SEM analyzes of powdered actinide compounds Implementation in a hot laboratory

G. Jouan

DE LA RECHERCHE À L'INDUSTRIE

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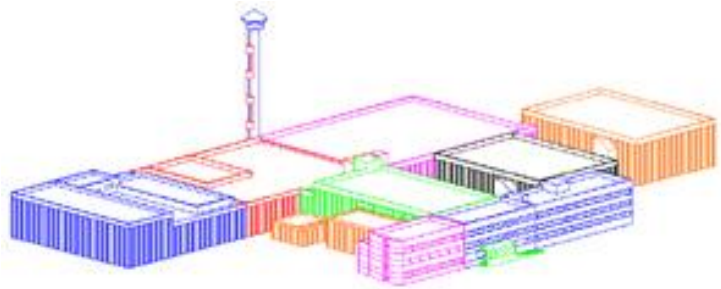
DEN/MAR/DTEC/SECA/LCC
Laboratoire de Caractérisation du Combustible

Marcoule – Atalante - INB 148

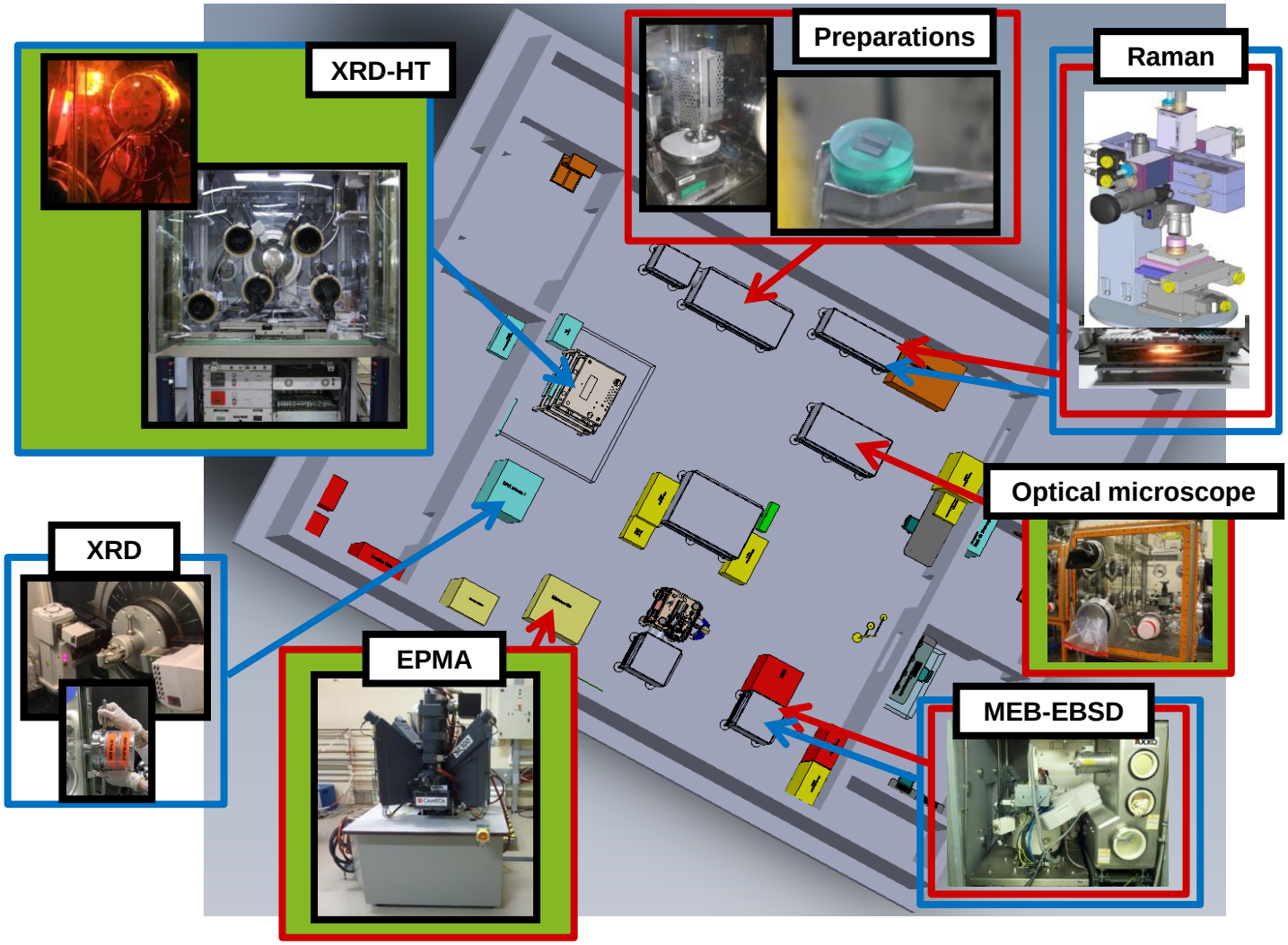


19 000 m² on 3 levels
12 shielded lines
17 laboratories (250 glove boxes)

LCC, laboratory L26 (end of 2017)

L26 ≈ 130 m²

12 agents



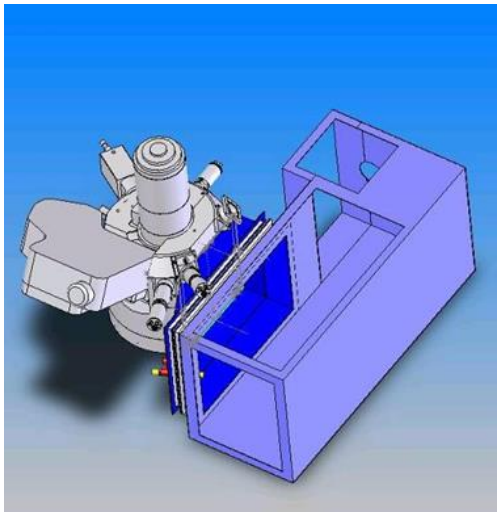
LES216 SEM-FEG
for radioactives powders

since 2010

SEM-FEG ZEISS SUPRA 55 WDS

* patent WO2013/068321 A1

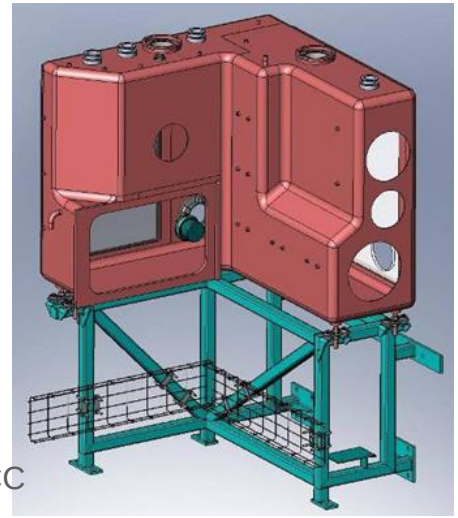
+



+

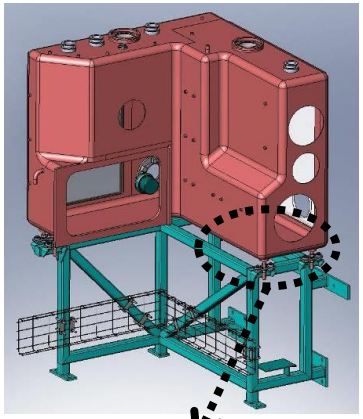
Hot cell already in the lab
(20 mCi)

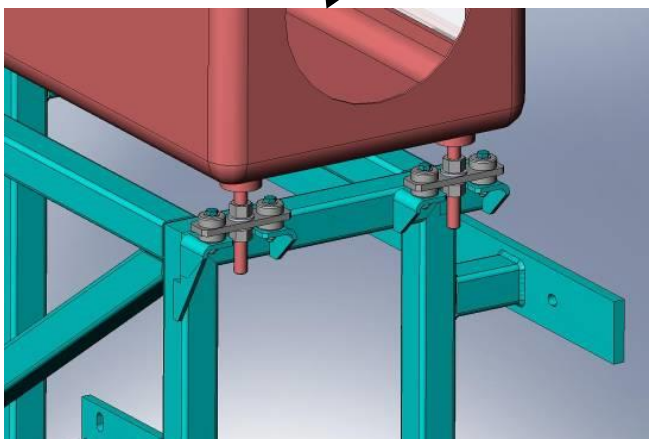
glove box
specially designed



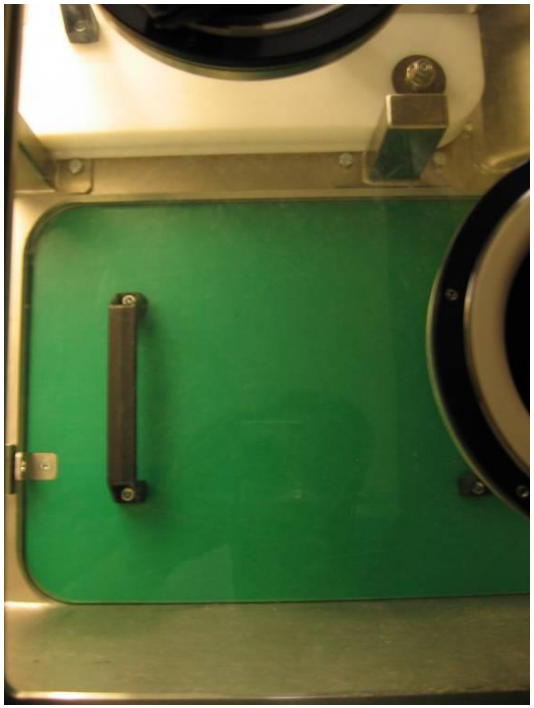
Vibrations reduction for imaging

Glove box on Silent blocks



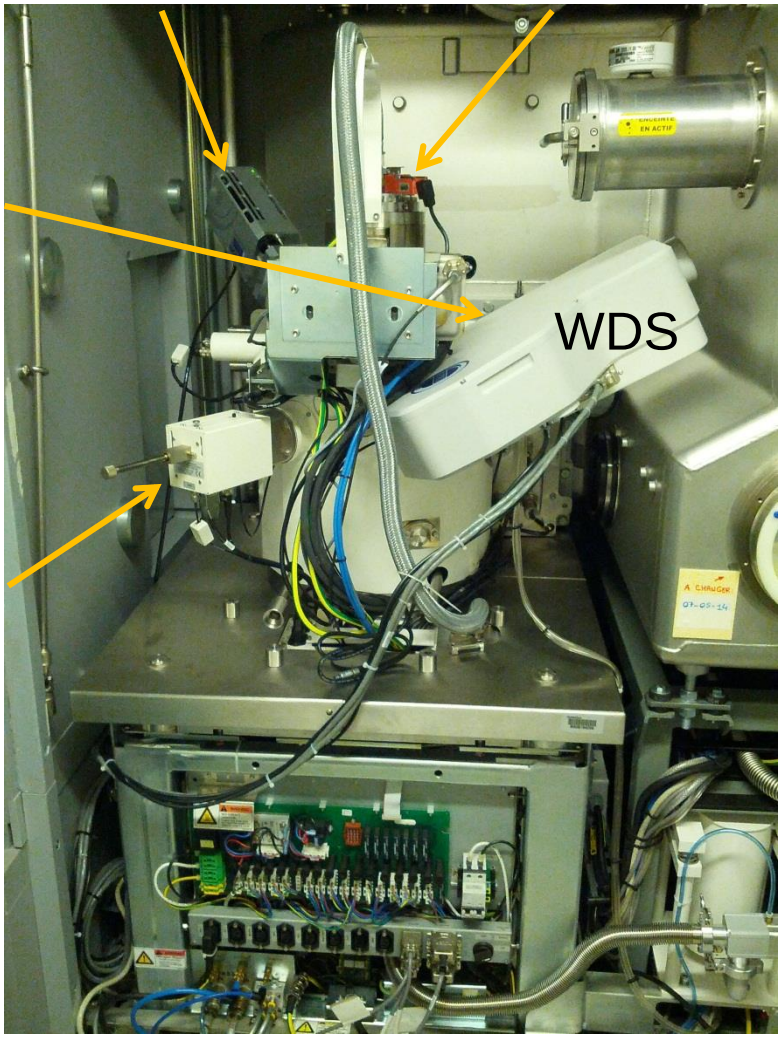


shutter



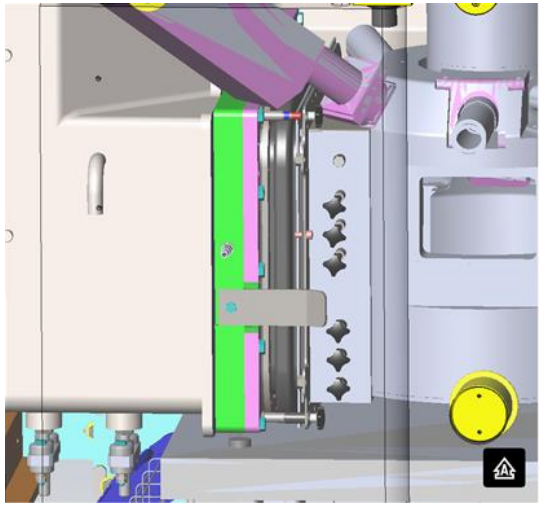
Pump distant

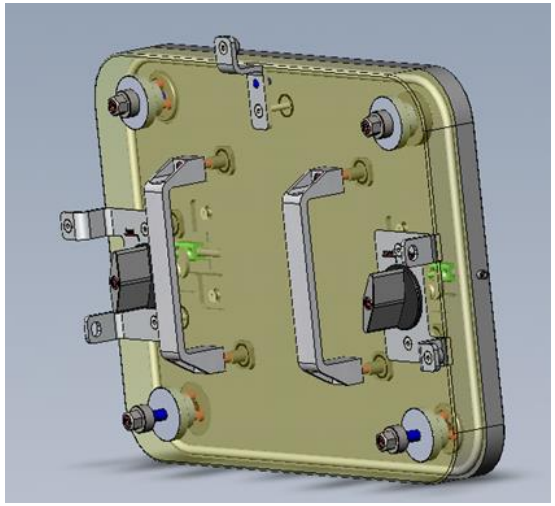
SEM-FEG ZEISS SUPRA 55 WDS



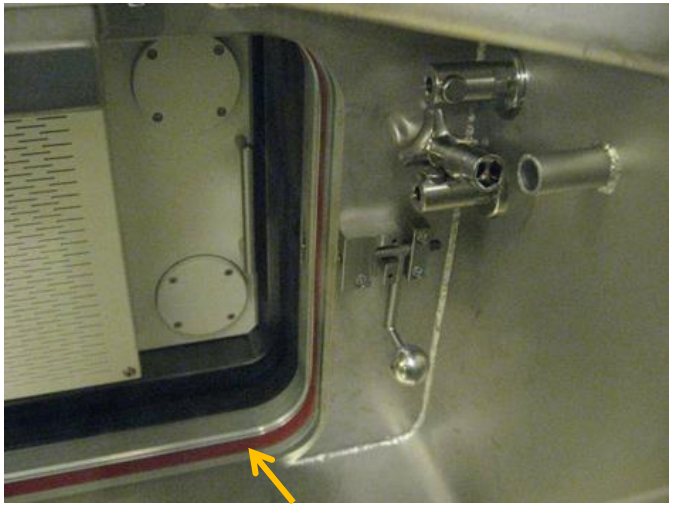
How to maintain it ?

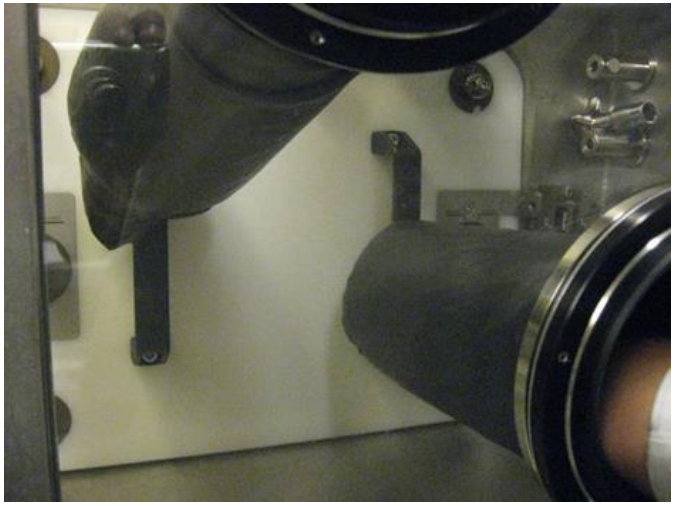
Maintenance





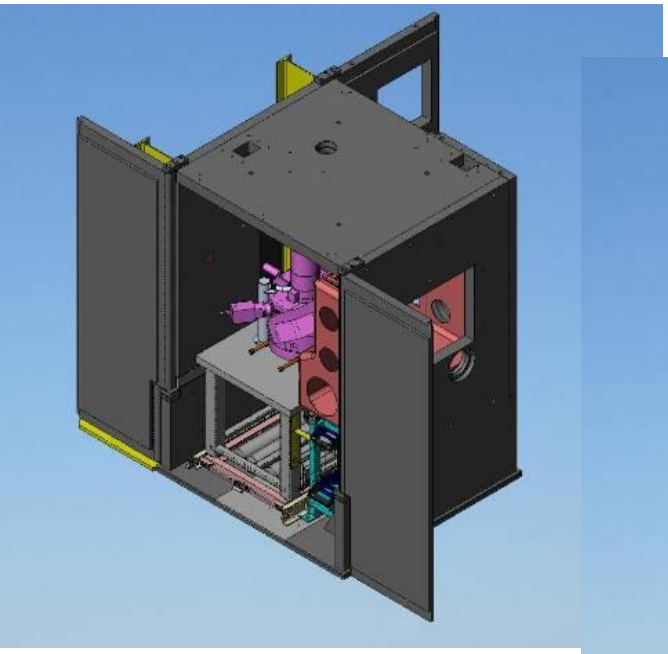
Disconnection without breaking containment

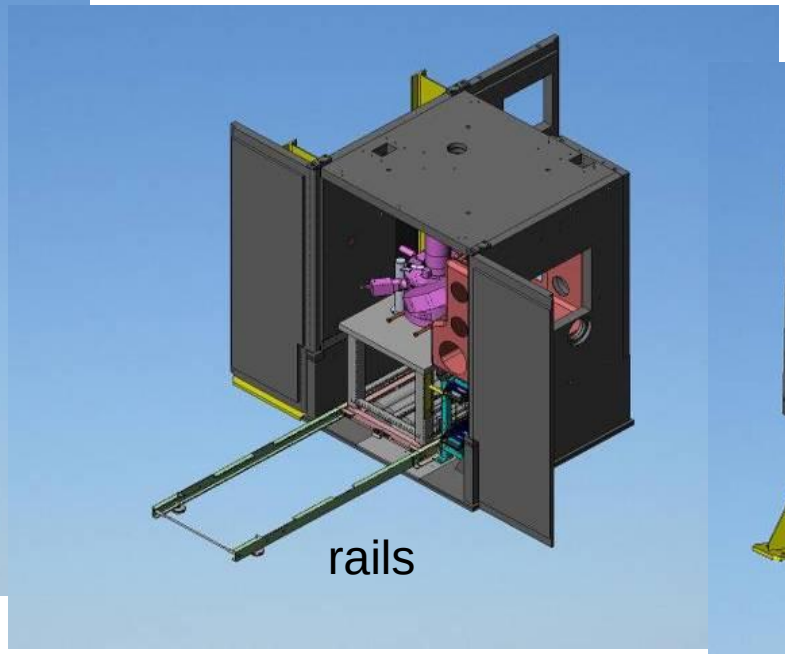


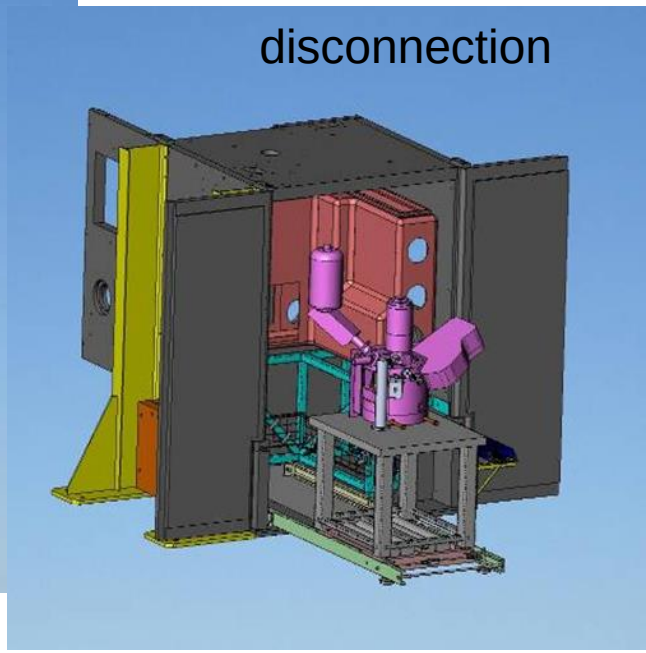


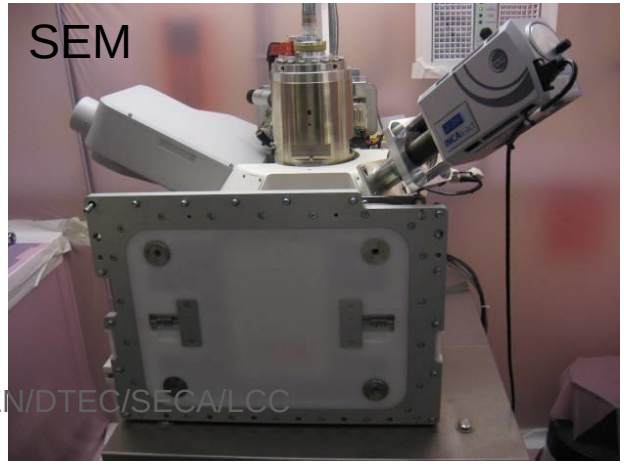
Inflatable joint

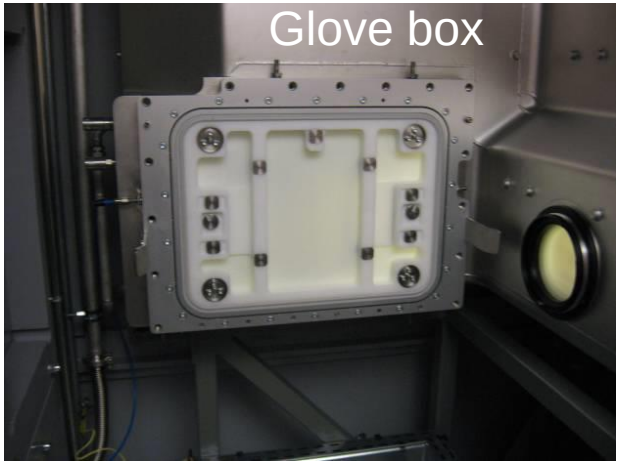
Maintenance





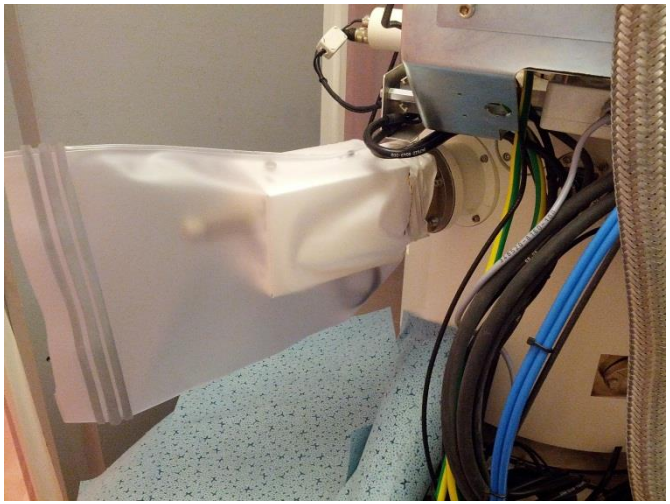






Examples

BSE scintillator replacement



Diaphragms replacement

Magnetic brake ajustement

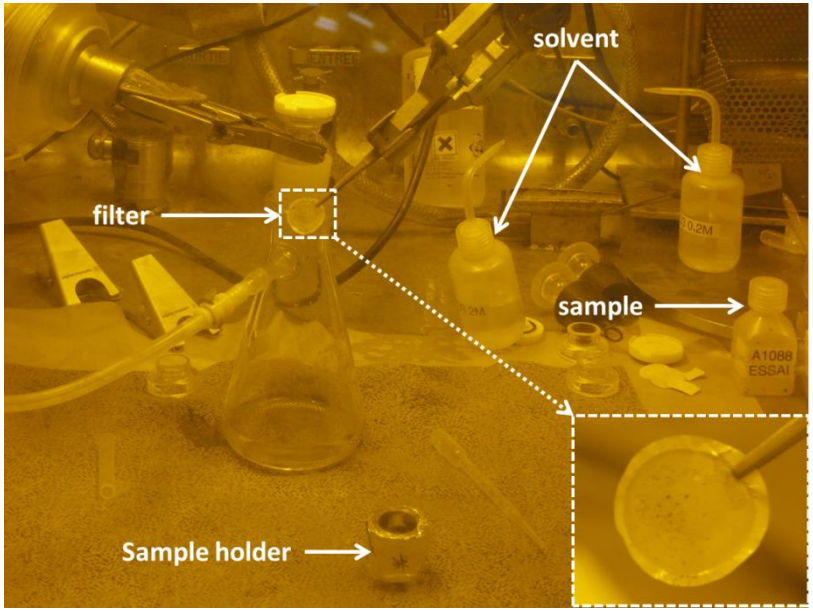
EDS chip replacement

Sample preparations
and
imaging examples

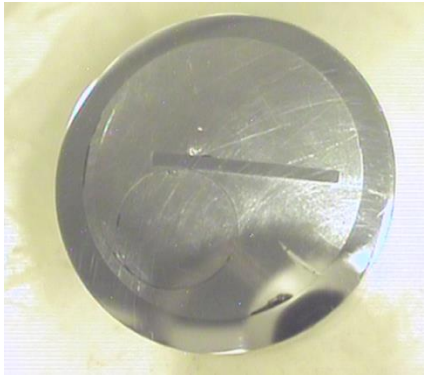
Preparations

Glove box

Hot cell



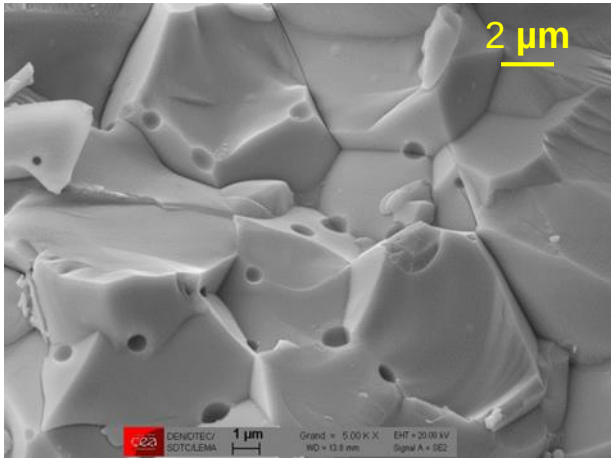
wood alloy



Examples

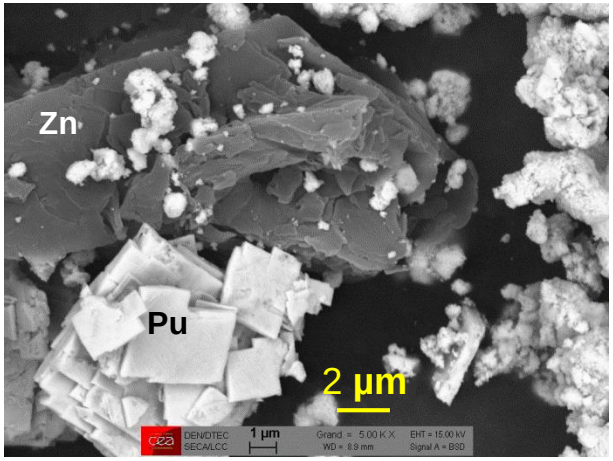
Everhart-Thornley

UO_2 pellet lixiviated with iron

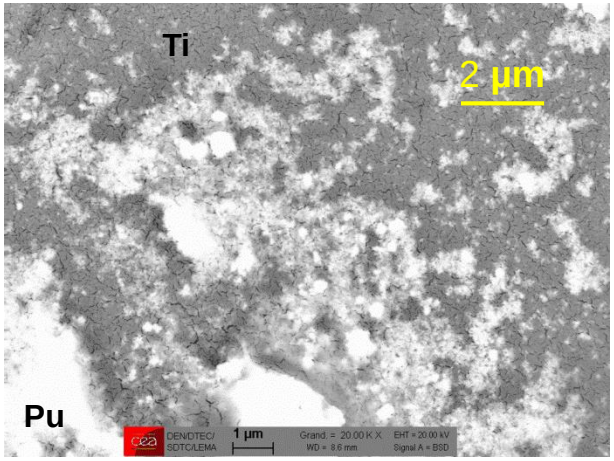


Sintered U-AmO_2 powder

BSD (chemical contrast)

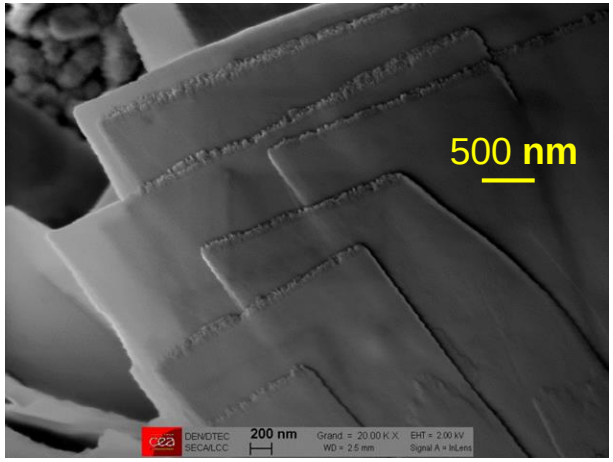


PuO_2/St-Zn mixing

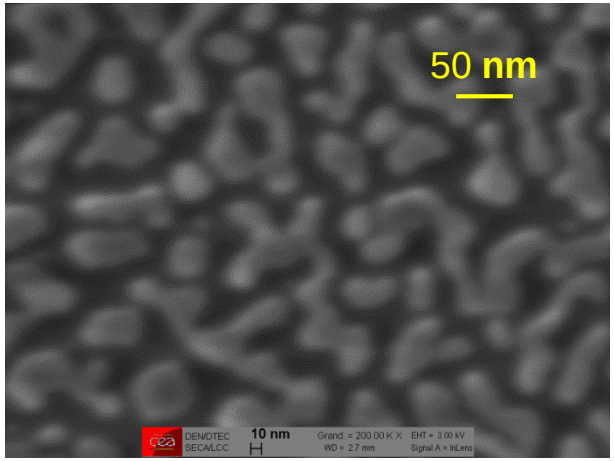


Dissolution of PuO_2 assisted by US

InLens (high resolution)



UO_2 pellet lixiviated with iron

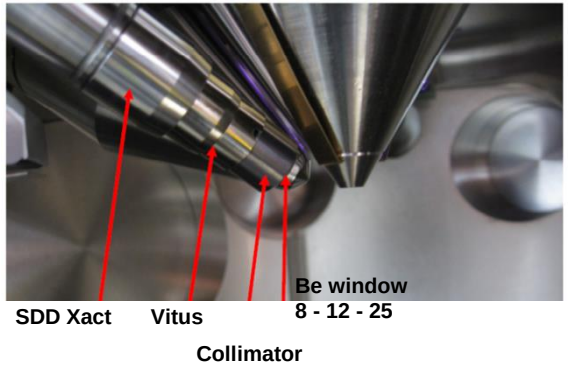


Plutonium colloid

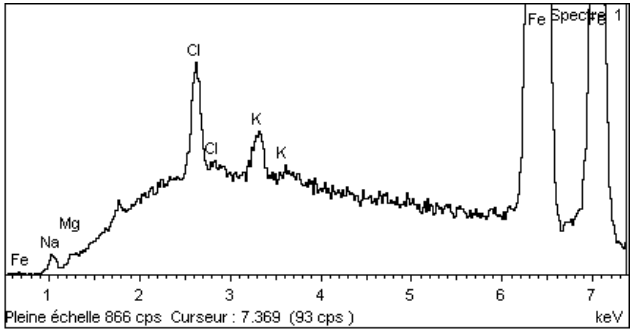
EDS measurements

Special EDS configuration

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



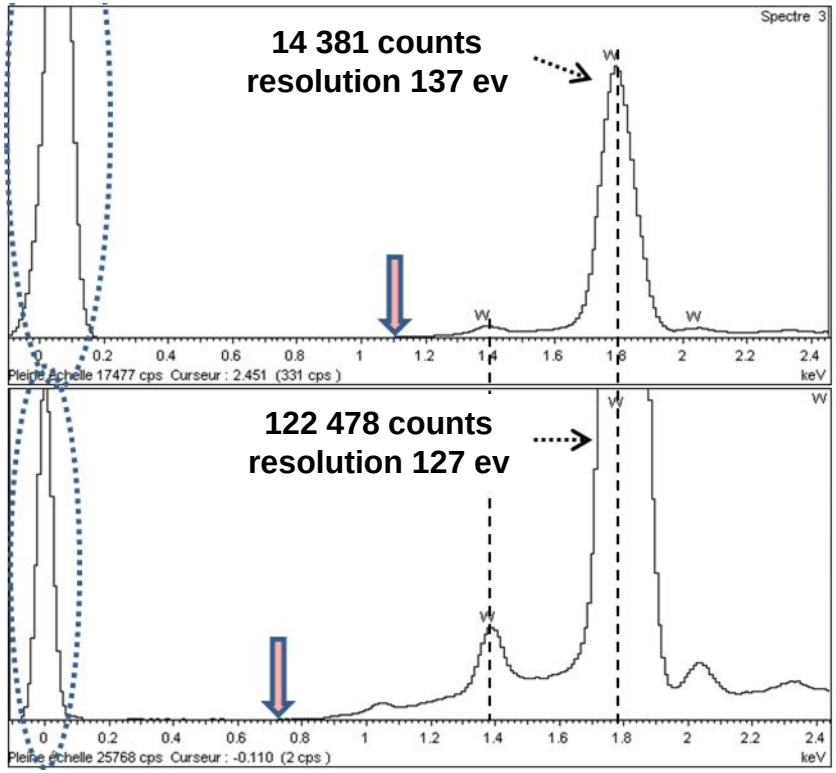
First element measurable : Na
+
Low counts measured at 15 kV minimum
with the largest diaphragm

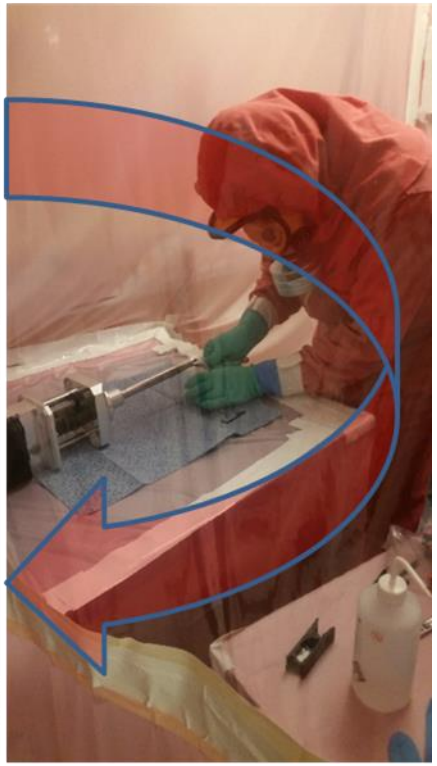


Bad spatial resolution
Long counting time
Destruction of many samples

Special EDS configuration

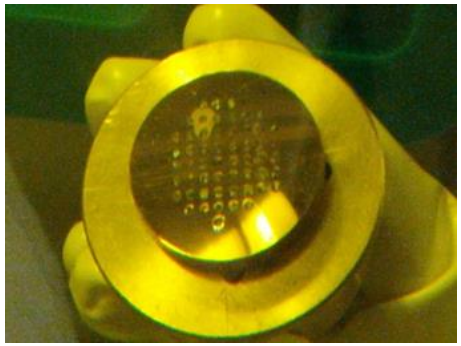
- 25 µm Be window
- high distance sample / detector
- 4 years old chip





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

Calibration is essential !



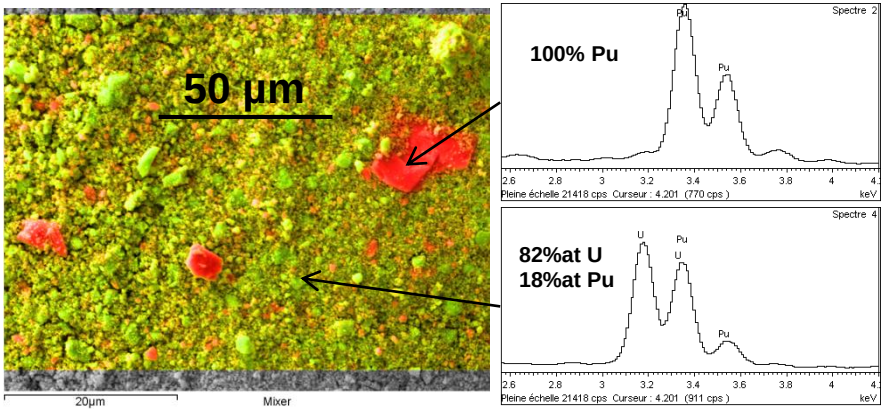
MAC55 Standard

Home made standard for actinides

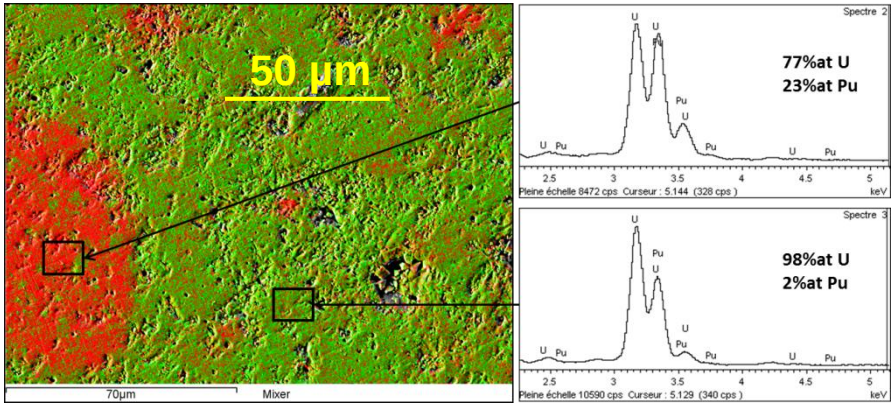
EDS analysys examples

Plutonium / Uranium mapping (red/green)

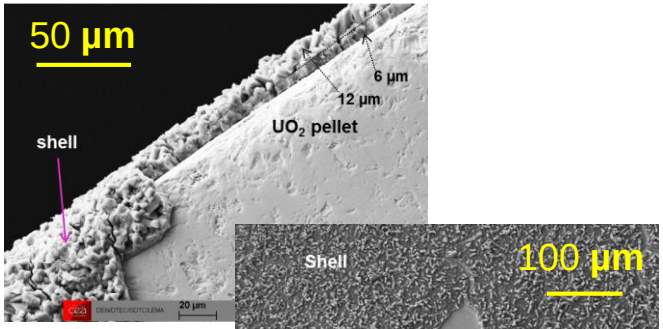
UO2 / PuO2 powders mixed

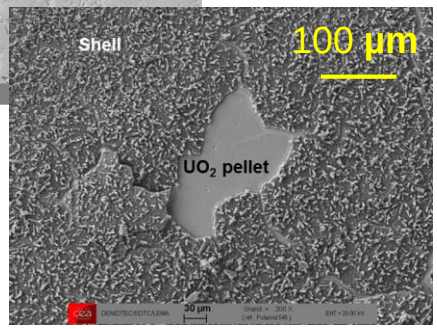


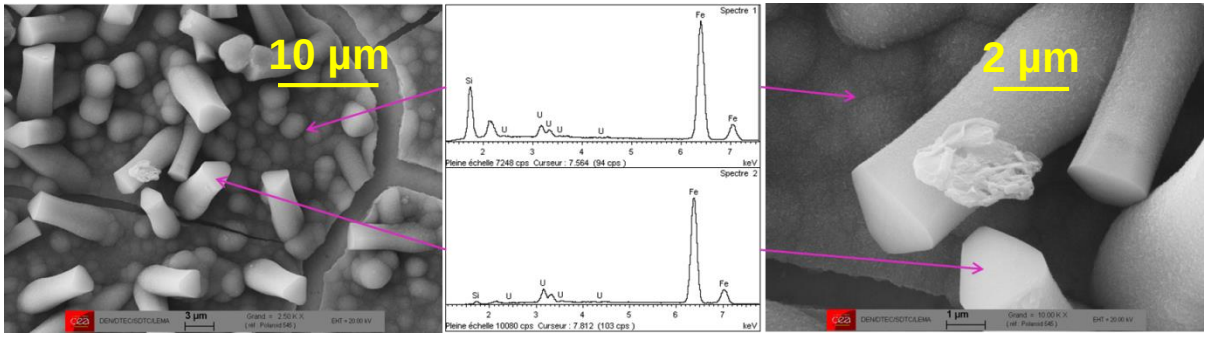
MOX sintered pellet



Spectrum on UO_2 pellet lixiviated in water for 1 year in presence of an iron foil

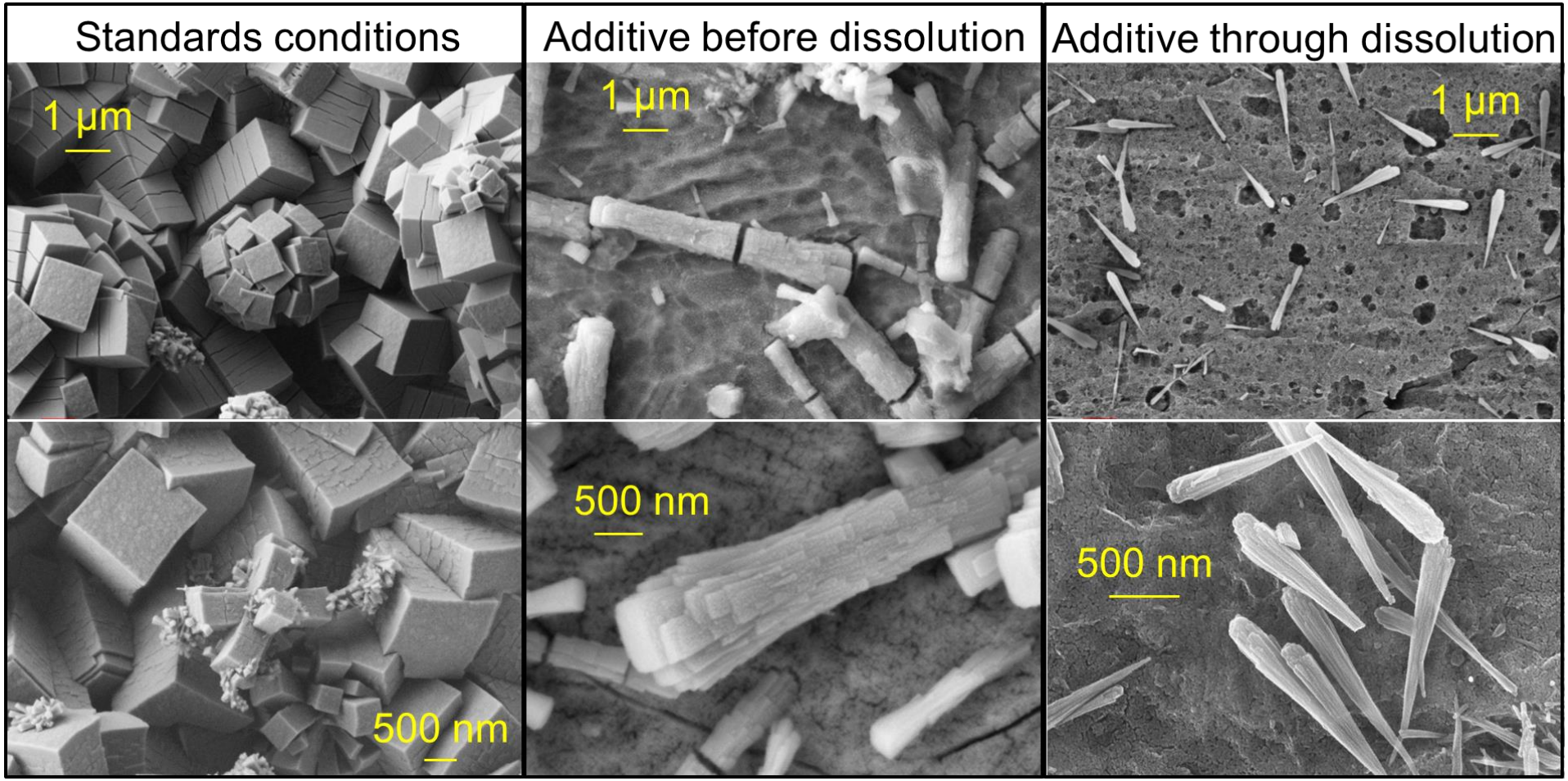




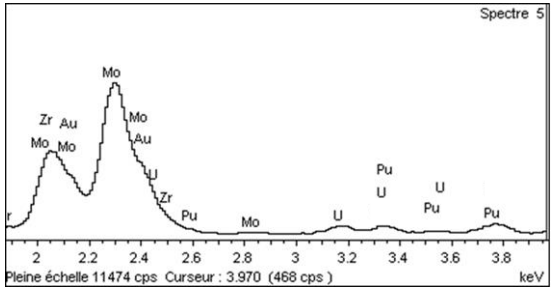


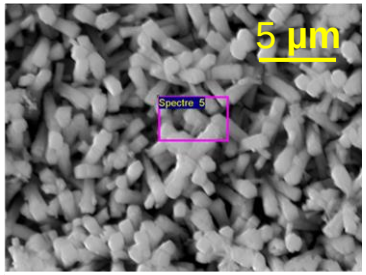
Images and EDS on Mo/Zr precipitates (raw)

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

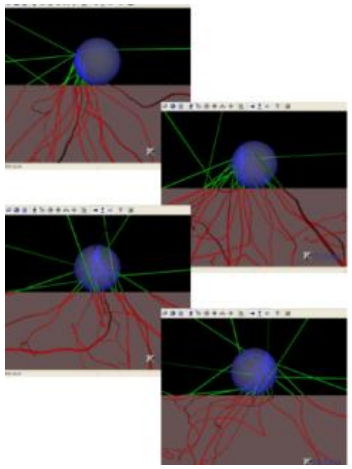


Half-quantitative results on rough surfaces ?





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



55A	Zr *	Mo*	M *	U*	Pu*	
average	29.1	57.6	6.2	6.2	0.9	32 spectrum
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	18 spectrum
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	35 spectrum
SD	7.14	7.07	0.91	0.49	0.32	
RSD	17%	14%	22%	21%	30%	

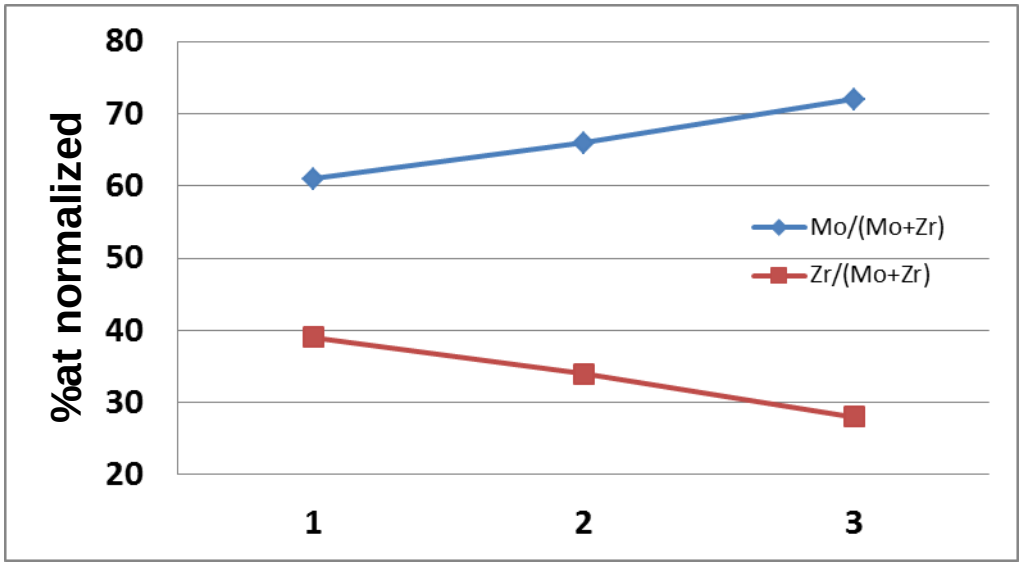
EDS results are average on numerous spectrum
Number of spectrum depends of the SD and RSD

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 ?

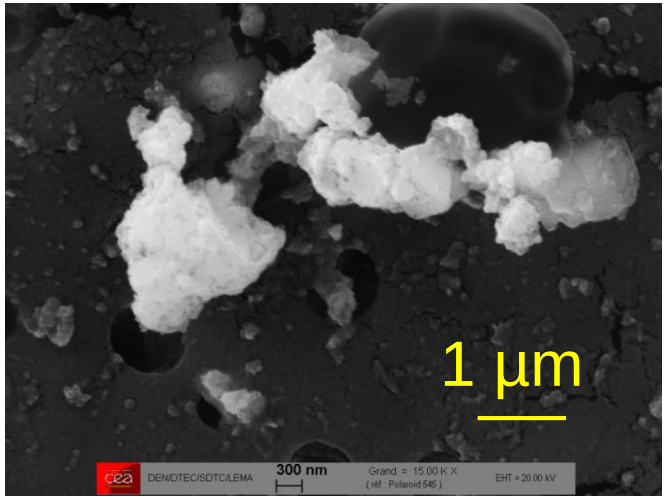
close ionique radius

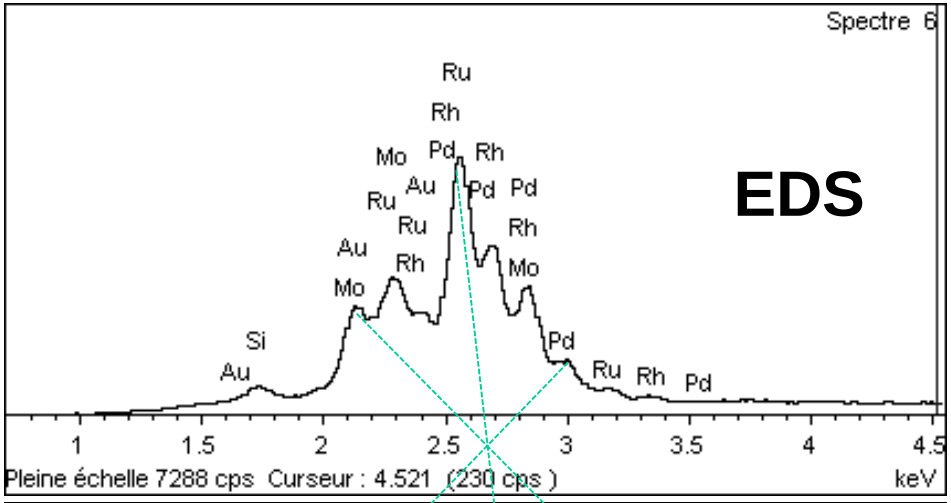
Octahedral coordination for both

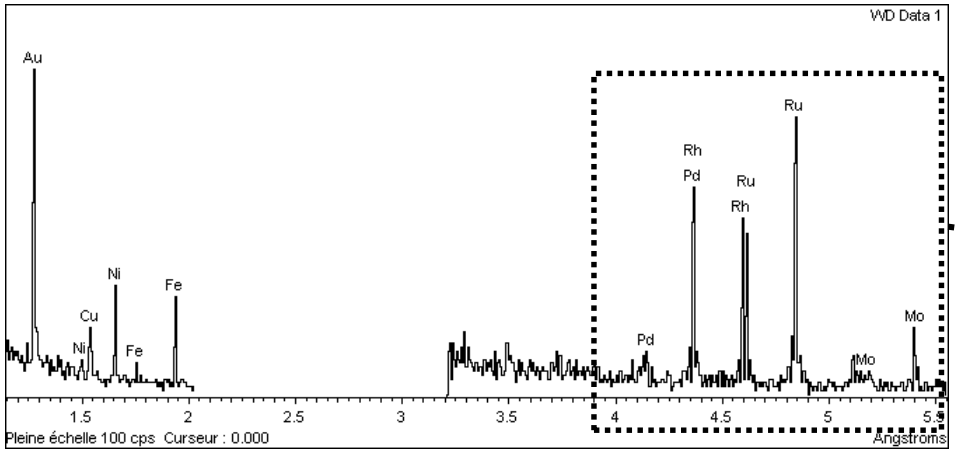
- XRD mesurements to confirm

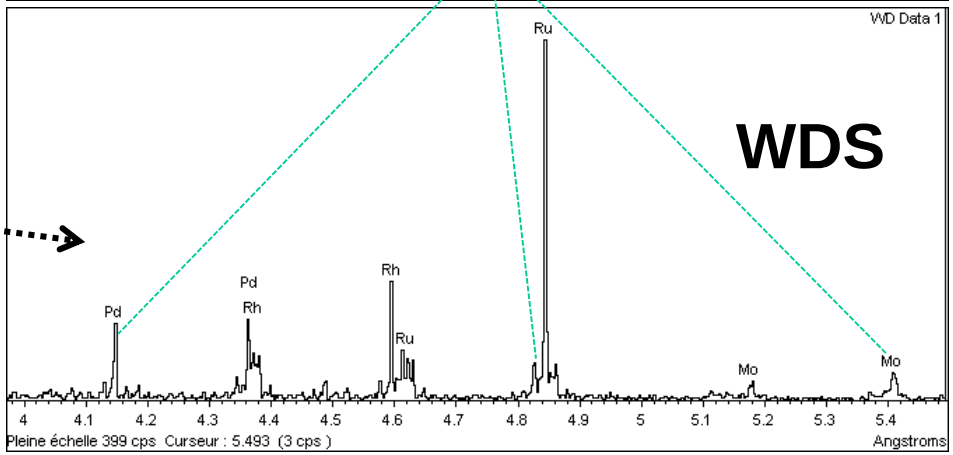
WDX measurements

Eamples on dissolution residues of irradiated fuel

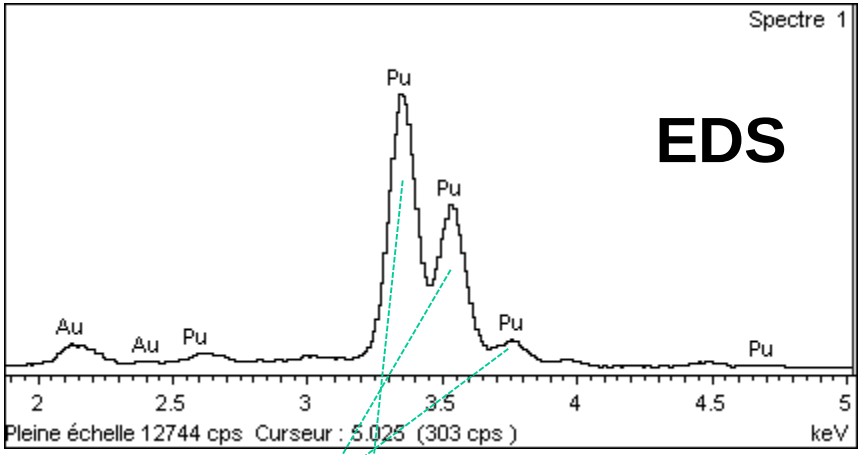


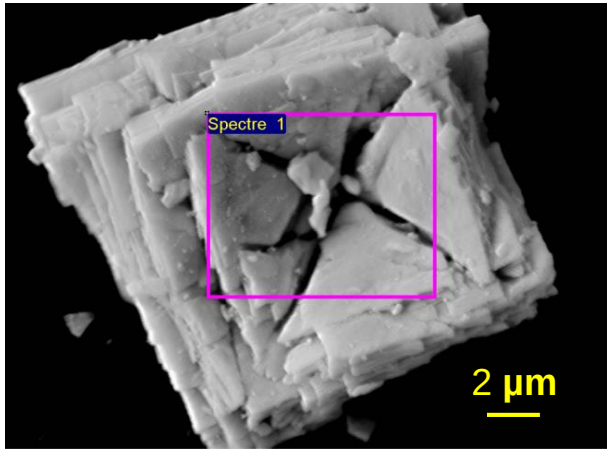


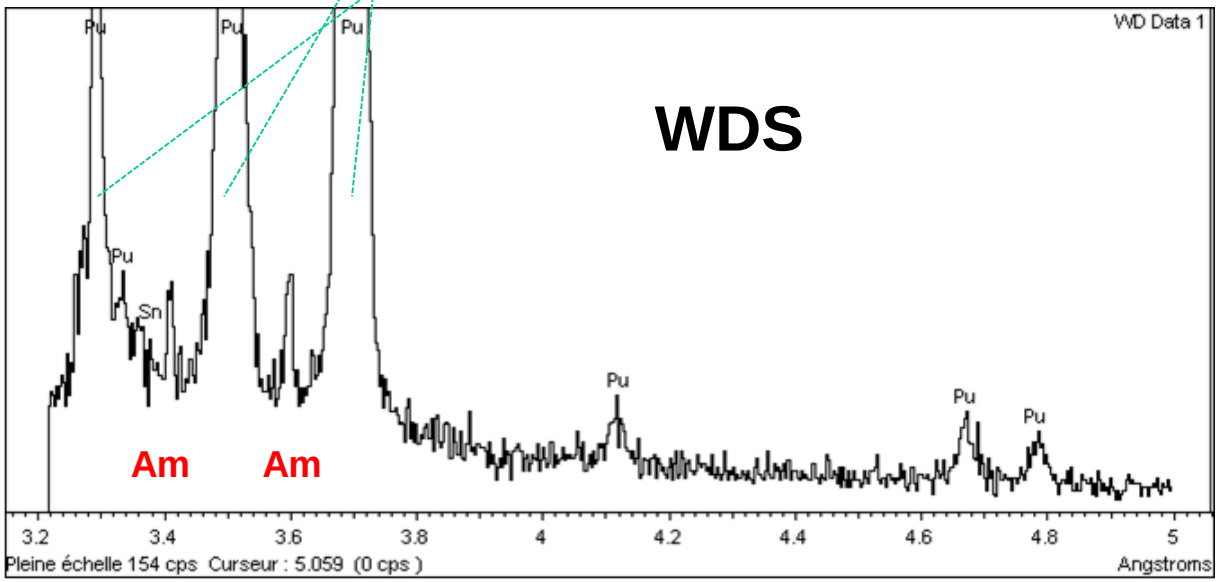




Americium detection in PuO_2 powders

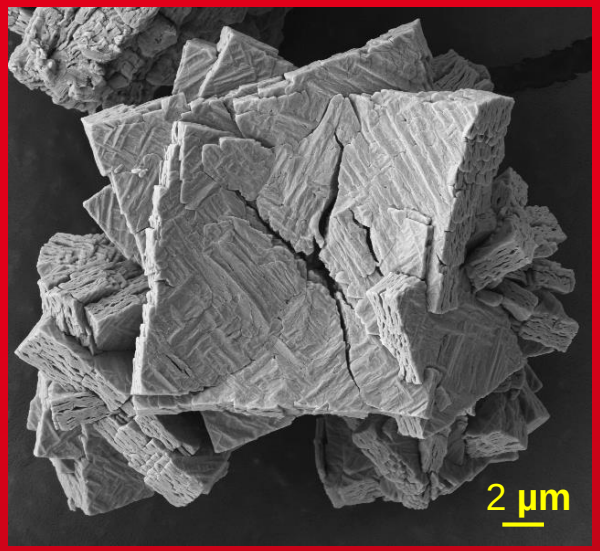






DEN/DTEC/SECA/LCC

Pu oxalate



Thank you for your attention

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