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SEM analyzes of powdered actinide compounds Implementation in a hot laboratory

G. Jouan

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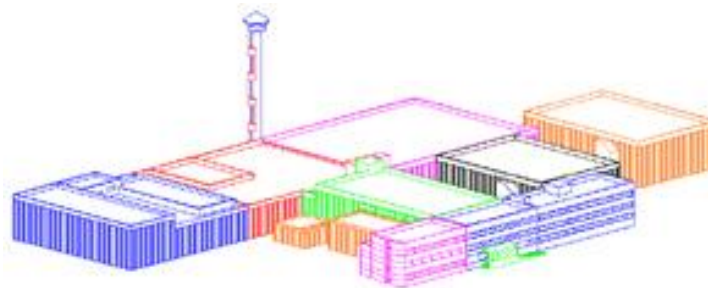
SEM analyzes of powdered actinide compounds: Implementation in a hot laboratory

G. JOUAN

DEN/MAR/DTEC/SECA/LCC

Laboratoire de Caractérisation du Combustible

Marcoule – Atalante - INB 148

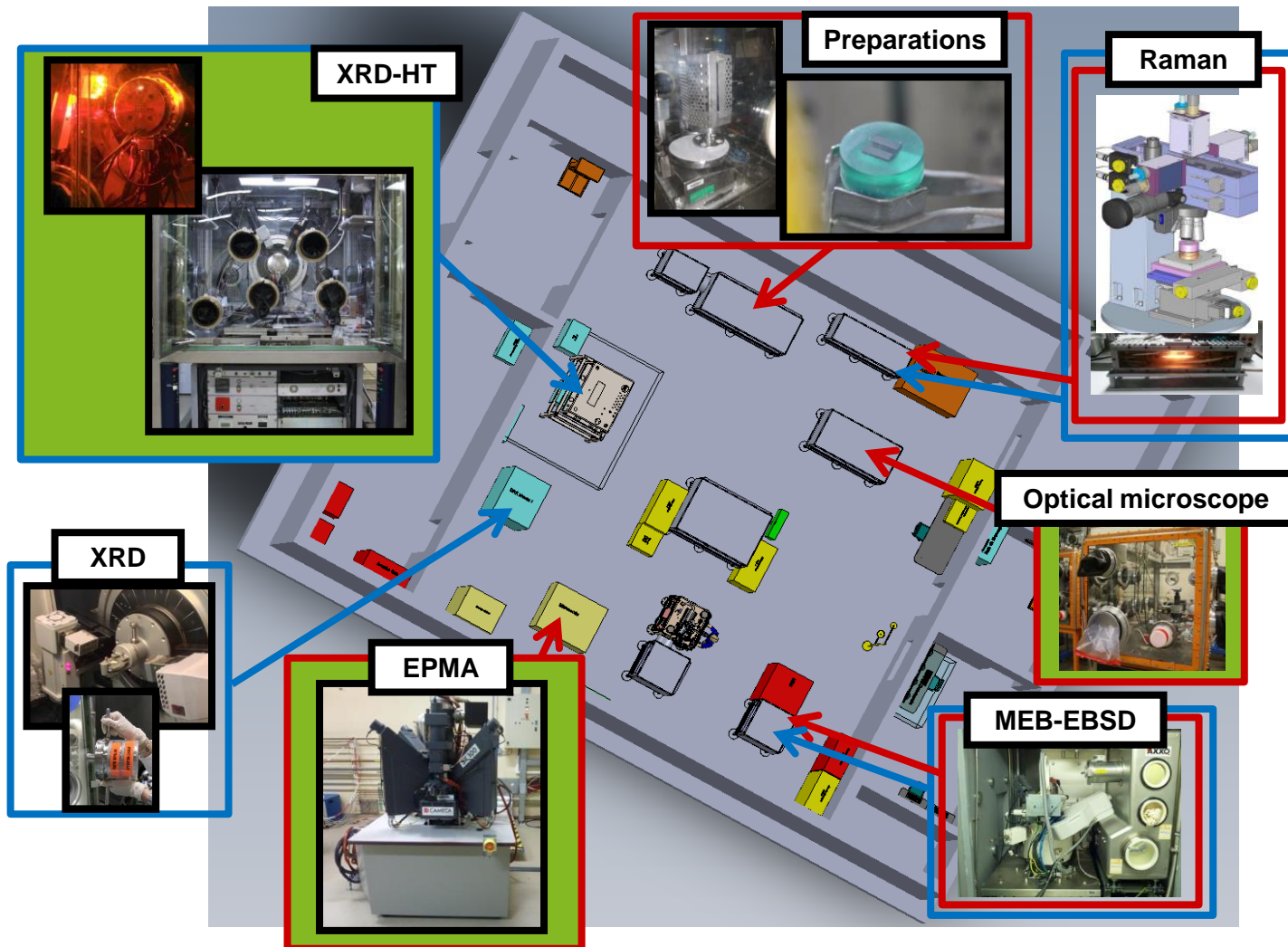


19 000 m² on 3 levels
12 shielded lines
17 laboratories (250 glove boxes)

LCC, laboratory L26 (end of 2017)

L26 \approx 130 m²

12 agents



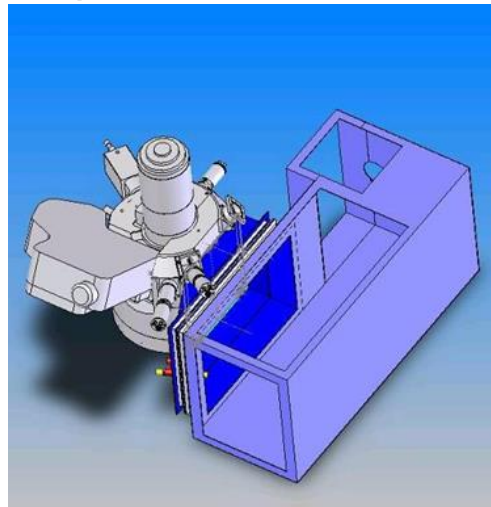
LES216 SEM-FEG for radioactives powders since 2010

SEM-FEG ZEISS SUPRA 55 WDS

* patent WO2013/068321 A1



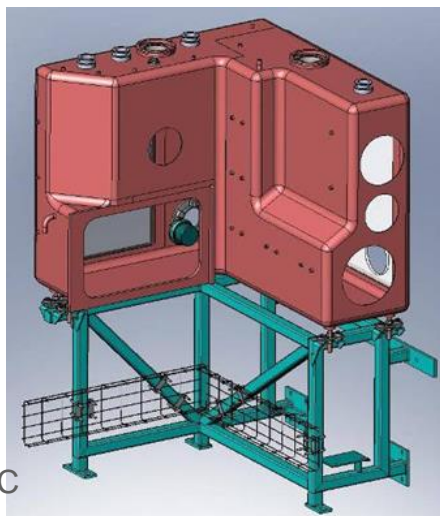
+



+



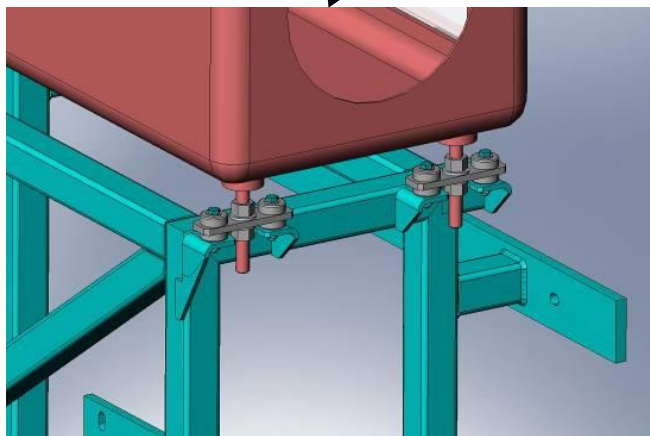
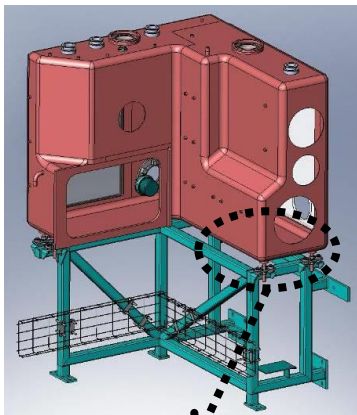
glove box
specially designed



DEN/DTEC/SECA/LCC

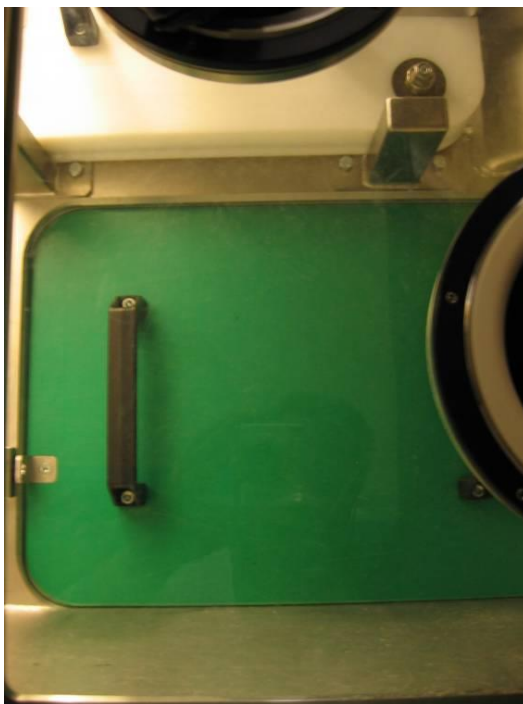
Hot cell already in the lab
(20 mCi)

Glove box on Silent blocks

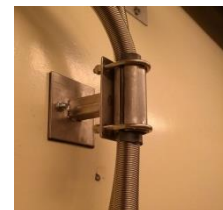


DEN/DTEC/SECA/LCC

shutter



Pump distant



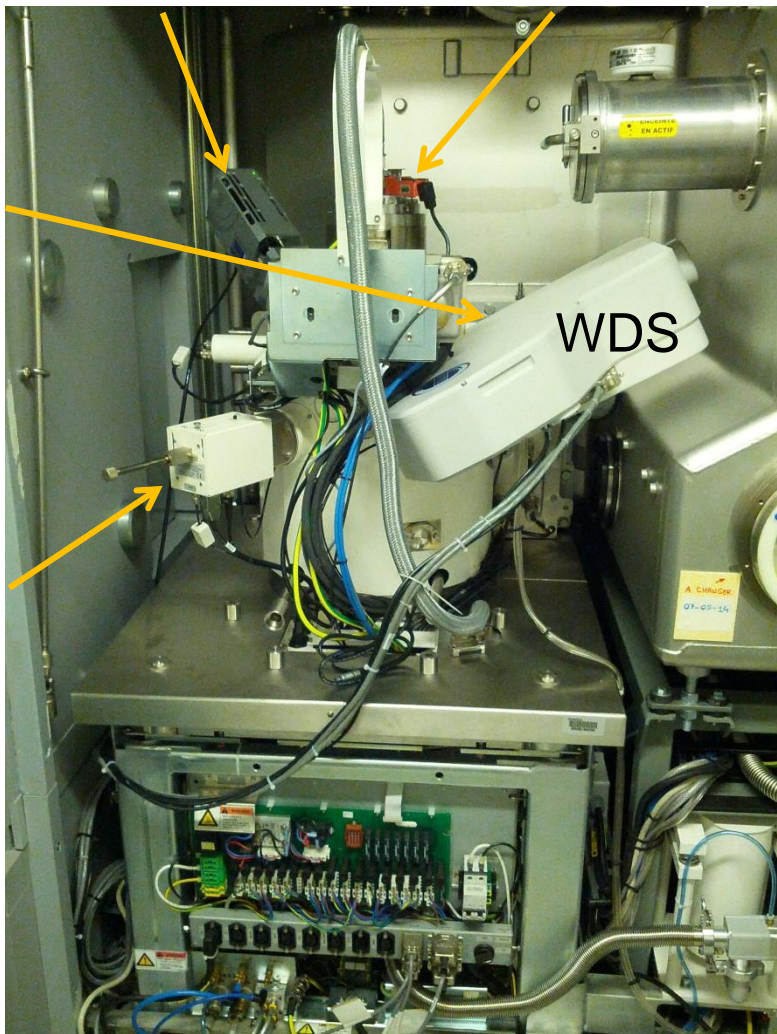
SEM-FEG ZEISS SUPRA 55 WDS

Everhart-Thornley
BSE

EDS

INLENS

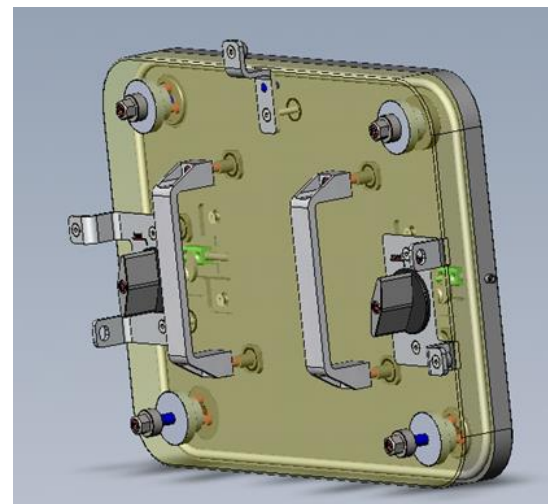
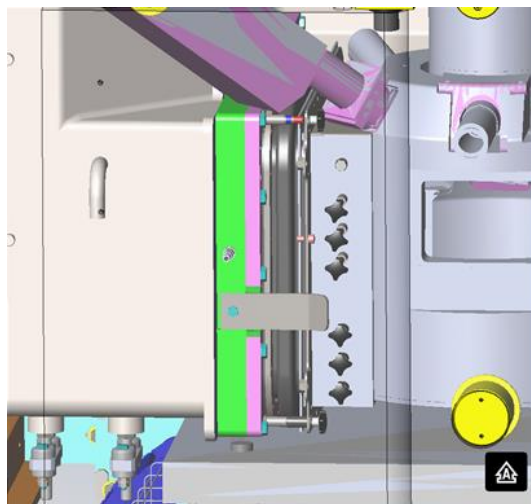
WDS



DEN/DTEC/SECA/LCC



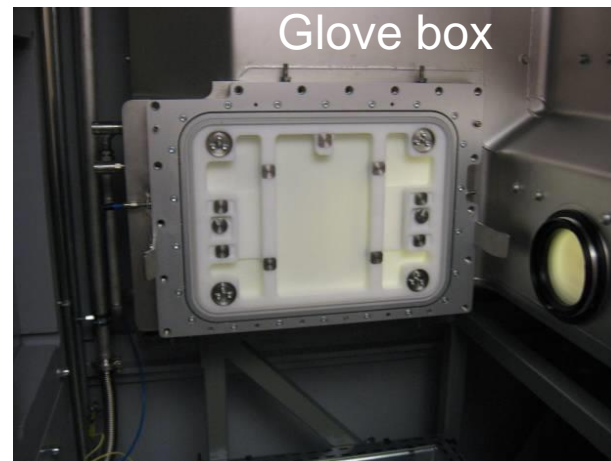
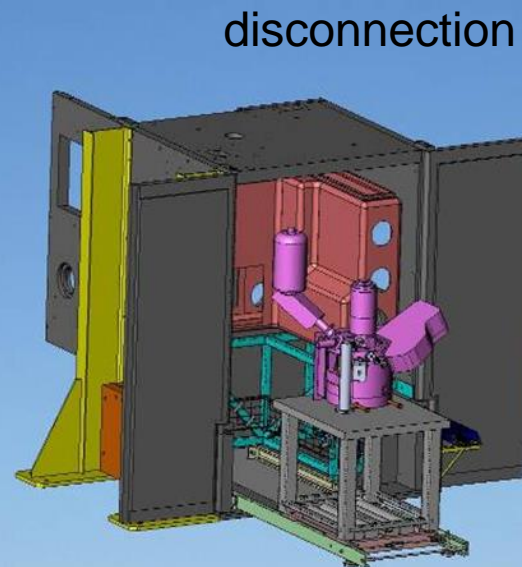
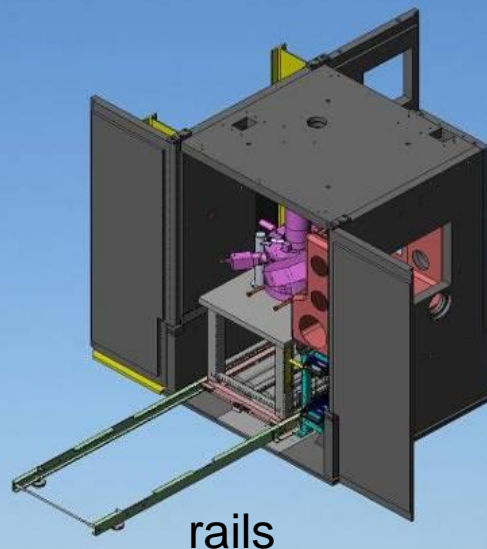
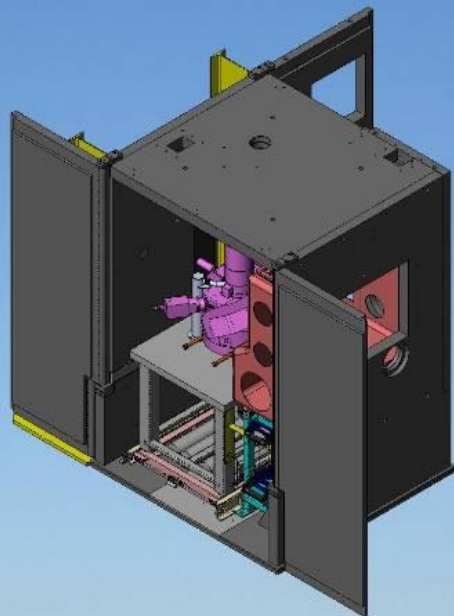
How to maintain it ?



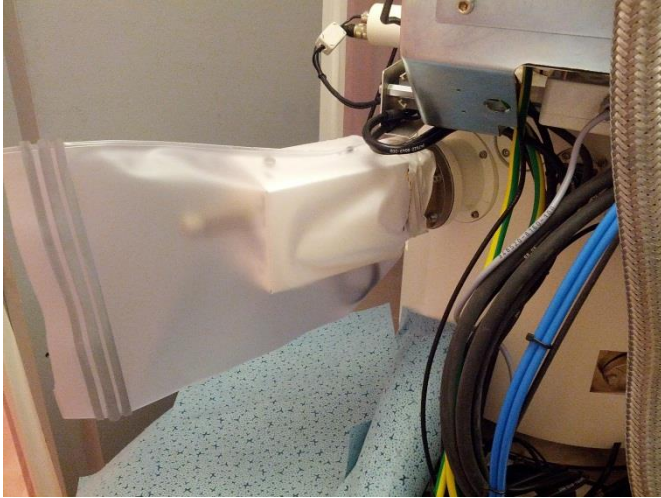
Disconnection without breaking containment



Inflatable joint



BSE scintillator replacement



Magnetic brake ajustement



EDS chip replacement



Diaphragms replacement

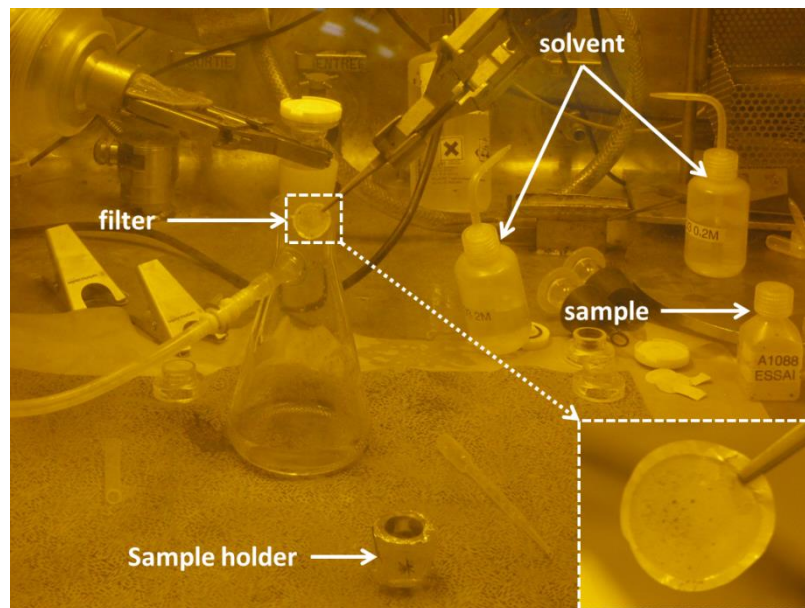


Sample preparations and imaging examples

Glove box



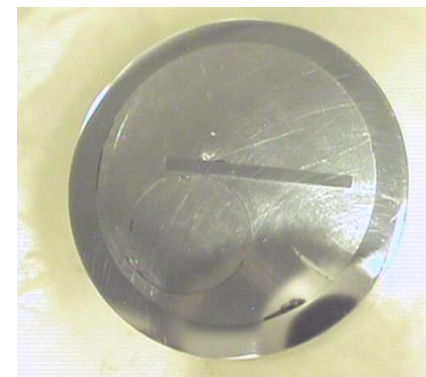
Hot cell



DEN/DTEC/SECA/LCC



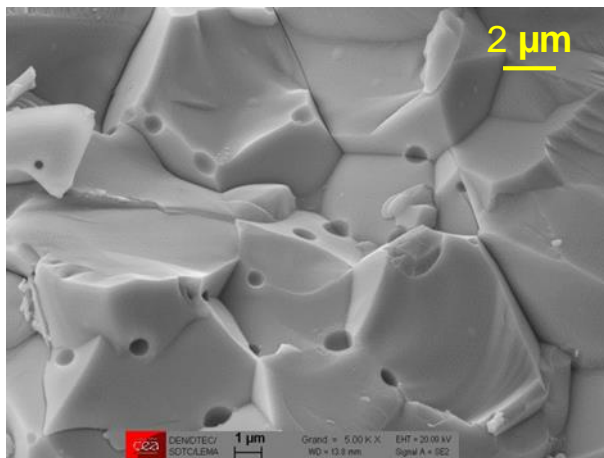
wood alloy



Everhart-Thornley

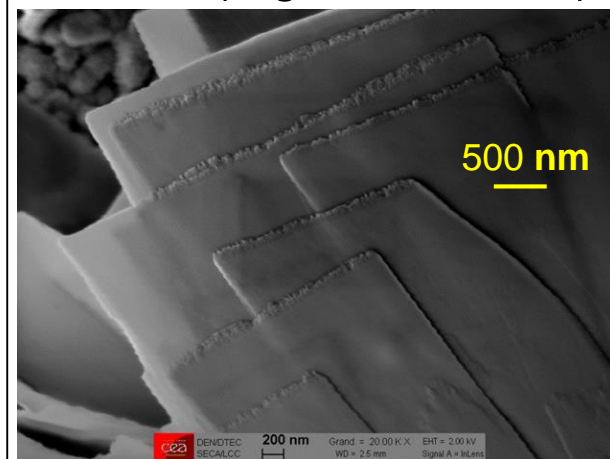


UO₂ pellet lixiviated with iron



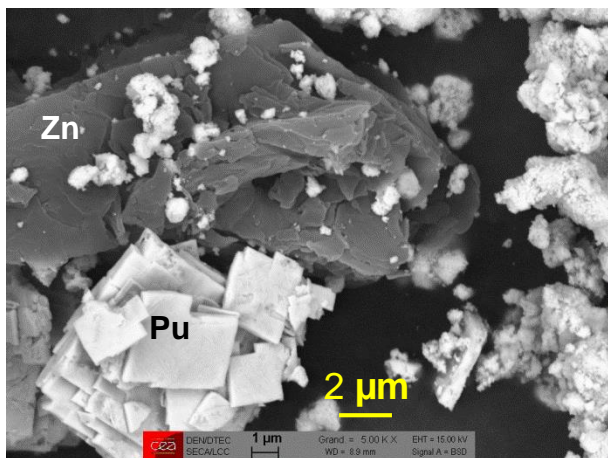
Sintered U-AmO₂ powder

InLens (high resolution)

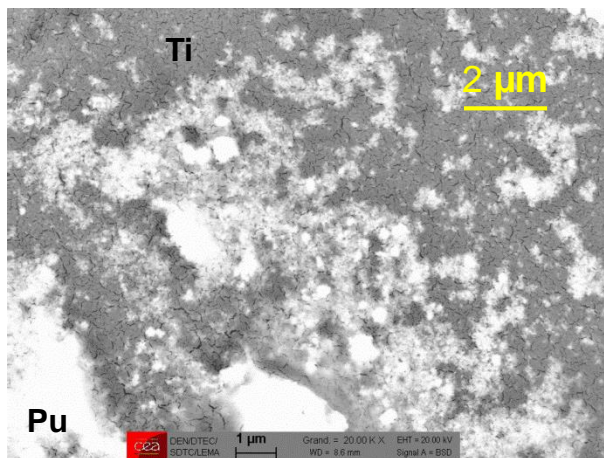


UO₂ pellet lixiviated with iron

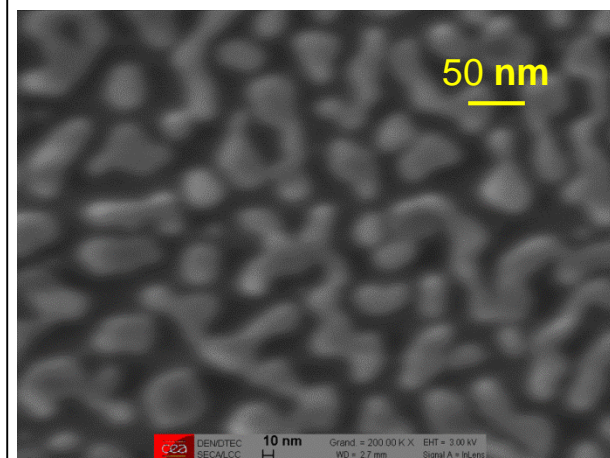
BSD (chemical contrast)



PuO₂/St-Zn mixing



Dissolution of PuO₂ assisted by US



Plutonium colloid

EDS measurements



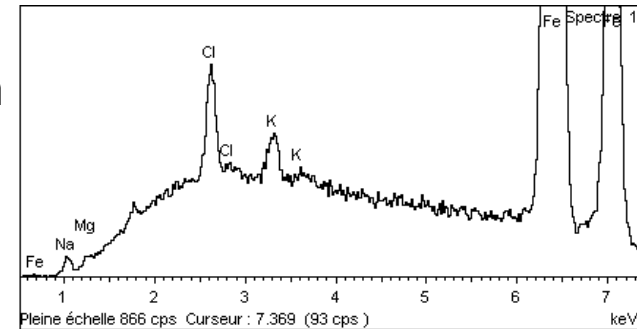
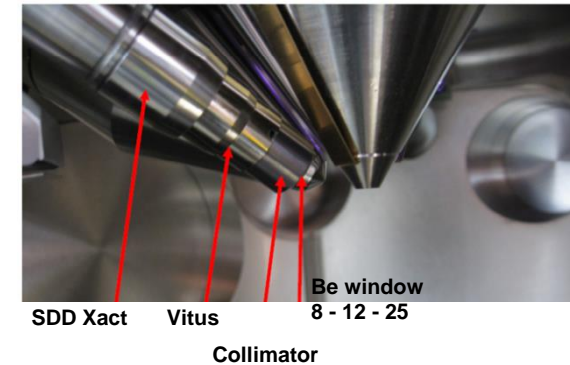
Be window used as a shield
 +
 EDS/sample distance increased
 +
 0.5 mm Collimator of (standard at 2 mm)



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



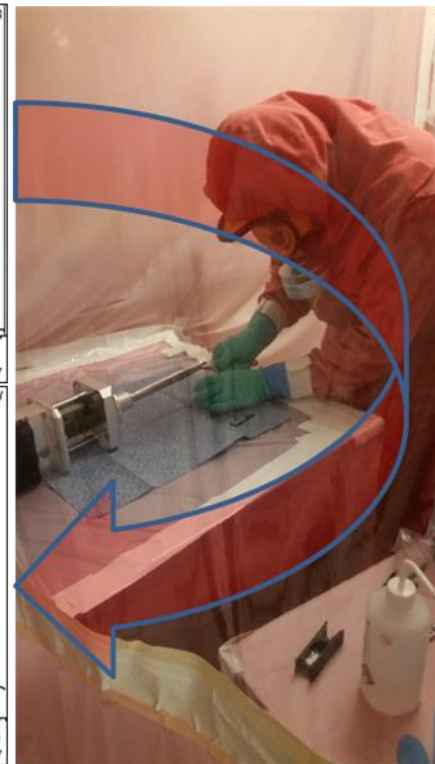
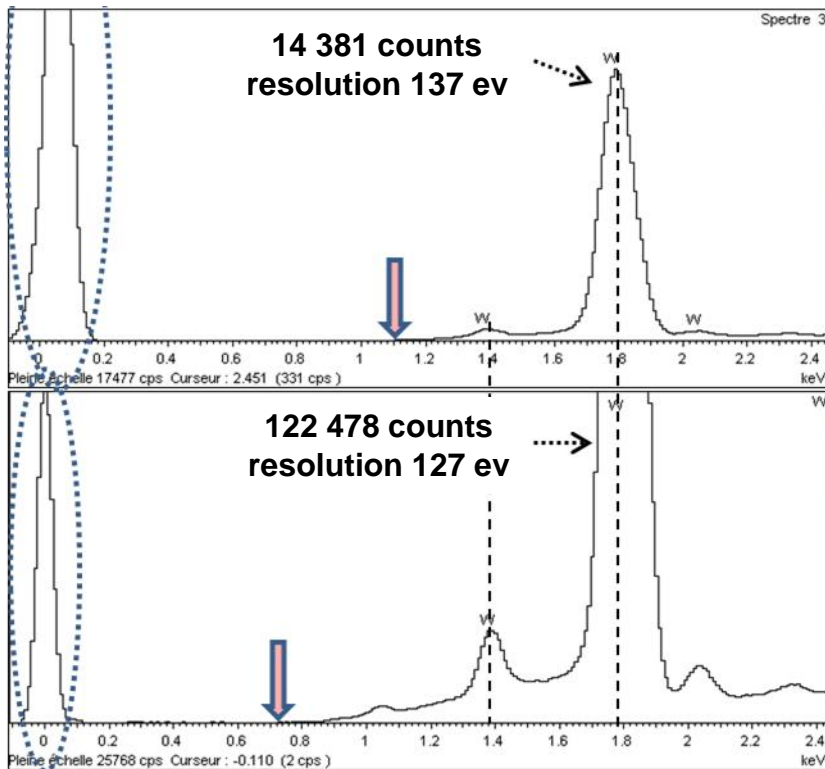
cea Special EDS configuration

- 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 !

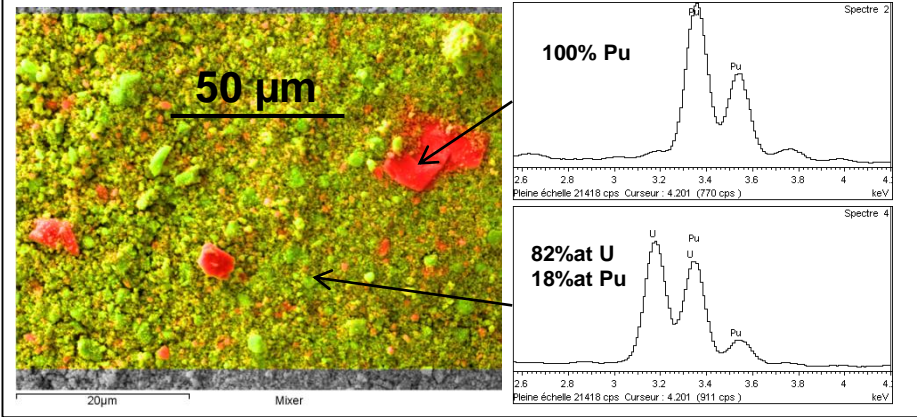


MAC55 Standard

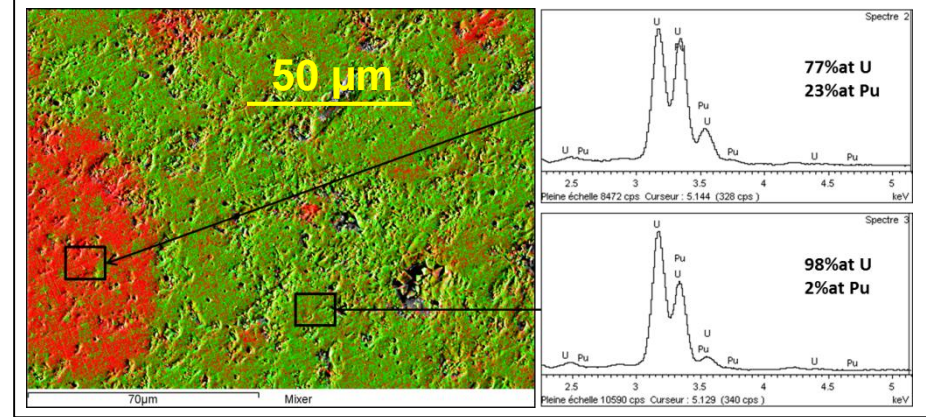
Home made standard for actinides

Plutonium / Uranium mapping (red/green)

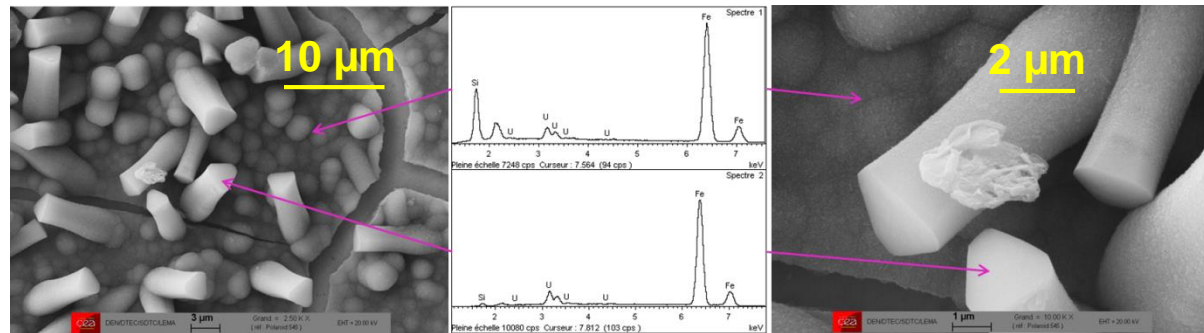
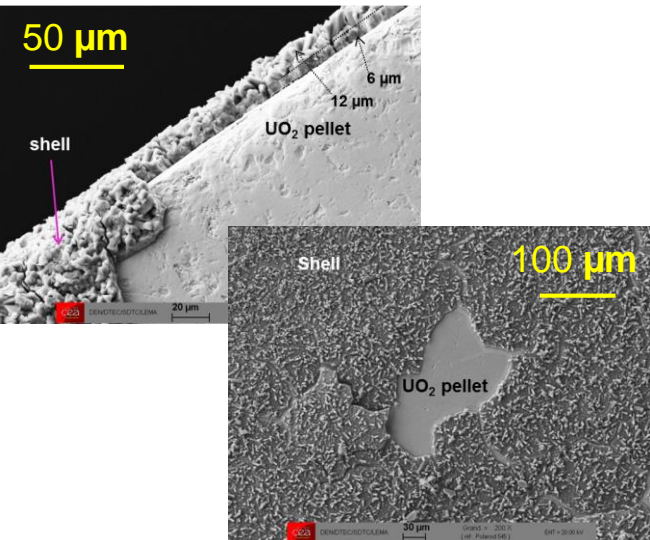
UO₂ / PuO₂ powders mixed



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



Additive before dissolution

1 μm



Additive through dissolution

1 μm



500 nm



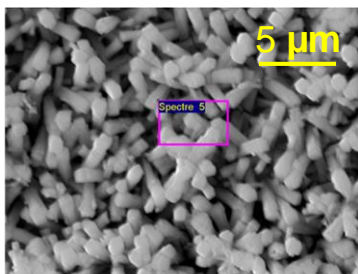
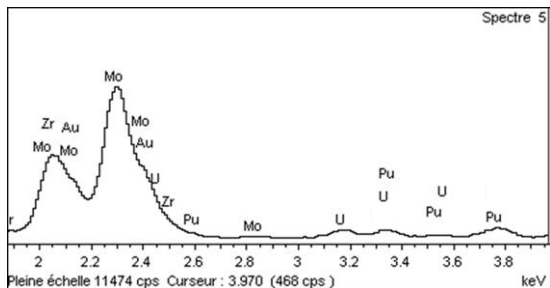
500 nm



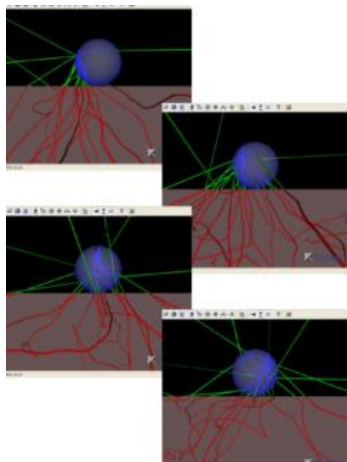
500 nm



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



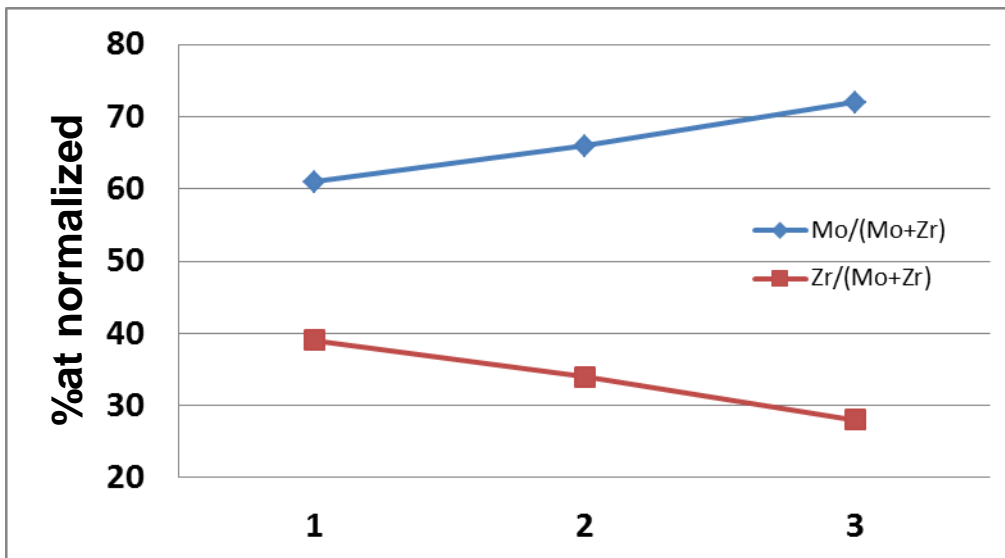
EDS results are average on numerous spectrum
 Number of spectrum depends of the SD and RSD

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 ?

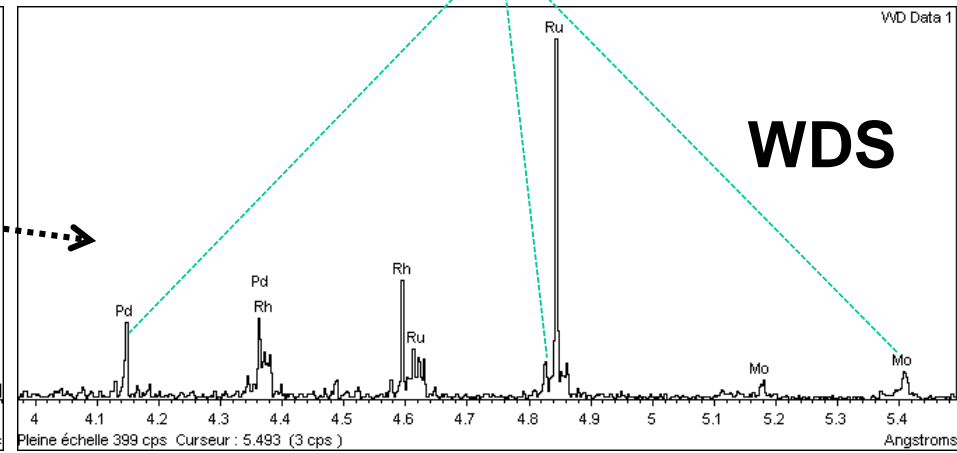
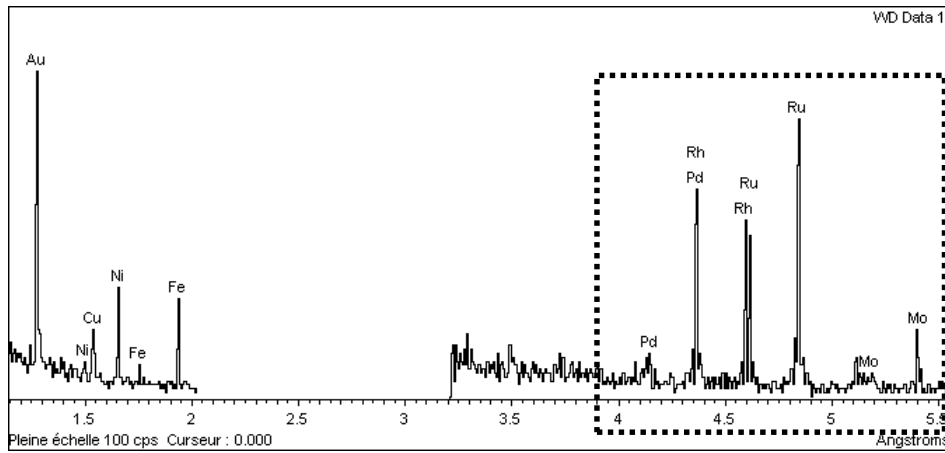
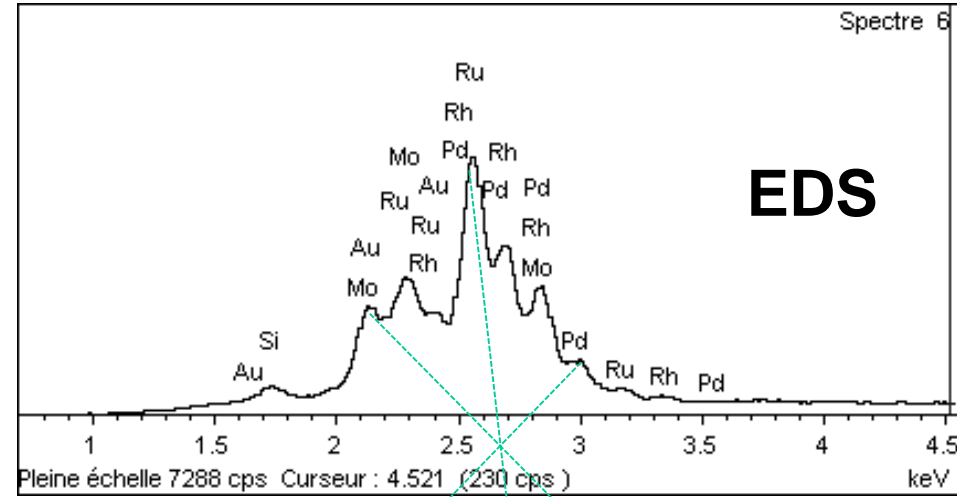
close ionique radius

Octahedral coordination for both

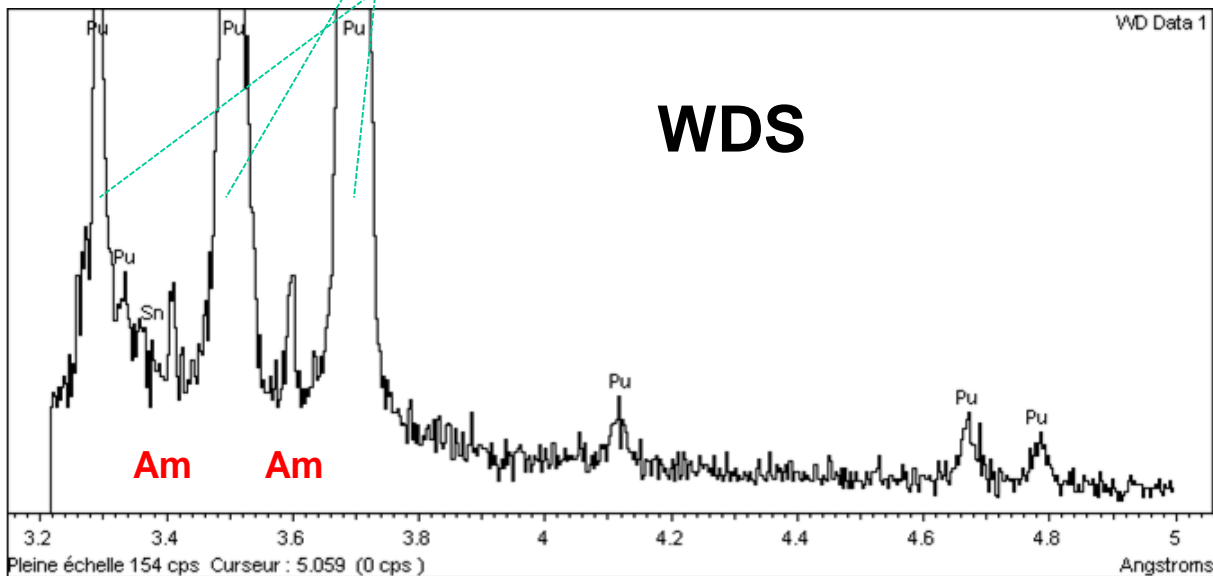
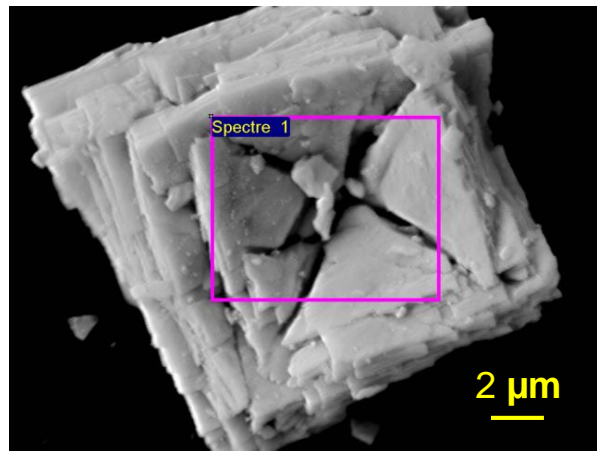
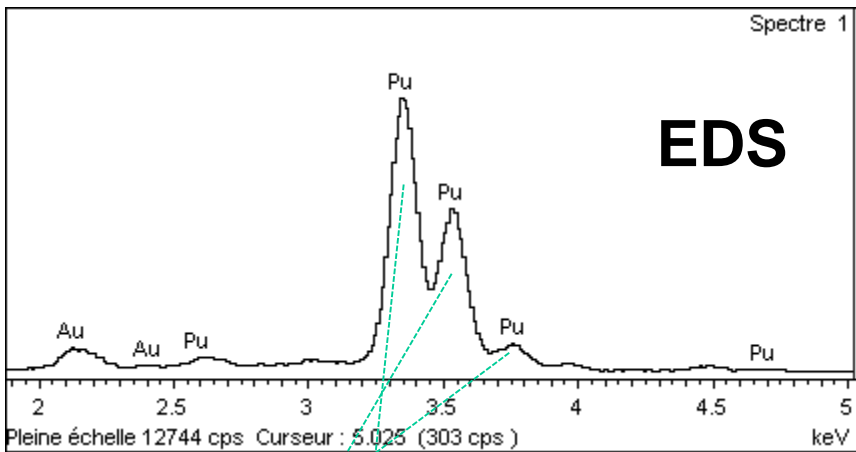
- XRD measurements to confirm

WDX measurements

Exemples on dissolution residues of irradiated fuel

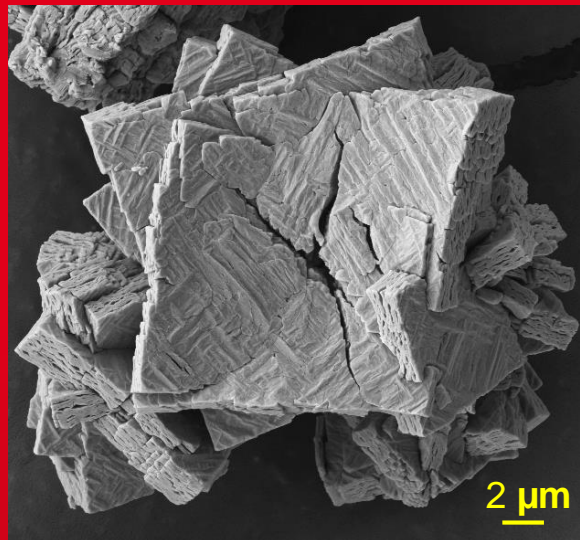


Americium detection in PuO₂ powders





Pu oxalate



*Thank you
for your attention*

Commissariat à l'énergie atomique et aux énergies alternatives
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