



SEM analyzes of powdered actinide compounds Implementation in a hot laboratory

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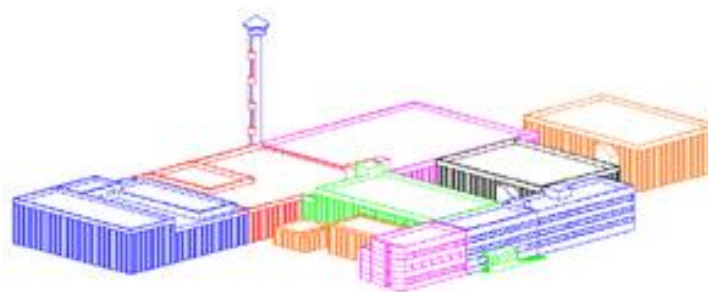
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SEM analyzes of powdered actinide compounds: Implementation in a hot laboratory

G. JOUAN

DEN/MAR/DTEC/SECA/LCC
Laboratoire de Caractérisation du Combustible

Marcoule – Atalante - INB 148

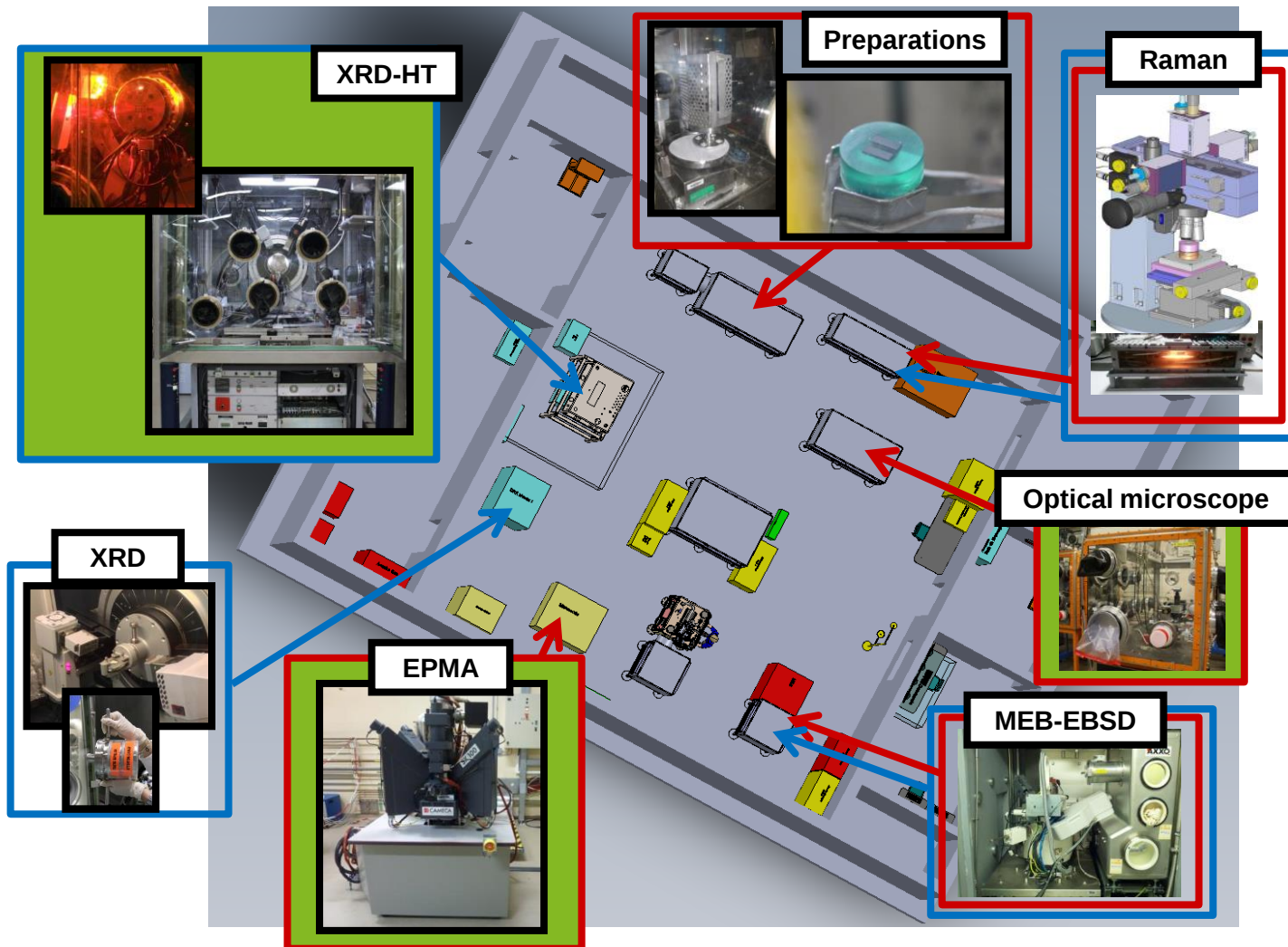


19 000 m² on 3 levels
12 shielded lines
17 laboratories (250 glove boxes)

LCC, laboratory L26 (end of 2017)

L26 $\approx 130 \text{ m}^2$

12 agents

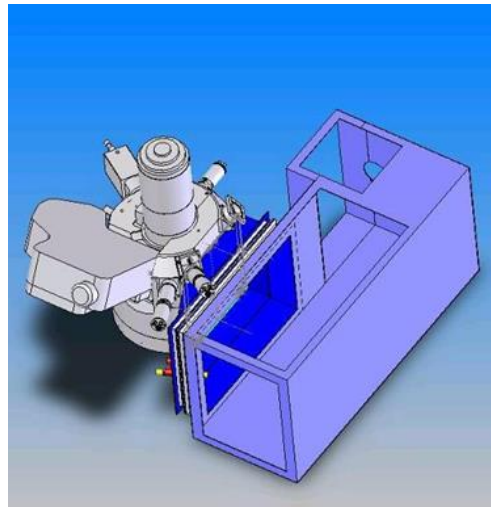


LES216 SEM-FEG for radioactives powders since 2010

* patent WO2013/068321 A1



+



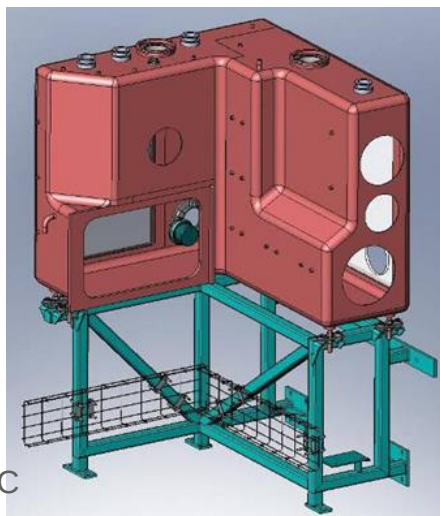
+



Hot cell already in the lab
(20 mCi)

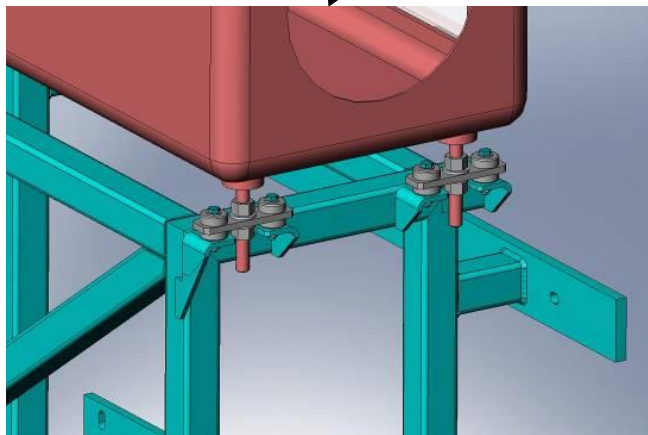
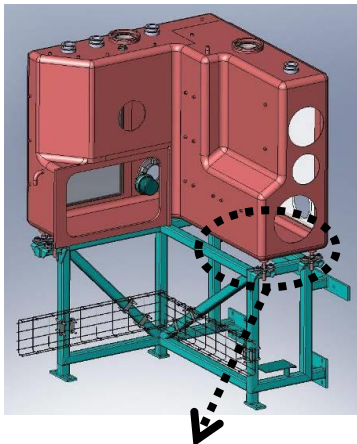


glove box
specially designed



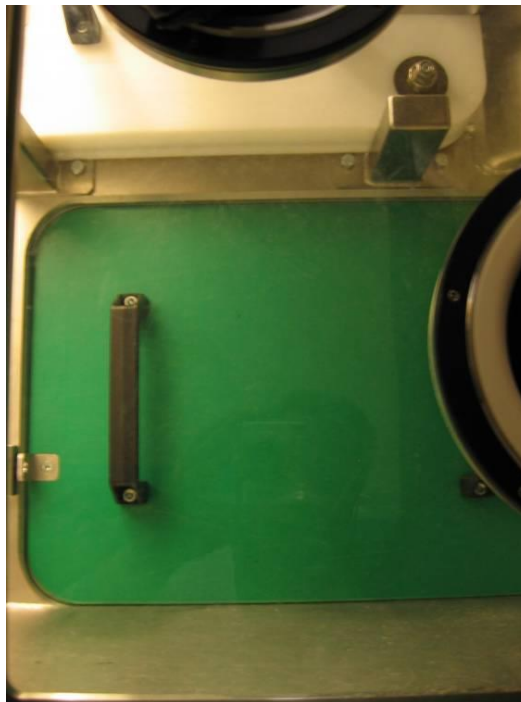
DEN/DTEC/SECA/LCC

Glove box on Silent blocks



DEN/DTEC/SECA/LCC

shutter



Pump distant



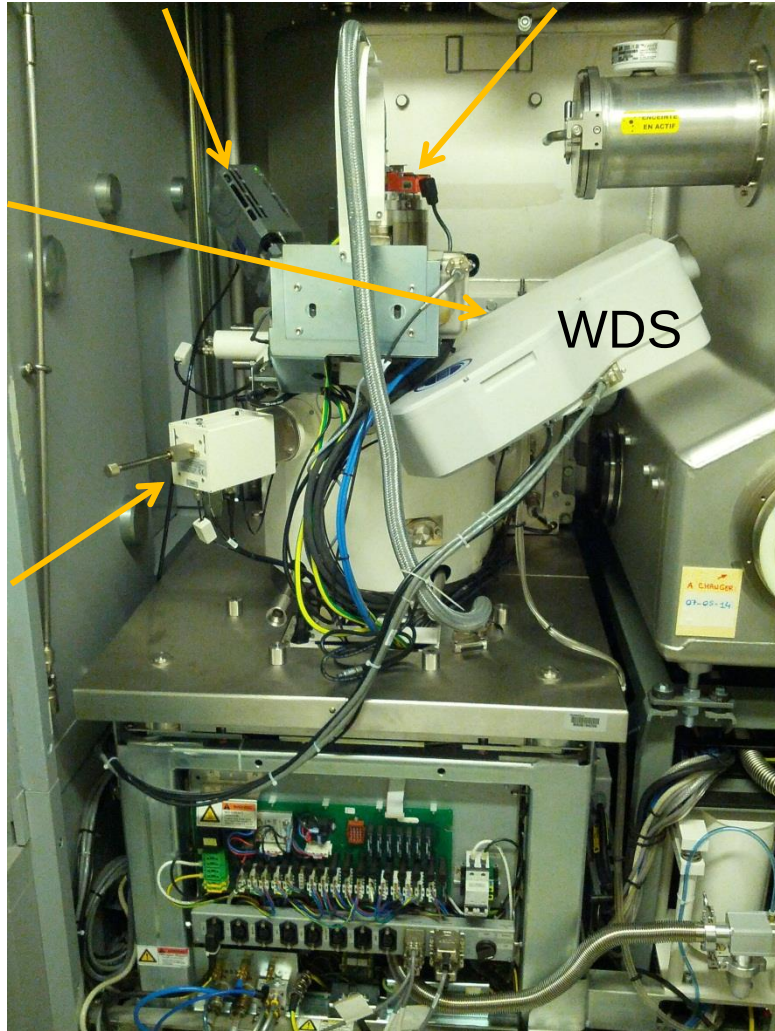
EDS

INLENS

WDS

Everhart-Thornley

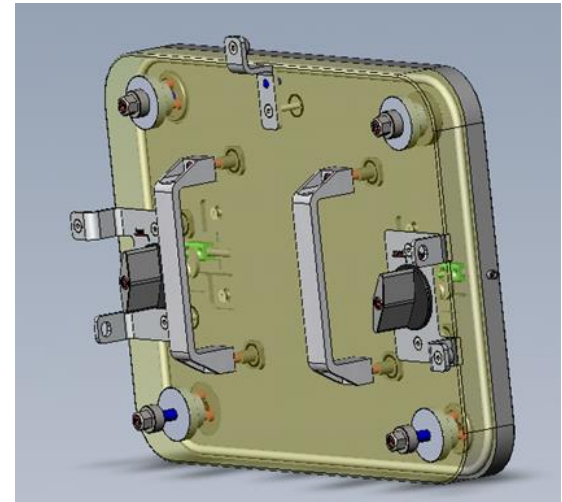
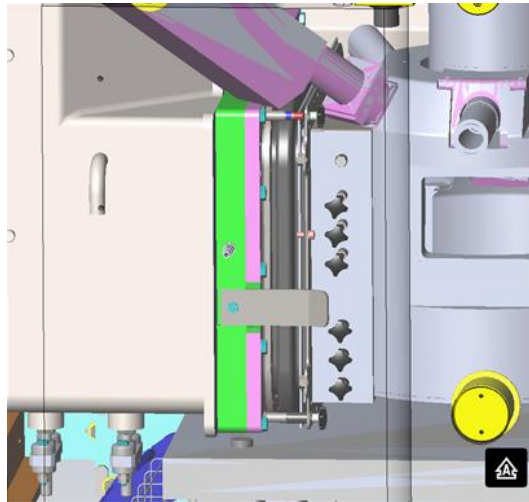
BSE



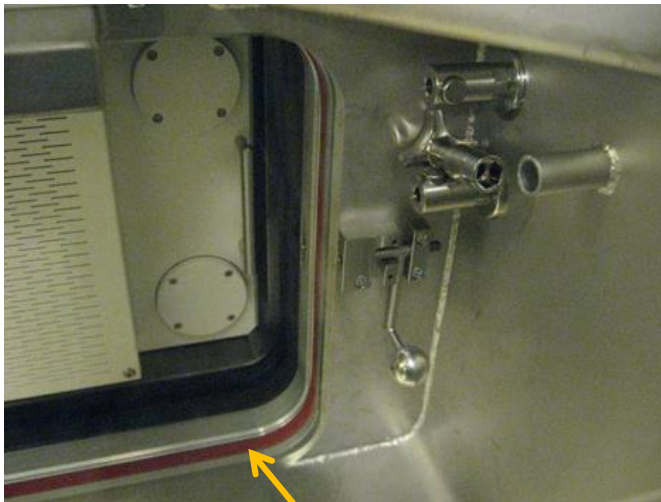
DEN/DTEC/SECA/LCC



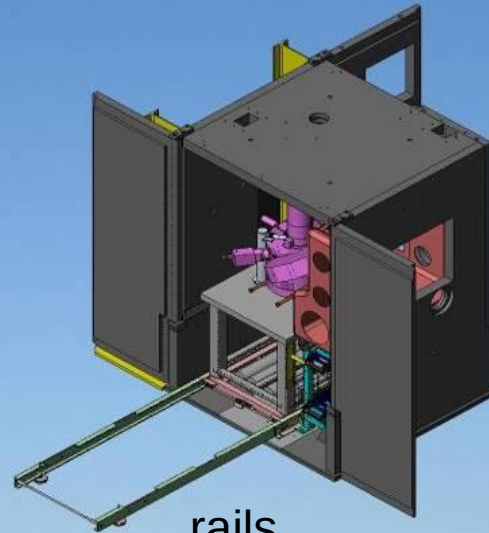
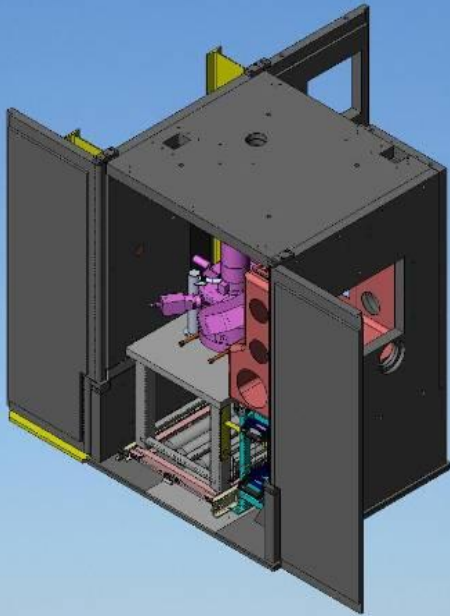
How to maintain it ?



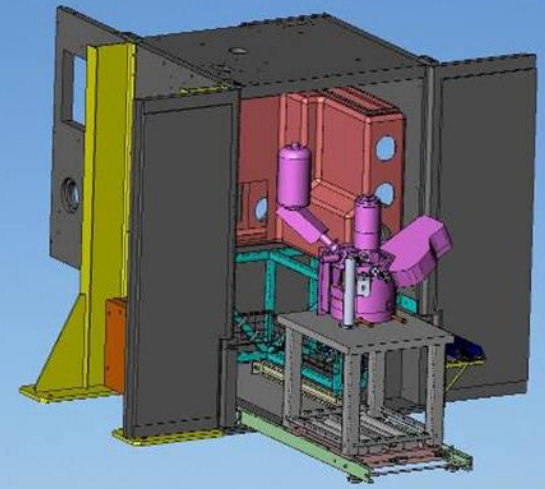
Disconnection without breaking containment



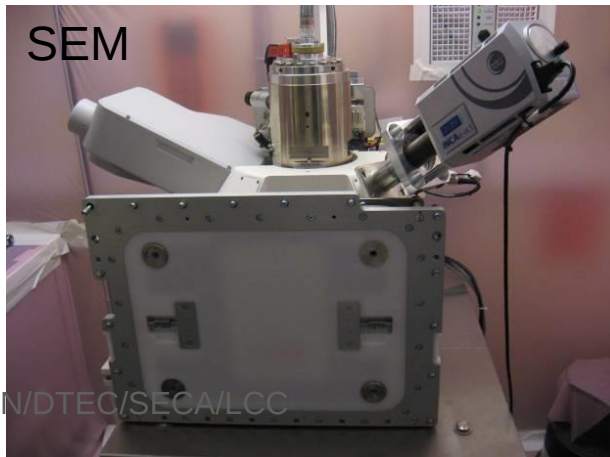
Inflatable joint



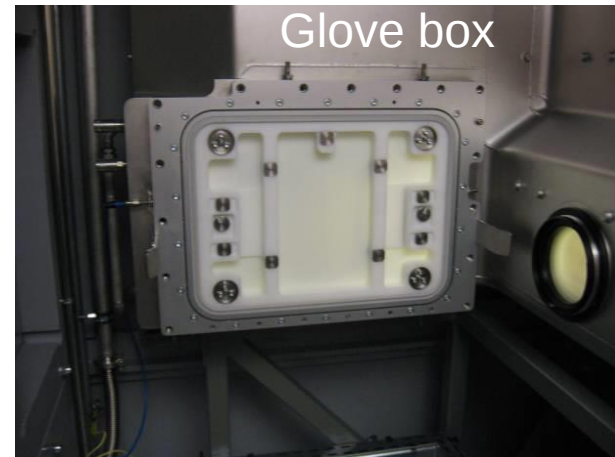
rails



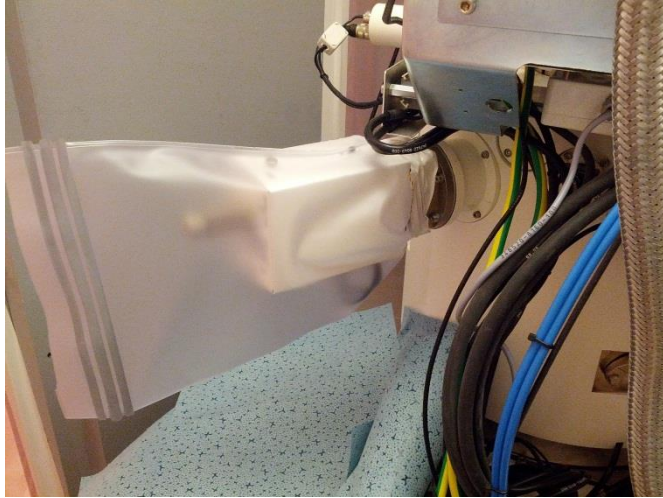
disconnection



DEN/DTEC/SECA/LCC



BSE scintillator replacement



Magnetic brake ajustement



EDS chip replacement



Diaphragms replacement

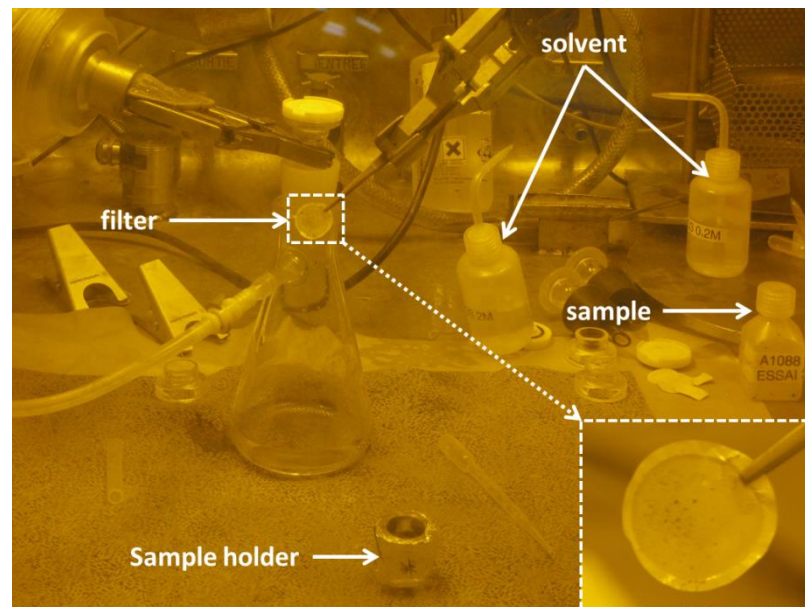


DEN/DTEC/SECA/LCC

Sample preparations and imaging examples

Glove
box

Hot cell



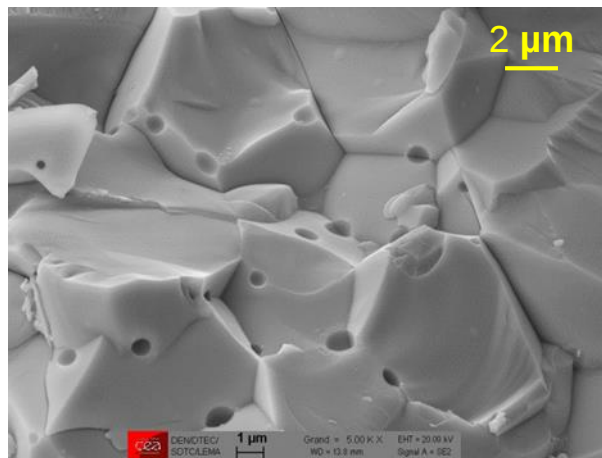
DEN/DTEC/SECA/LCC



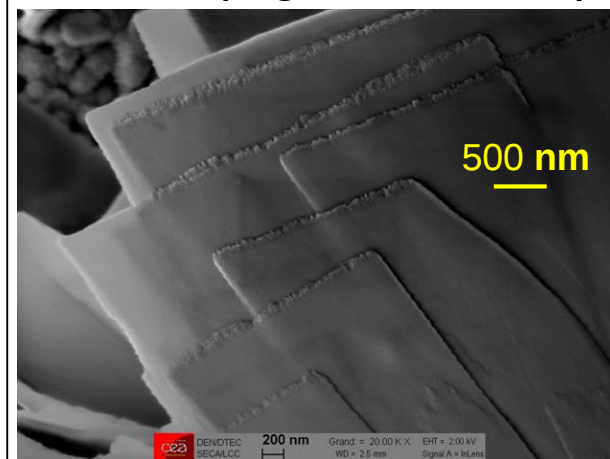
wood alloy



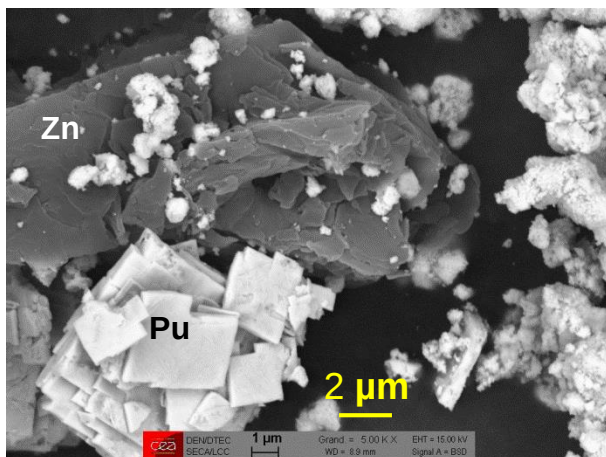
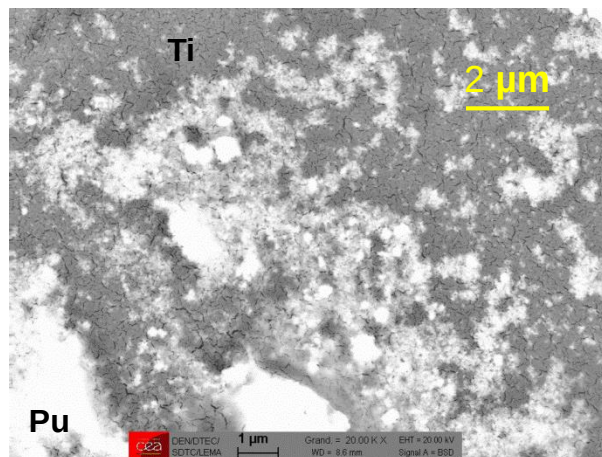
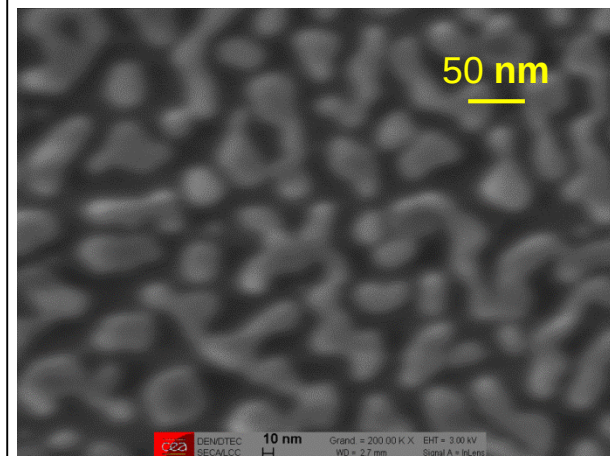
Everhart-Thornley

UO₂ pellet lixiviated with ironSintered U-AmO₂ powder

InLens (high resolution)

UO₂ pellet lixiviated with iron

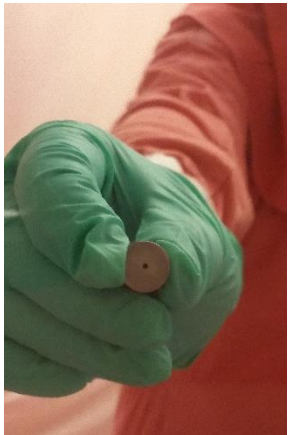
BSD (chemical contrast)

PuO₂/St-Zn mixingDissolution of PuO₂ assisted by US

Plutonium colloid

EDS measurements

Special EDS configuration



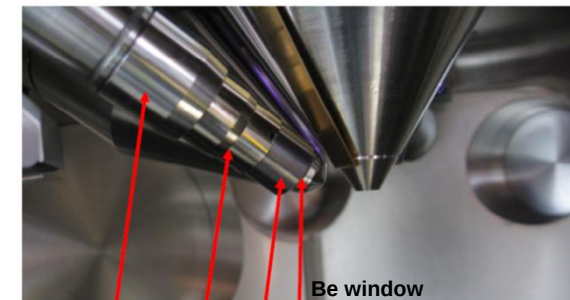
Be window used as a shield
 +
 EDS/sample distance increased
 +
 0.5 mm Collimator of (standard at 2 mm)



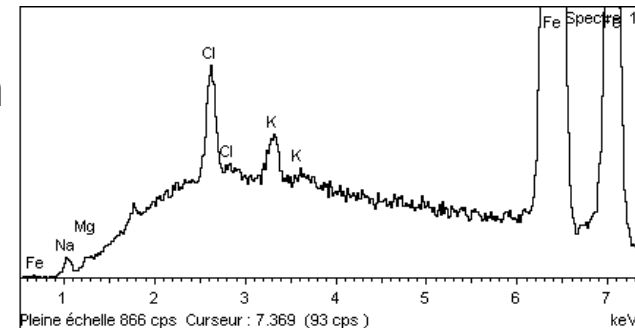
First element measurable : Na
 +
 Low counts measured at 15 kV minimum
 with the largest diaphragm



Bad spatial resolution
Long counting time
Destruction of many samples



SDD Xact Vitus
 Collimator
 Be window
 8 - 12 - 25



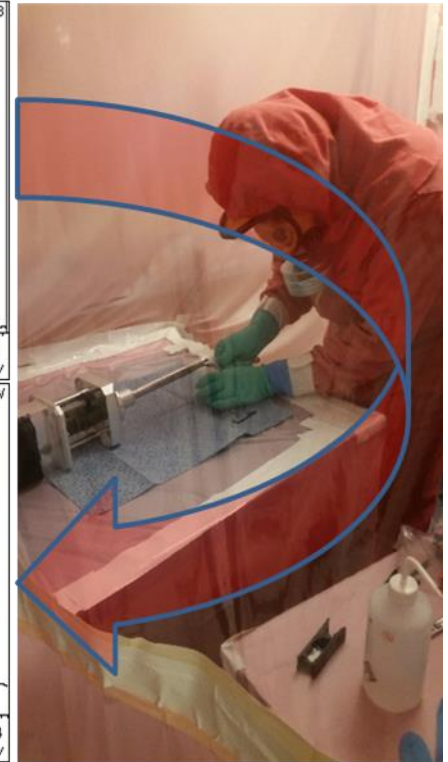
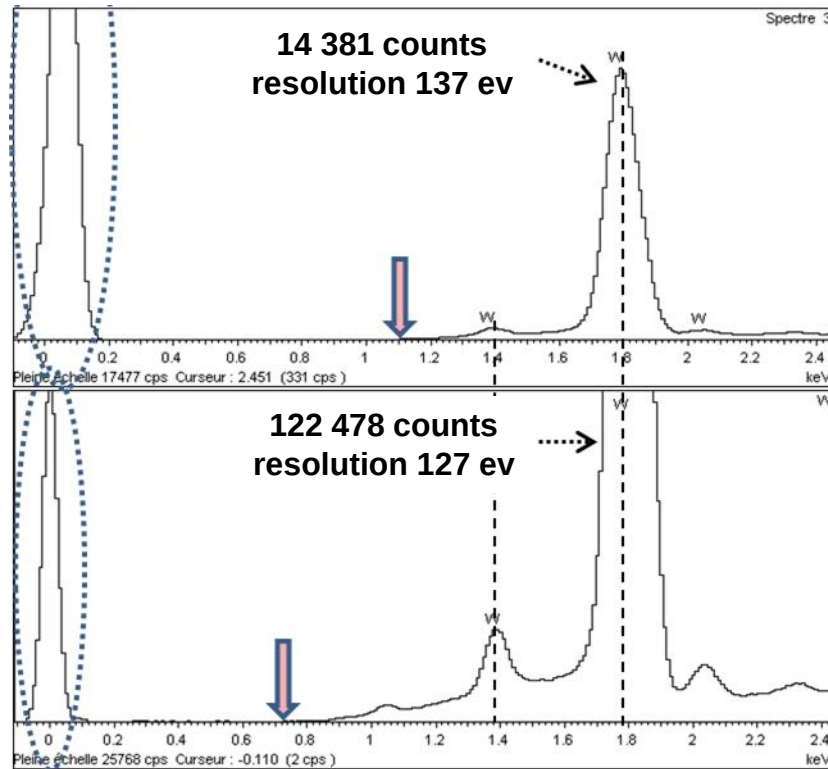
Special EDS configuration

- 25 μm Be window
- high distance
sample / detector
- 4 years old chip

- 12 μm Be window
- medium distance
sample / detector
- New chip



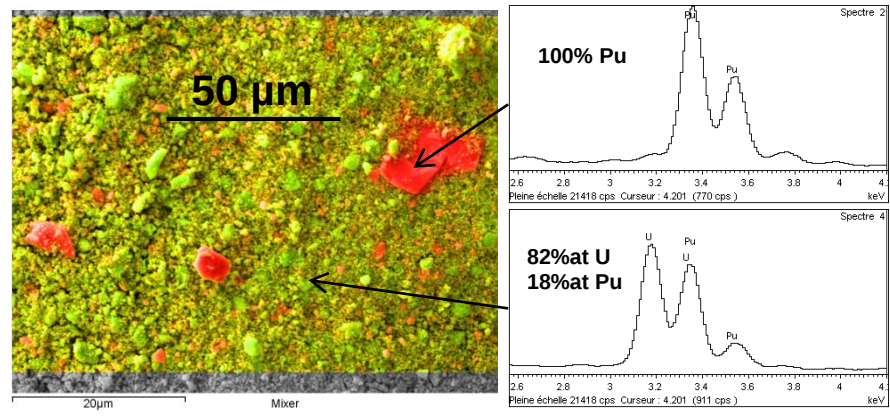
Calibration is essential !



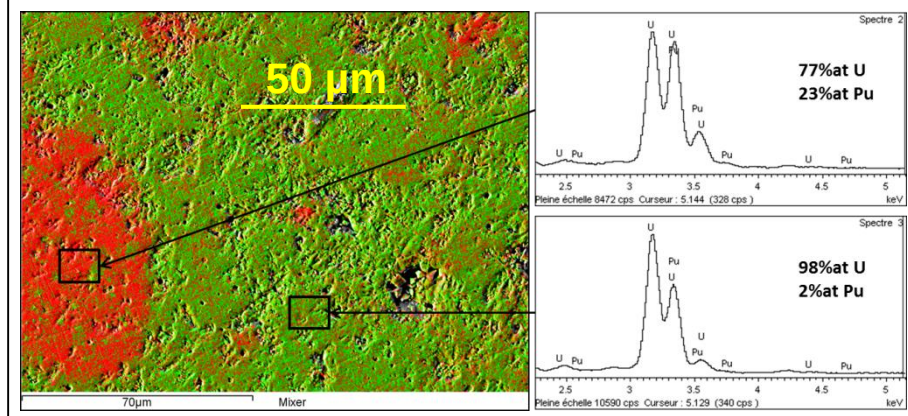
MAC55 Standard

Home made standard for actinides

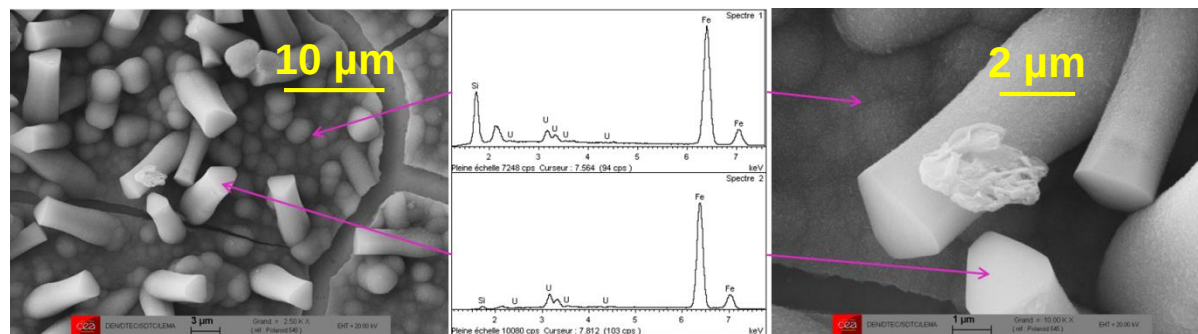
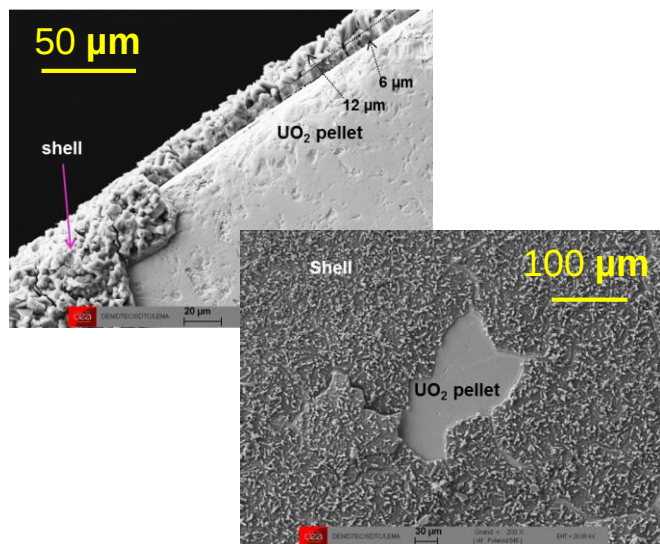
Plutonium / Uranium mapping (red/green)

UO₂ / PuO₂ powders mixed

MOX sintered pellet



Spectrum on UO₂ pellet lixiviated in water for 1 year in presence of an iron foil

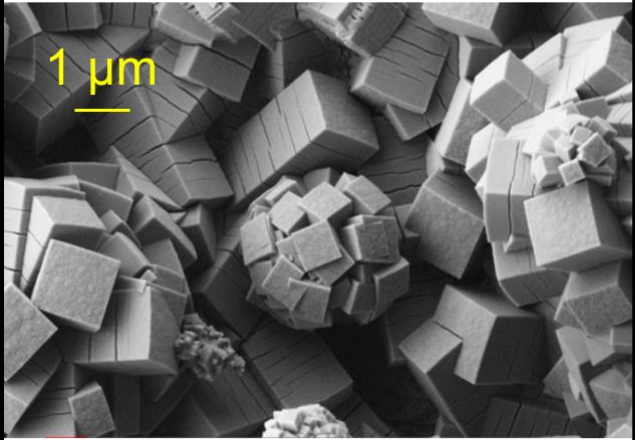


Images and EDS on Mo/Zr precipitates (raw)

22h of precipitation (other experiment during 29h et 35h)

Standards conditions

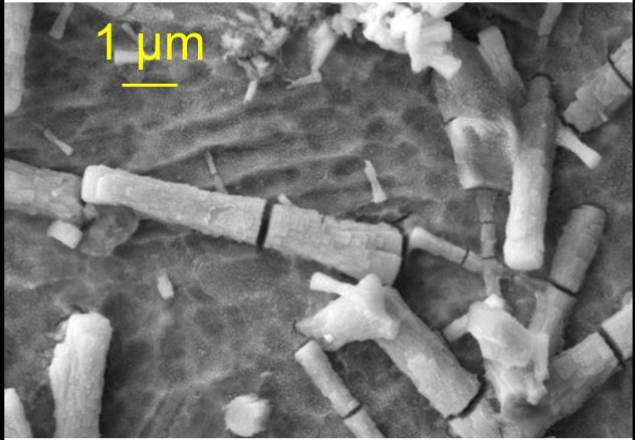
1 μm



This SEM image shows a dense collection of Mo/Zr precipitates under standard conditions. The precipitates are primarily cubic in shape, with some smaller, more irregular fragments interspersed. The overall texture is granular and three-dimensional.

Additive before dissolution

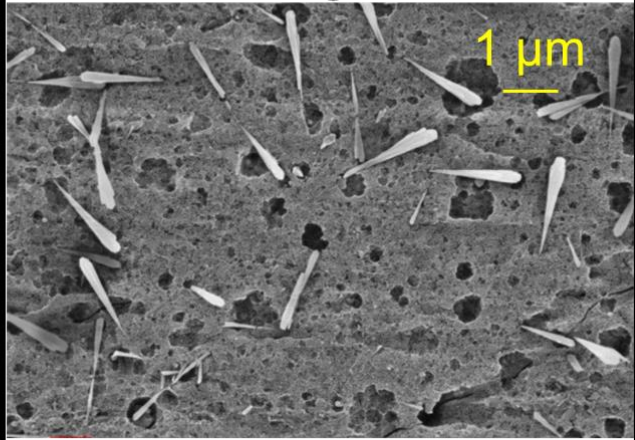
1 μm



This SEM image shows Mo/Zr precipitates when an additive was present before dissolution. The precipitates appear as elongated, needle-like or rod-like structures, contrasting with the cubic shapes seen in the standard conditions.

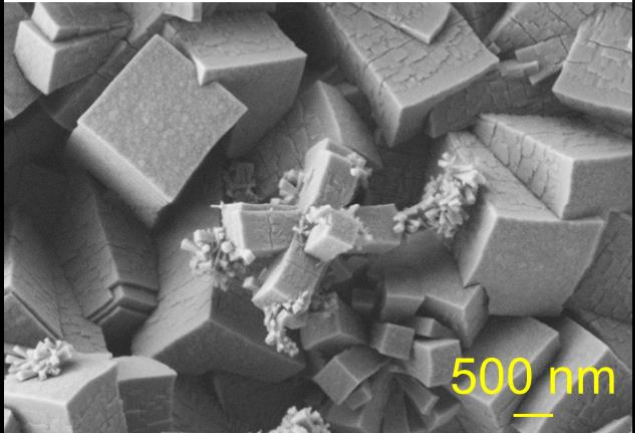
Additive through dissolution

1 μm



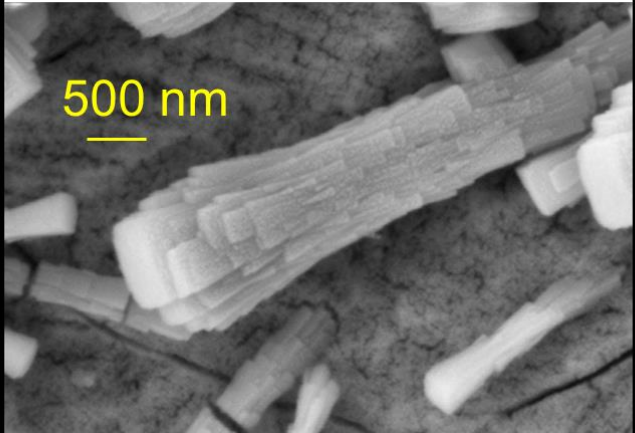
This SEM image shows Mo/Zr precipitates when an additive was present during the dissolution process. The precipitates are highly elongated and needle-like, with some showing a distinct layered or fibrous internal structure.

500 nm



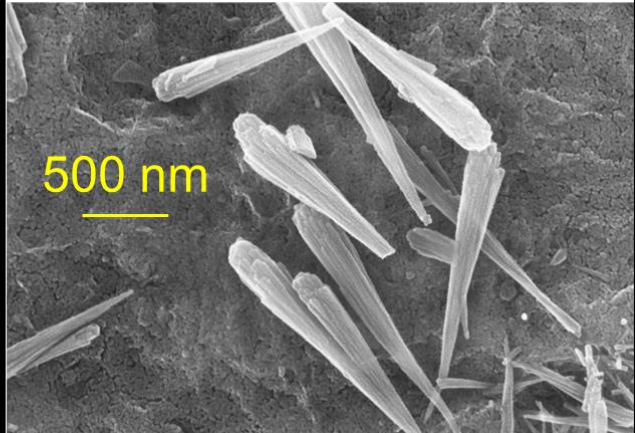
This higher magnification SEM image of the standard conditions sample shows the cubic precipitates in more detail. They are well-defined, polyhedral crystals with flat faces and sharp edges.

500 nm



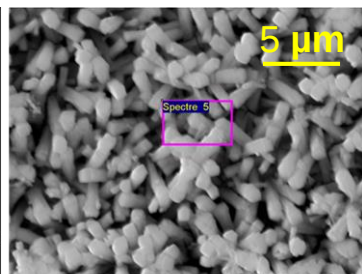
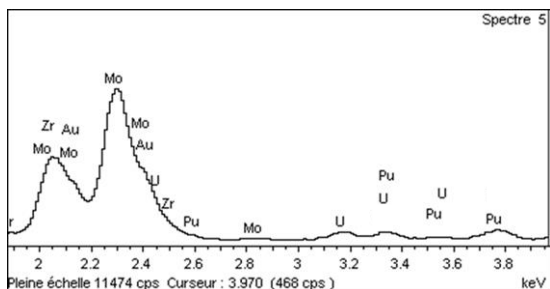
This higher magnification SEM image of the 'additive before dissolution' sample shows the elongated precipitates more clearly. They appear as thin, parallel plate-like or rod-like structures.

500 nm

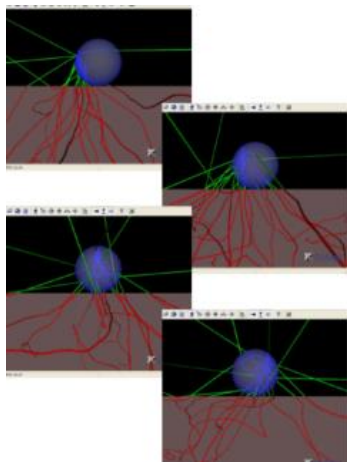


This higher magnification SEM image of the 'additive through dissolution' sample shows the needle-like precipitates in detail. They are long, thin, and exhibit a clear layered morphology, suggesting a specific crystal growth habit.

Half-quantitative results on rough surfaces ?



The supra 55 is devoted to **powders** characterisation



55A	Zr *	Mo*	M *	U*	Pu*
average	29.1	57.6	6.2	6.2	0.9
SD	1.40	2.76	0.62	2.93	0.16
RSD	5%	5%	10%	72%	18%
56B	Zr *	Mo*	M *	U*	Pu*
average	29.8	59.8	5.4	4.3	0.8
SD	1.79	1.59	0.91	1.12	0.23
RSD	6.0%	2.6%	17.7%	24.9%	30.9%
57C	Zr *	Mo*	M *	U*	Pu*
average	37.6	55.0	4.5	2.1	0.9
SD	7.14	7.07	0.91	0.49	0.32
RSD	17%	14%	22%	21%	30%

32 spectrum

18 spectrum

35 spectrum



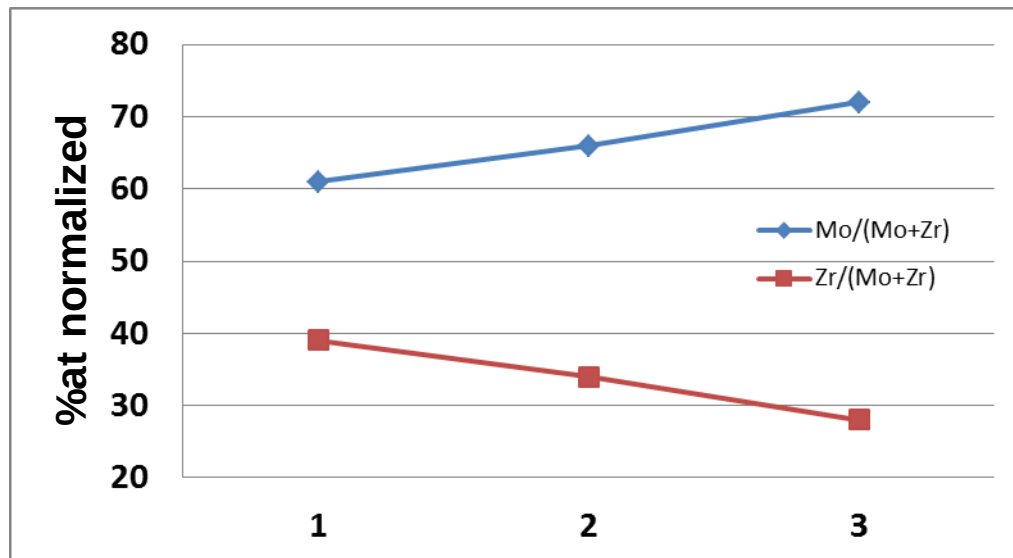
EDS results are average on numerous spectrum
Number of spectrum depends of the SD and RSD

Images and EDS on Mo/Zr precipitates (raw)

■ Time influence on composition is insignificant

%at*	Mo	Zr	M	Mo/(Mo+Zr)	Zr/(Mo+Zr)	(Mo+M)/(Mo+Zr+M)
1 - while	56	36	6	61%	39%	63
2 - before	58	30	5	66%	34%	68
3 - without	70	27	0	72%	28%	72

* (% incertitude not include)



■ Mo substitution ?

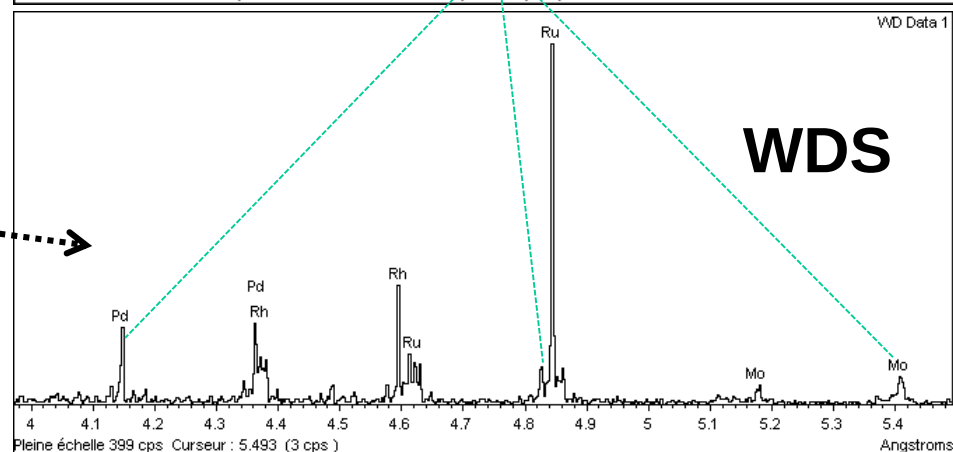
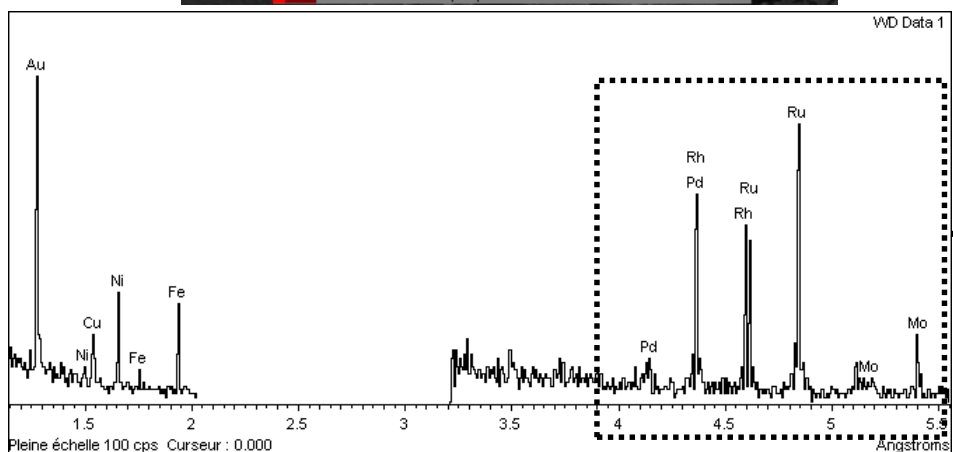
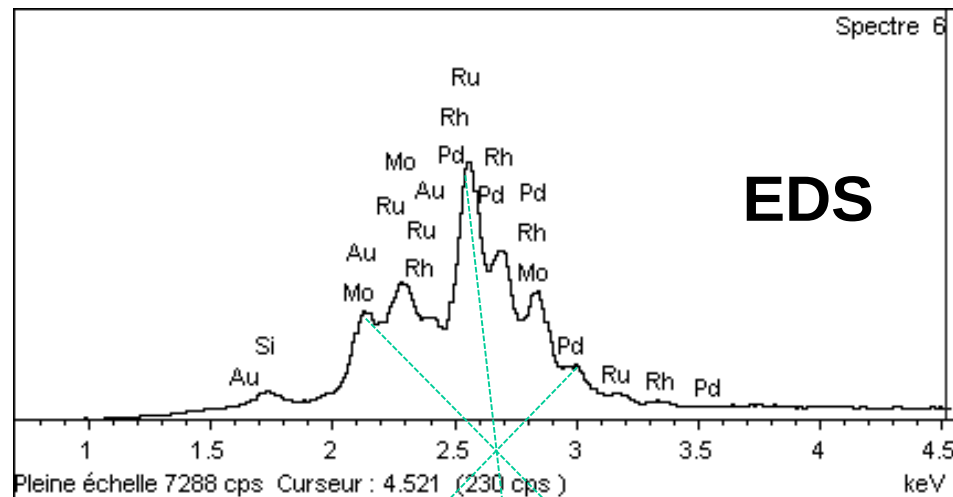
close ionique radius

Octahedral coordination for both

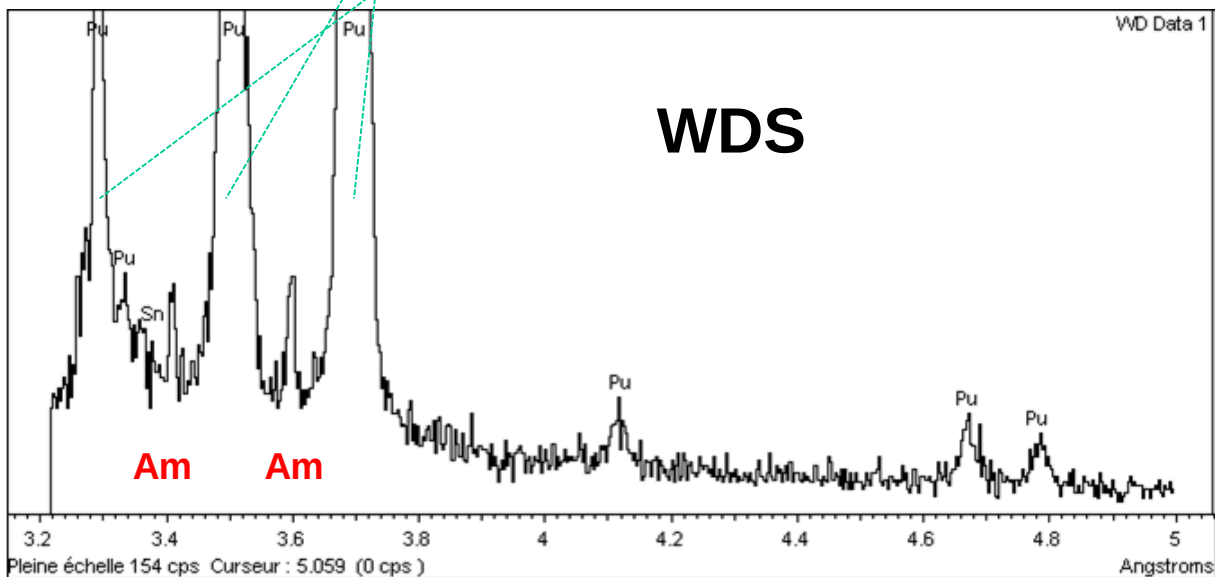
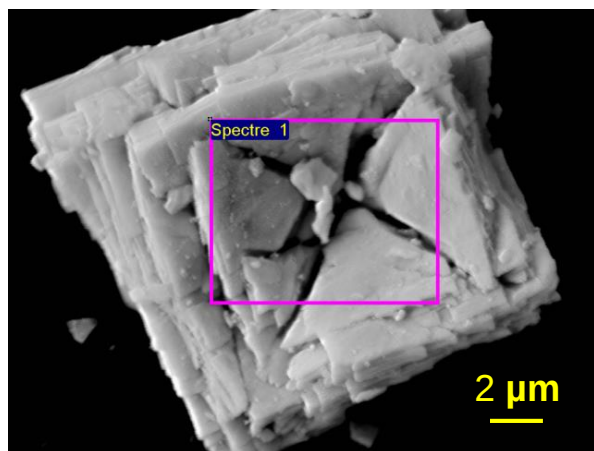
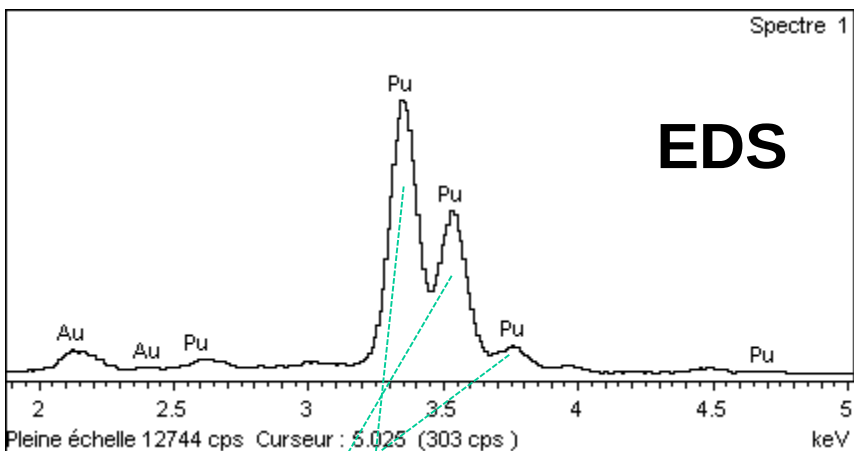
■ XRD measurements to confirm

WDX measurements

Exemples on dissolution residues of irradiated fuel

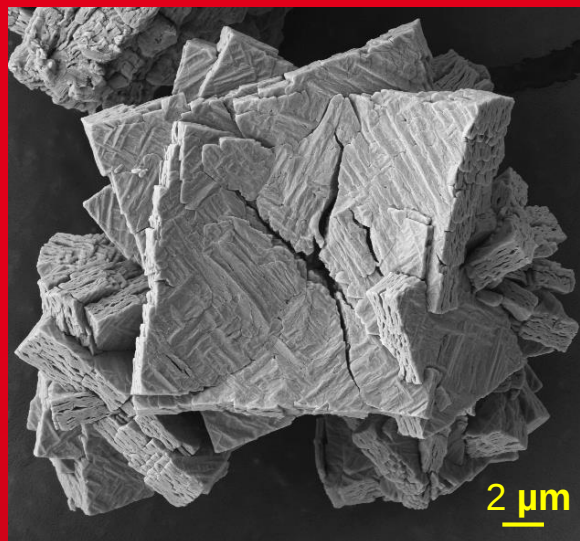


Americium detection in PuO_2 powders





Pu oxalate



*Thank you
for your attention*

Commissariat à l'énergie atomique et aux énergies alternatives
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