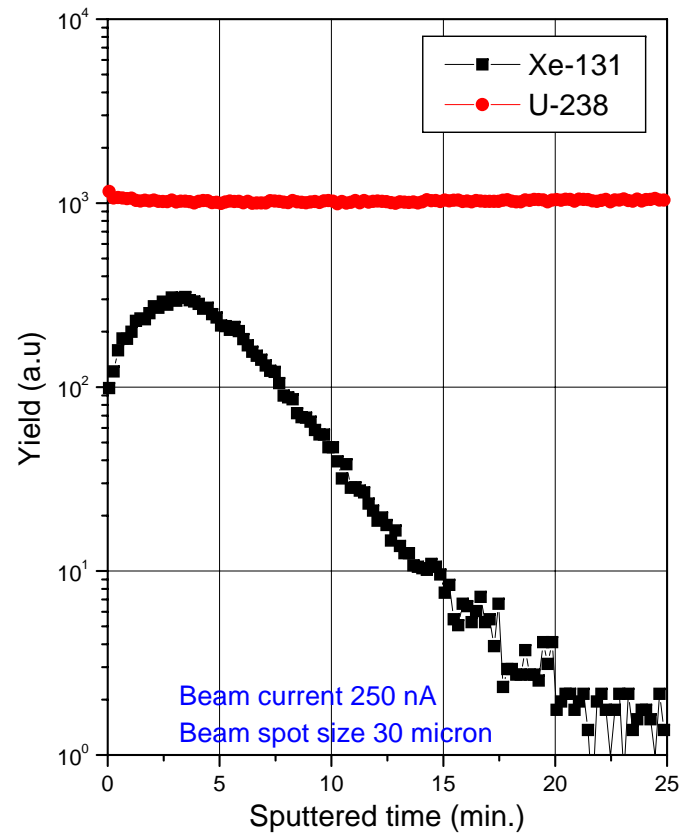


Linescan using SIMS on Fuel specimen



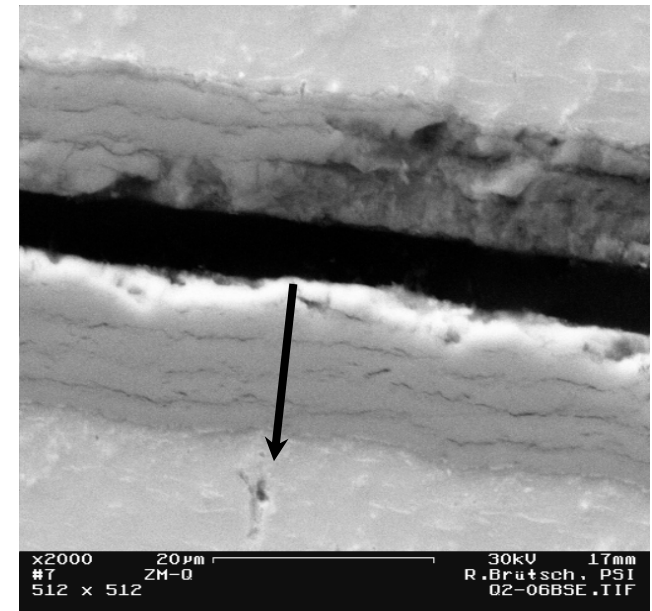
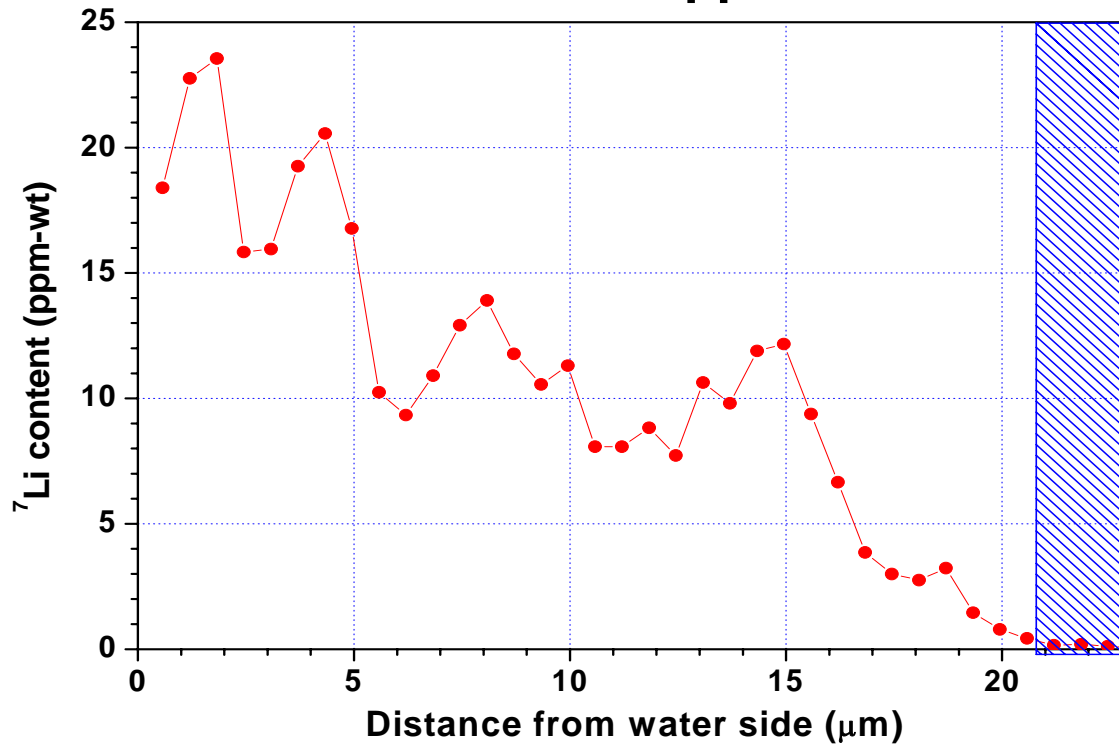
SIMS – Xe measurement in implanted UO_2

UO_2 pellet implanted with 500 keV $^{131}\text{Xe}^+$ to a dose of $2 \times 10^{16} \text{ cm}^{-2}$ in.



SIMS - Li quantitative distribution determination in the corrosion layer of irradiated Zircaloy

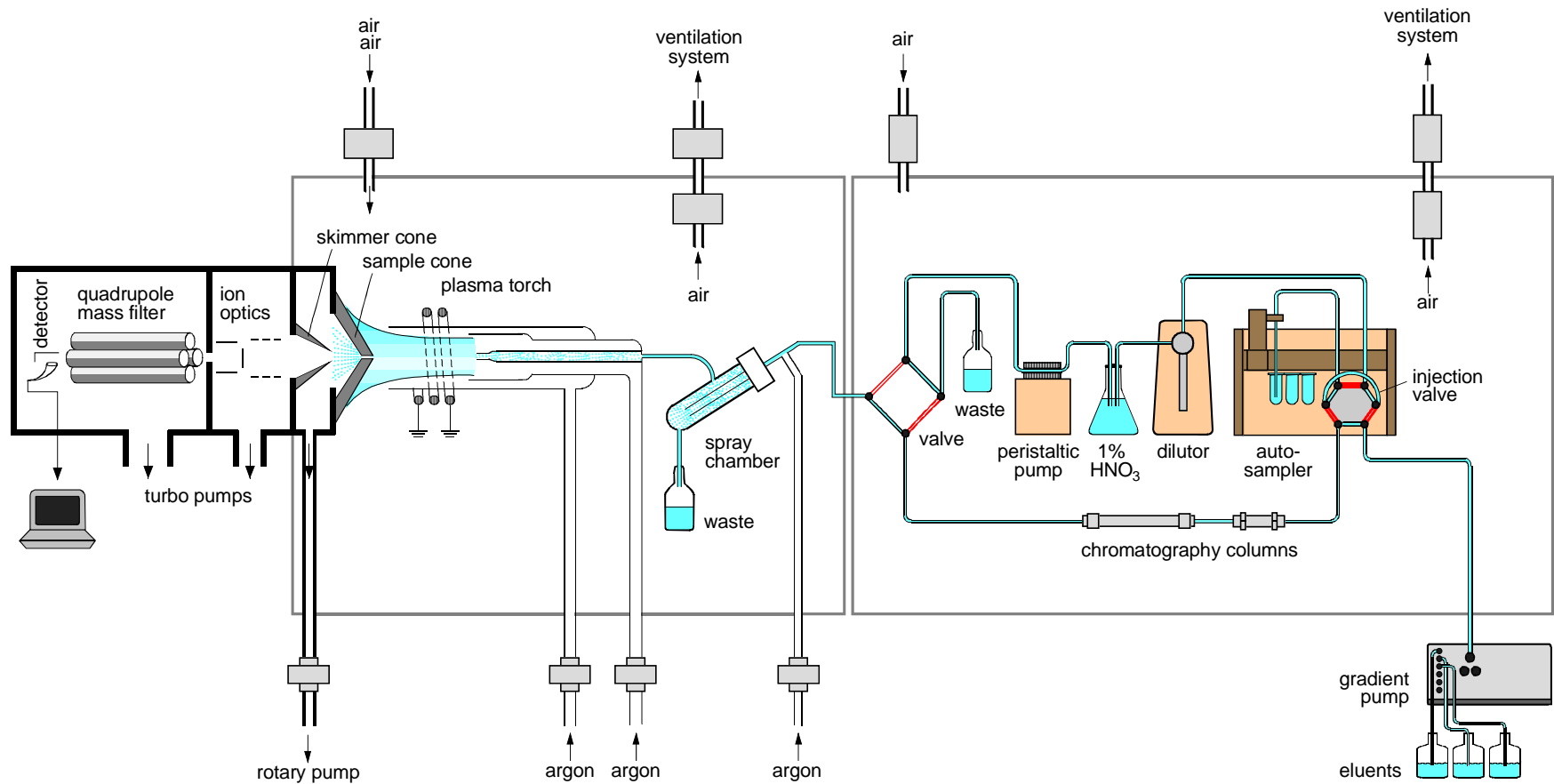
⁷Li content in ppm-wt



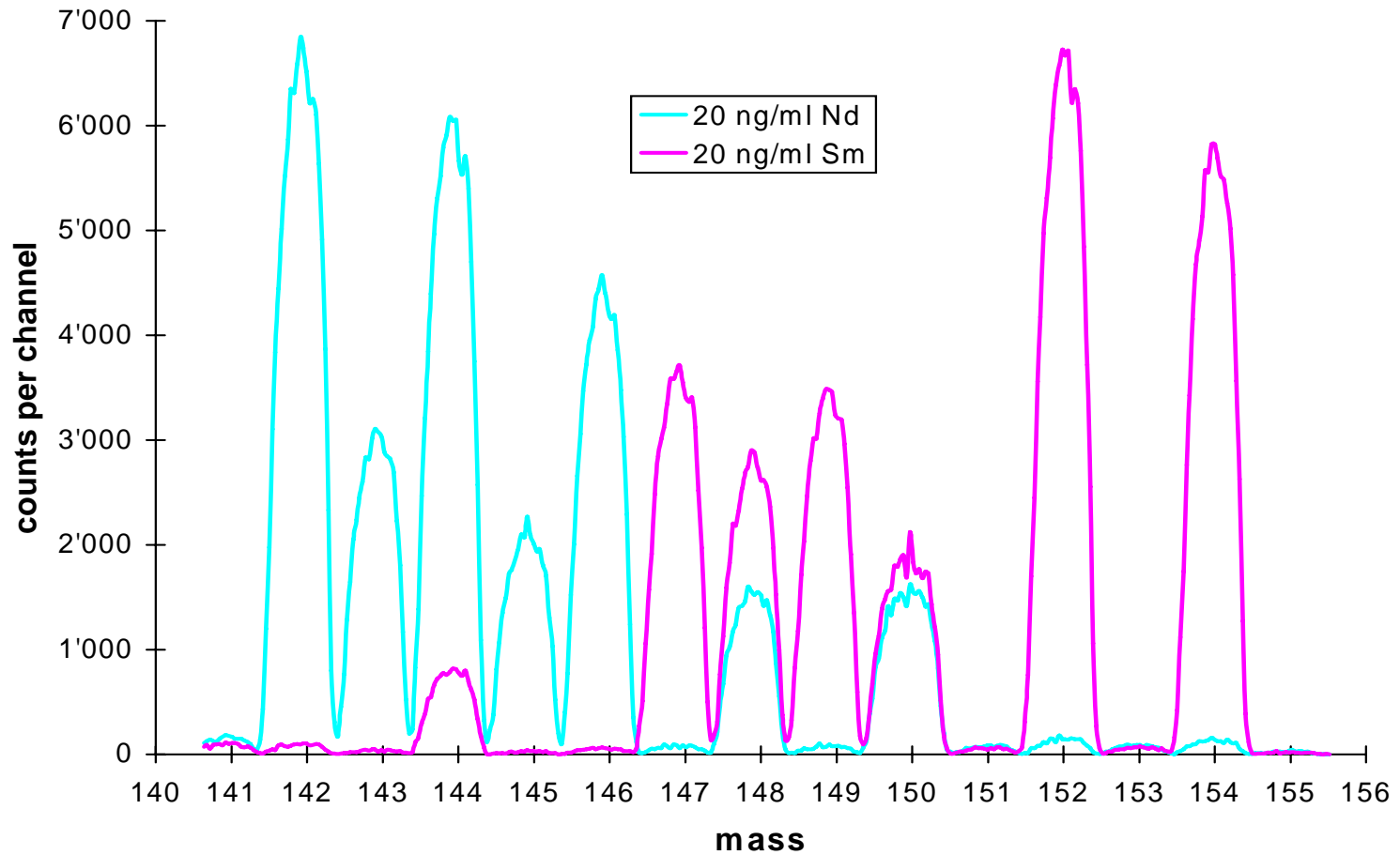
Isotope and Wet Chemical Analysis (Zlatan Kopajtic, Ines Günther-Leopold)

- Inductively Coupled Plasma Mass Spectrometer (mainly for liquid samples)
- Laser Ablation (for solid samples and gas inclusions)
- Gas Mass Spectrometer (fission gases)

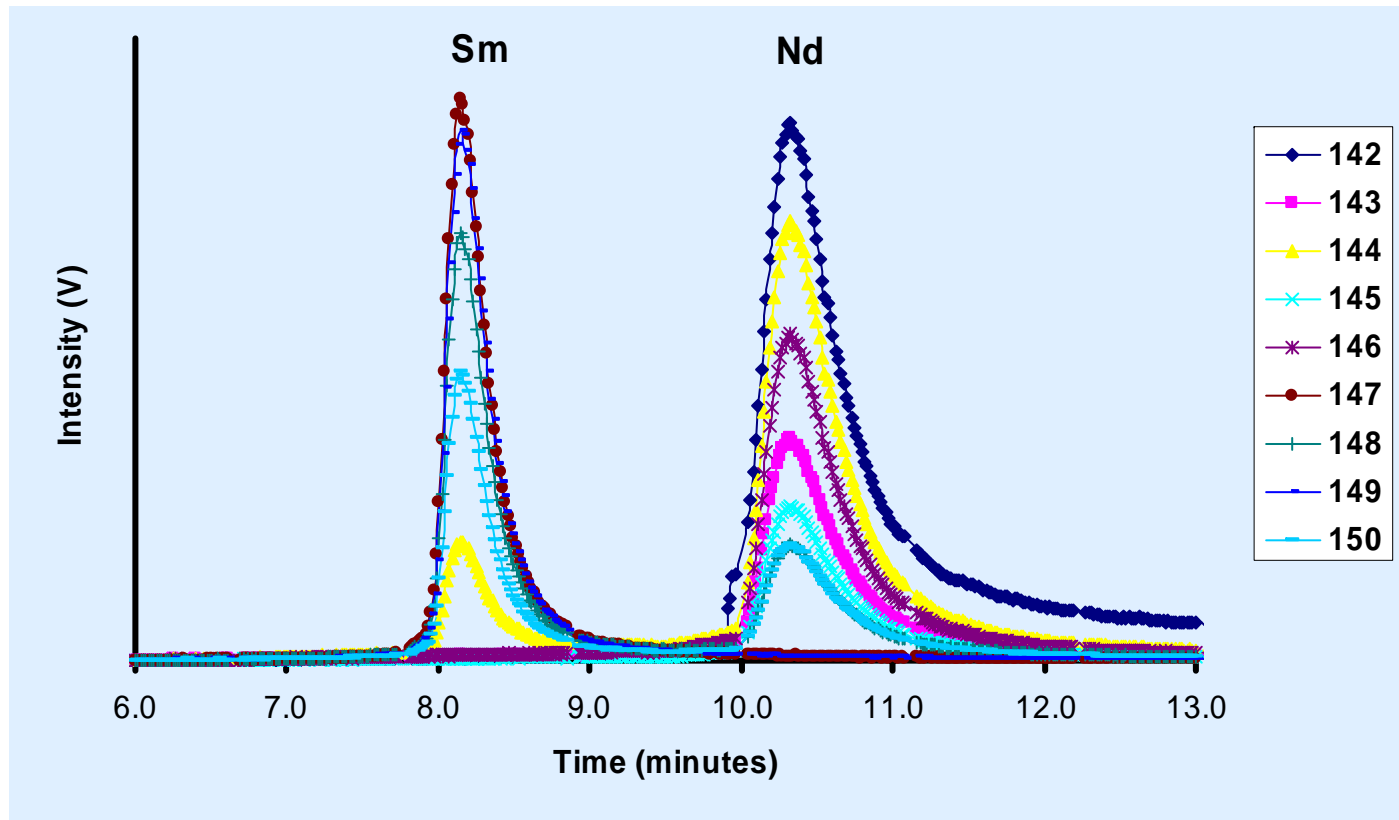
ICP Mass Spectrometry



ICP Mass Spectrometry



Separation of interfering elements by HPLC-MC-ICP-MS



Quantitative characterization of nuclear fuels

HPLC-MC-ICP-MS

^{234}U , ^{235}U , ^{236}U , ^{238}U
 ^{238}Pu , ^{239}Pu , ^{240}Pu , ^{241}Pu , ^{242}Pu
 ^{142}Nd , ^{143}Nd , ^{144}Nd , ^{145}Nd , ^{146}Nd , ^{148}Nd , ^{150}Nd
 ^{241}Am , $^{242\text{m}}\text{Am}$, ^{243}Am , ^{242}Cm , ^{243}Cm , ^{244}Cm , ^{245}Cm
 ^{90}Sr
 ^{133}Cs , ^{134}Cs , ^{135}Cs , ^{137}Cs
 ^{147}Sm , ^{148}Sm , ^{149}Sm , ^{150}Sm , ^{151}Sm , ^{152}Sm , ^{154}Sm
 ^{151}Eu , ^{153}Eu , ^{154}Eu , ^{155}Eu
 ^{147}Pm , ^{155}Gd

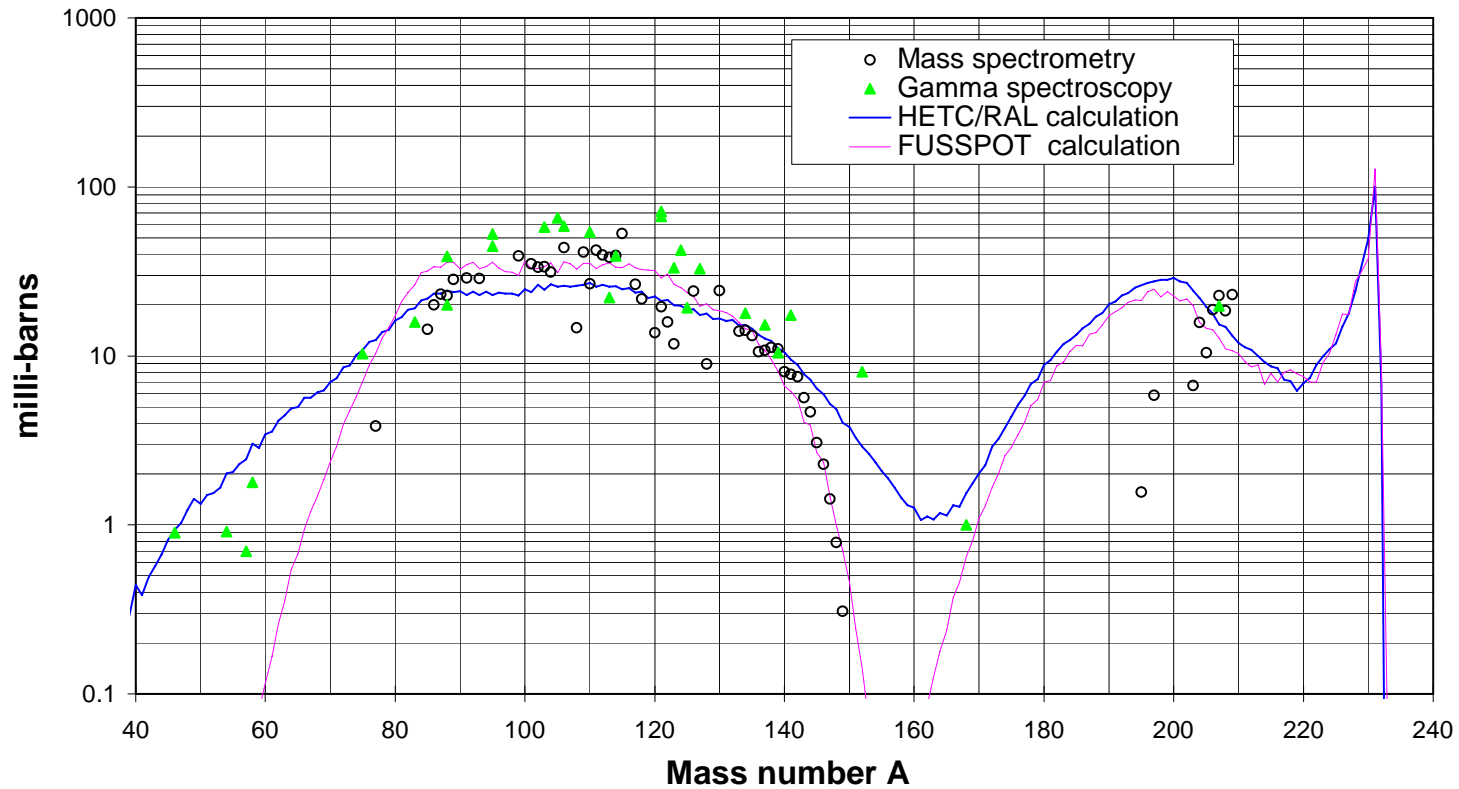
Q-ICP-MS

^{95}Mo , ^{99}Tc , ^{101}Ru , ^{103}Rh , ^{109}Ag , ^{237}Np

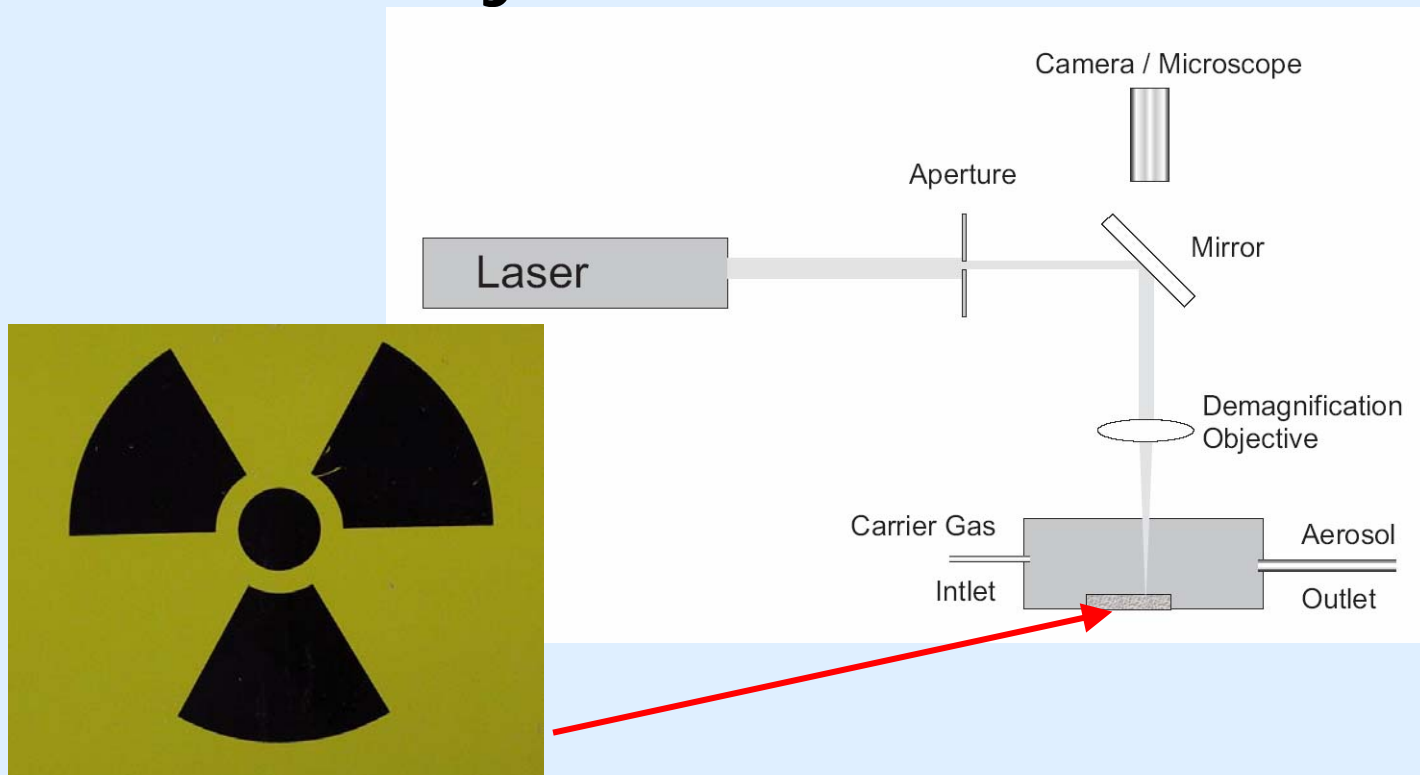
ATHENA

(Actinide transmutation using high energy accelerators)

Th-232 Isobaric Production Cross-Sections



A laser ablation ICP-MS system for the analysis of radioactive samples



Marcel Guillong, Peter Heimgartner, Ines Günther-Leopold, Matthias Horvath and Zlatan Kopajtic

Background and Motivation

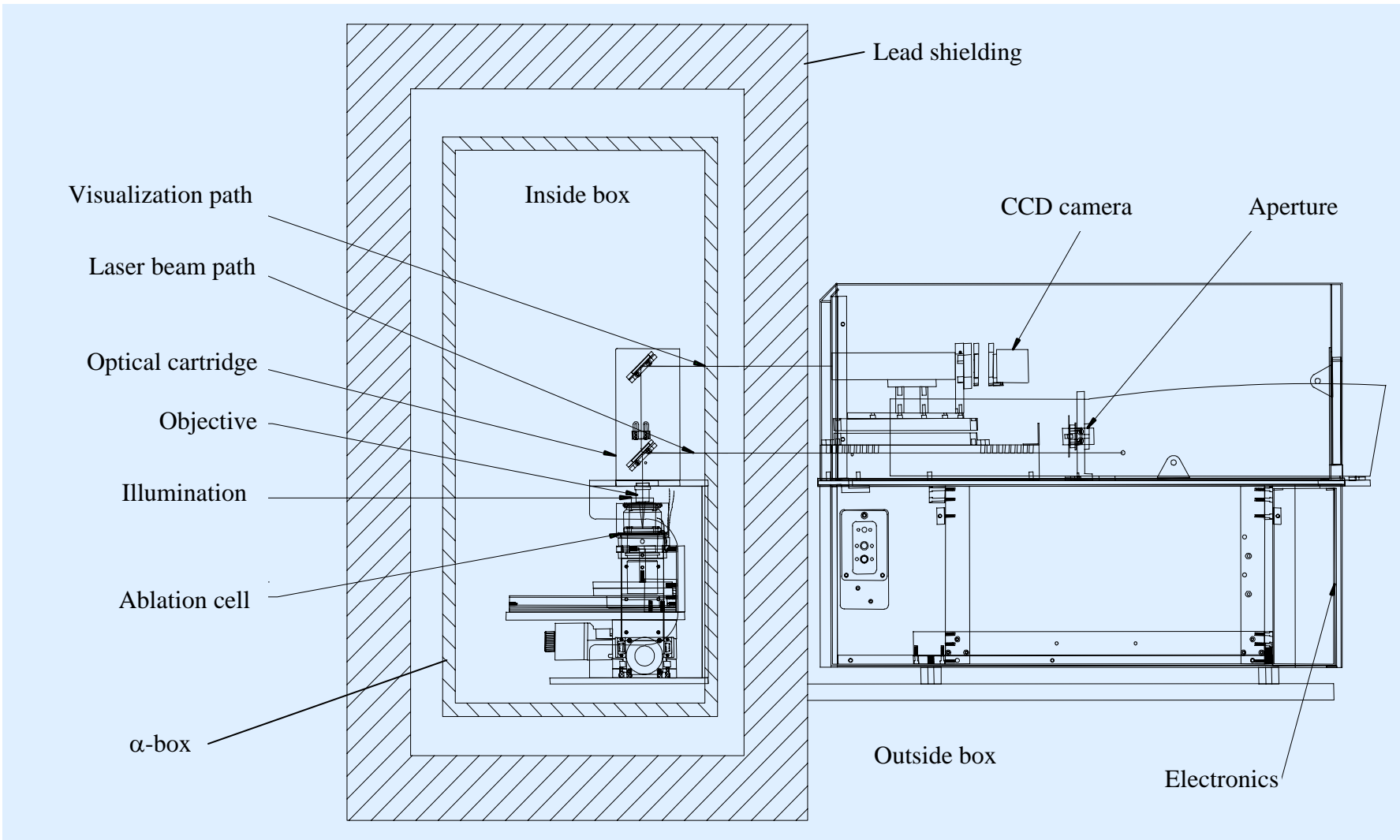
- Digestion, HPLC-MC-ICP-MS
(Bulk, very precise and time consuming)

- SIMS
(difficult quantification)

LA-ICP-MS a perfect tool?
(high spatial resolution, quantification possibilities)

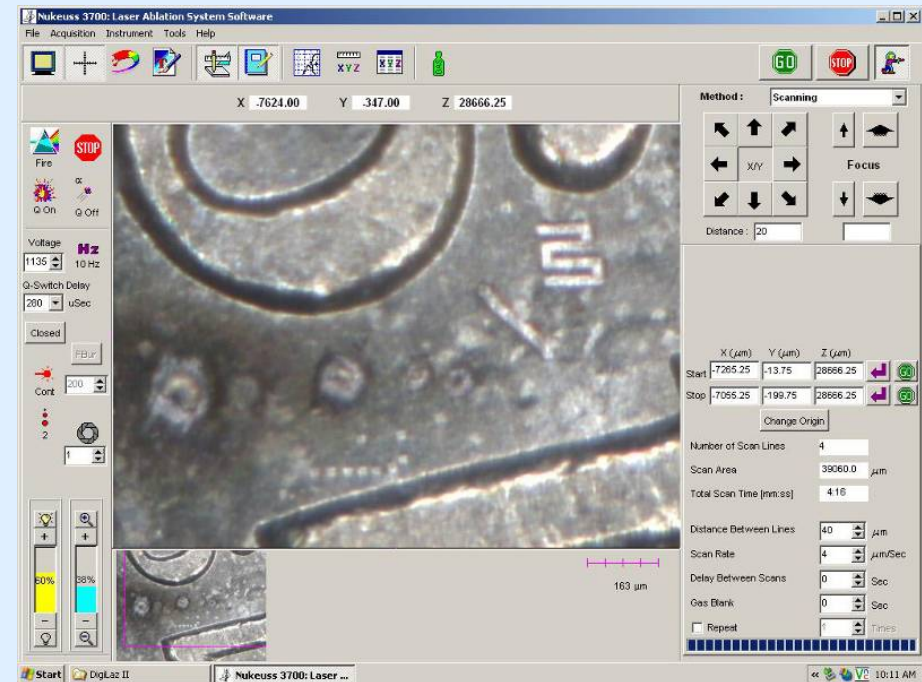
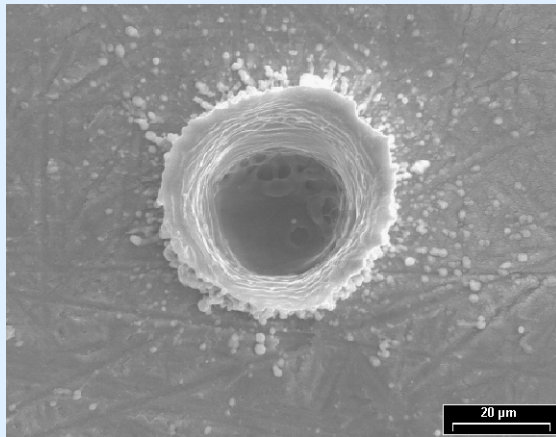


Instrumentation



Instrumentation

- CETAC LSX 500 / 3000 Nd YAG with 266 nm
- Output energy up to 60 mJ / pulse
- Crater sizes down to 10 μm (microns)

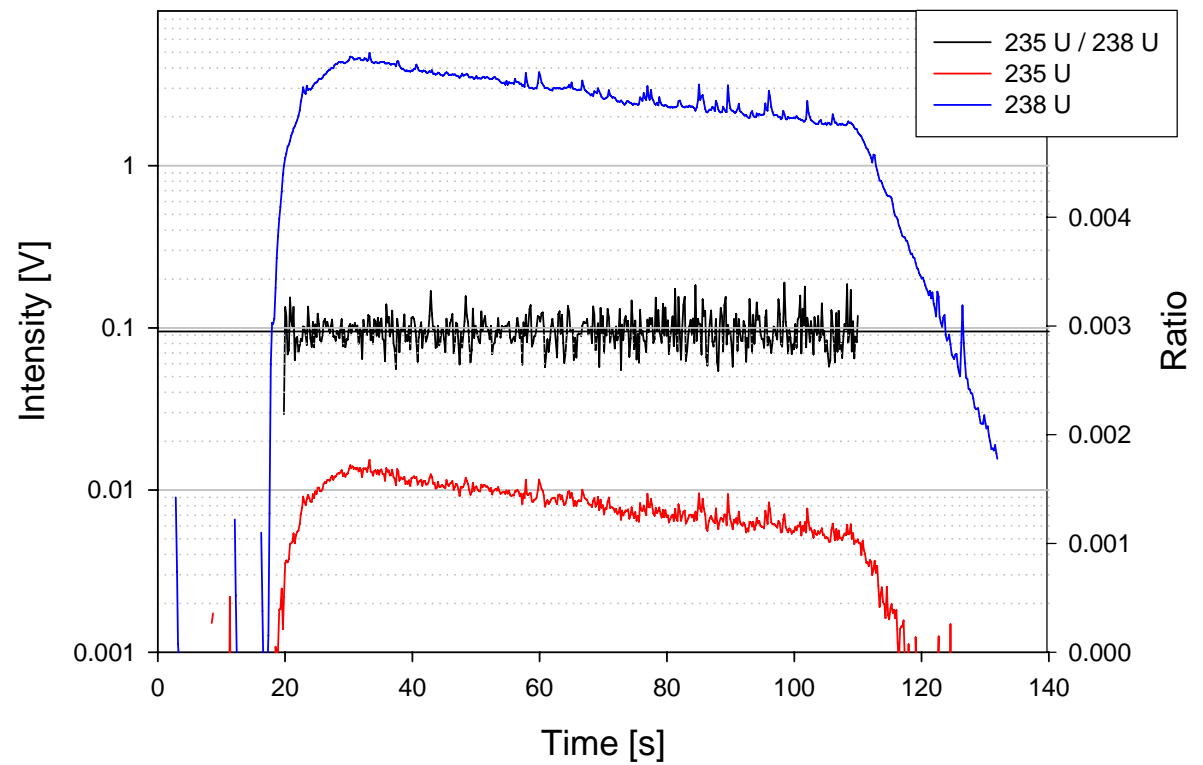




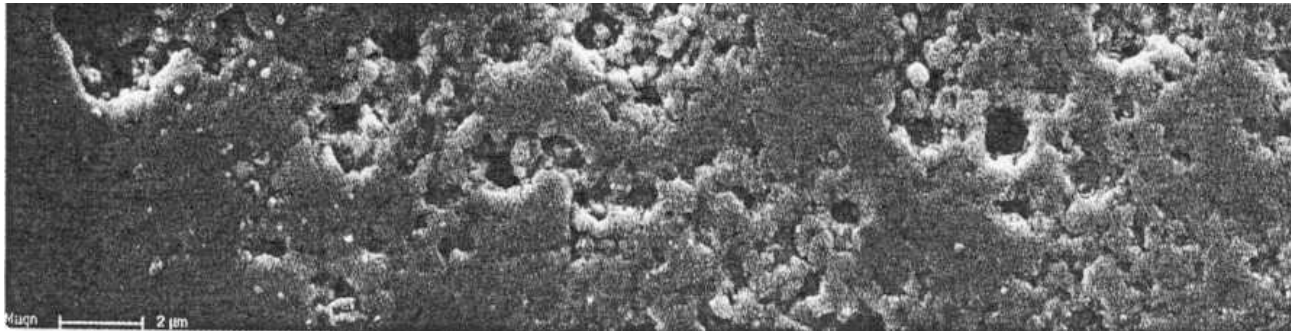
Transient
signal

Results

UO₂5/1
10Hz, 25 μm, 10 Jcm⁻², single hole



Outlook



JNM 257 (1998), 78-87

- High burn up structure (~ 80% ^{235}U are fissioned)
Xe and Kr in the matrix and in μm -bubbles under high pressure
Localize with EPMA, analysis with LA-ICP-MS