Preparation and characterization of actinide targets at JRC Geel, Belgium

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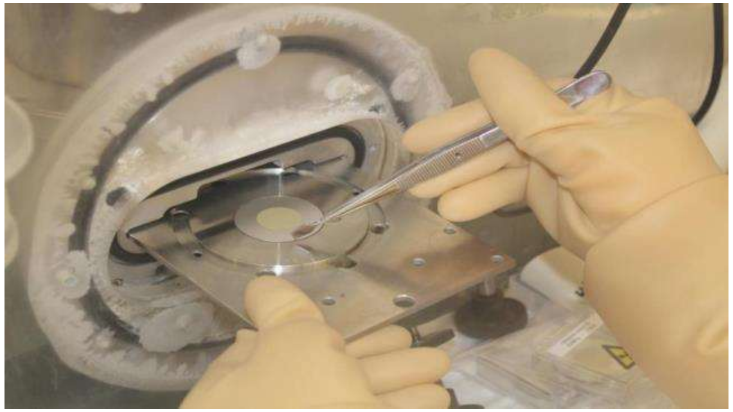
EC Joint Research Centre, Geel, Belgium

Workshop Ion Sources and Targets,
GANIL 6-8 Sept. 2023

This presentation

- Introduction
- Radiochemistry
- Preparation of actinide targets
- Preparation of substrates for actinide targets
- Characterization of actinide targets
- Ongoing R&D
- Resources and skills
- Summary



European Commission

Introduction

JRC sites

Headquarters in **Brussels** and research facilities located in **5 EU Countries:**

- Belgium (Geel)
- Germany (Karlsruhe)
- Italy (Ispra)
- The Netherlands (Petten)
- Spain (Seville)

Introduction

Nuclear laboratories at JRC - Geel

GELINA
neutron time-of-flight facility for high-resolution neutron measurements

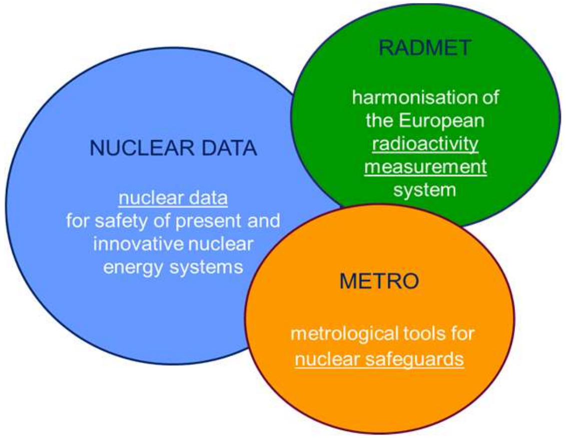
MONNET
tandem accelerator based fast neutron source

TARGET
nuclear target preparation laboratories

METRO
nuclear reference material and measurement facility

HADES
low-level gamma-spectrometry laboratory

RADMET
laboratories for standardisation of radionuclide activity



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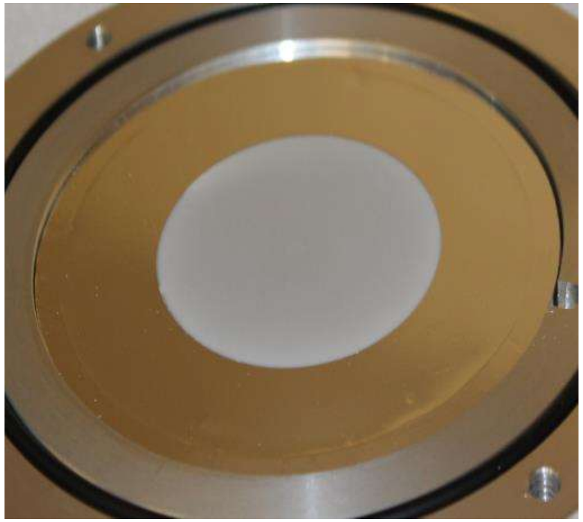
Introduction

What kind of targets are produced at JRC?

- Targets for neutron data measurements
- Commercially not available
- Not produced by other laboratories within EU
- Highly enriched in the isotope of interest
- Tailor made
- High quality (mechanically and chemically stable, homogeneous as far as possible)
- Well characterized (number of atoms per unit area or areal density of the nuclide under investigation, impurities, homogeneity)



^{241}Am deposit Ø 60mm on 25 µm Al foil on 2 mm thick Al-ring $Ø_{out}$ 110 mm, $Ø_{in}$ 100 mm

Introduction

Target preparation techniques

Thin layers

- by molecular plating (U, Pu, Np, Am, Th) on thin substrates
- by physical vapour deposition (^{235}U, ^{238}U, ^{6}LiF, ^{10}B, $C_{57}H_{110}O_6$, metal. Li) on thin substrates

Samples with a wide range of thicknesses

- by rolling and punching metal discs
- by pressing powders
- by dissolving and diluting solutions

Thin substrates

- by polymerization (polyimide foils)
- by gluing thin Al foils on metal rings

European Commission

Introduction

Target characterization techniques

- U, Pu, Np, Am, Th material: weighing, mass spectrometry
- U, Pu, Np, Am, Th deposits: low-geometry alpha-particle counting, alpha and gamma scanning
- ^{6}LiF, ^{10}B, $C_{57}H_{110}O_{6}$, metallic Li deposits: weighing and diameter determination
- Metal discs: weighing, thickness and area determination
- Compact powders: weighing, diameter and thickness determination
- Thin polyimide foils: spectrophotometry

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Radiochemistry

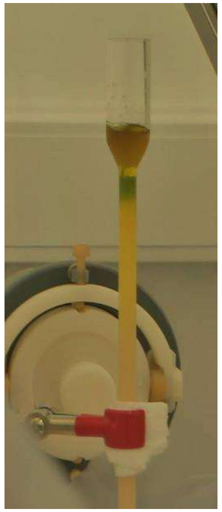
- Purification
  - by ion exchange
  - by extraction chromatography

Conditioning of Pu by a REDOX cycle

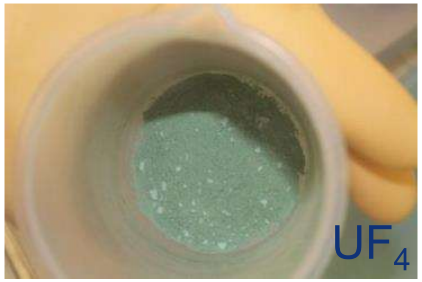
Pu(VI) → Pu(III) by adding 1.25 M $FeCl_2$

Pu(III) → Pu(IV) by adding 1 M $NaNO_2$

in HNO_3 (molarity depending on purification)



- Preparation of electrolyte for molecular plating
- Preparation of UF_4 for physical vapour deposition
  - Conversion of U_3O_8 into UF_4 via wet chemical precipitation method



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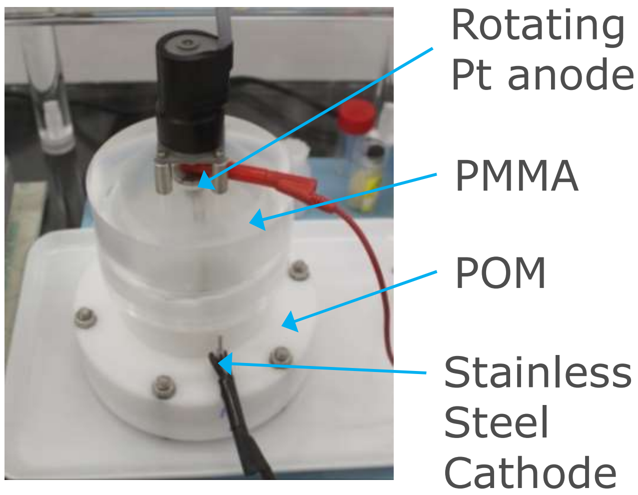
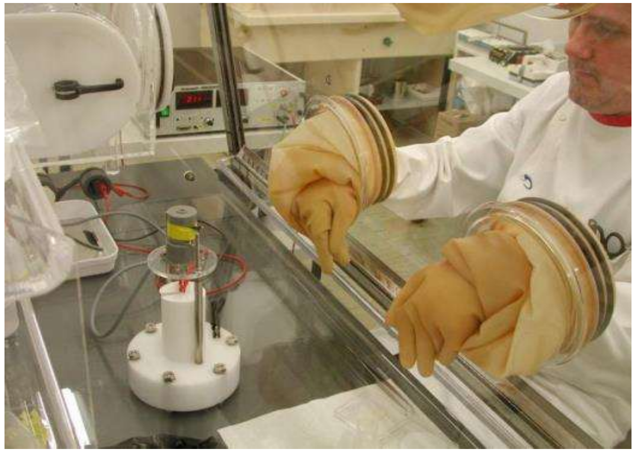
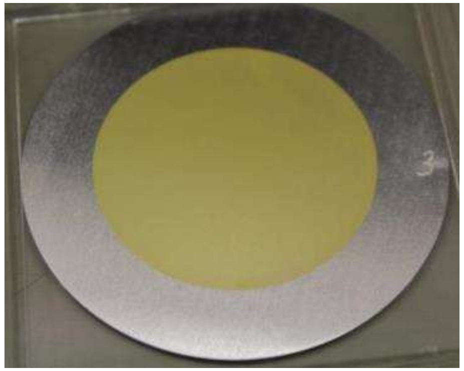
Preparation of actinide targets

Molecular plating based on the cathodic deposition of the actinide material in an isopropanol solution onto a substrate (aluminium)



Molecular plating cell





Material: 99.934% ^{235}U

Mass ^{235}U : 3.5 mg

Areal density ^{235}U: 279 µg·cm^{-2}

Deposit diameter: 40 mm

Backing: 0.25 mm Al Ø 60 mm

Preparation of actinide targets

Molecular plating

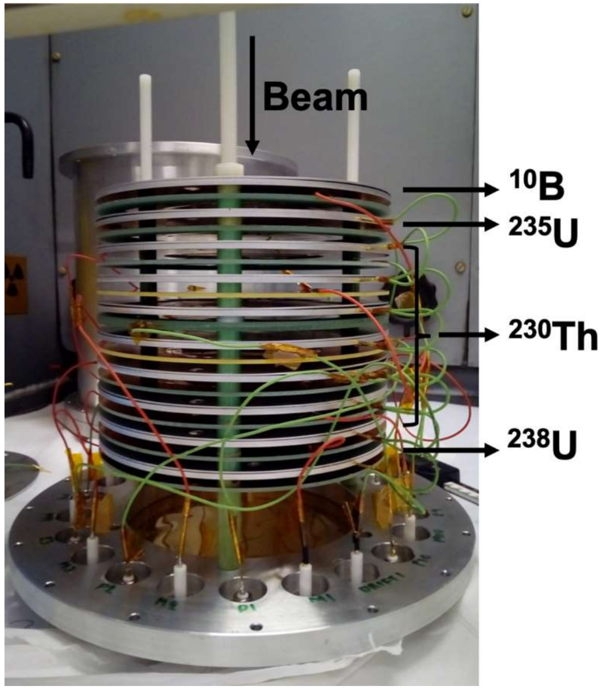
Measurement of the ^{230}Th(n,f) reaction cross-section at EAR-1 and EAR-2 of the CERN n_TOF facility

Material: 91.575% ^{230}Th

Activity ^{230}Th: 1.7 MBq
Mass ^{230}Th: 2.25 mg
Areal density ^{230}Th: 45 µg/cm^2

Deposit Ø: 80 mm

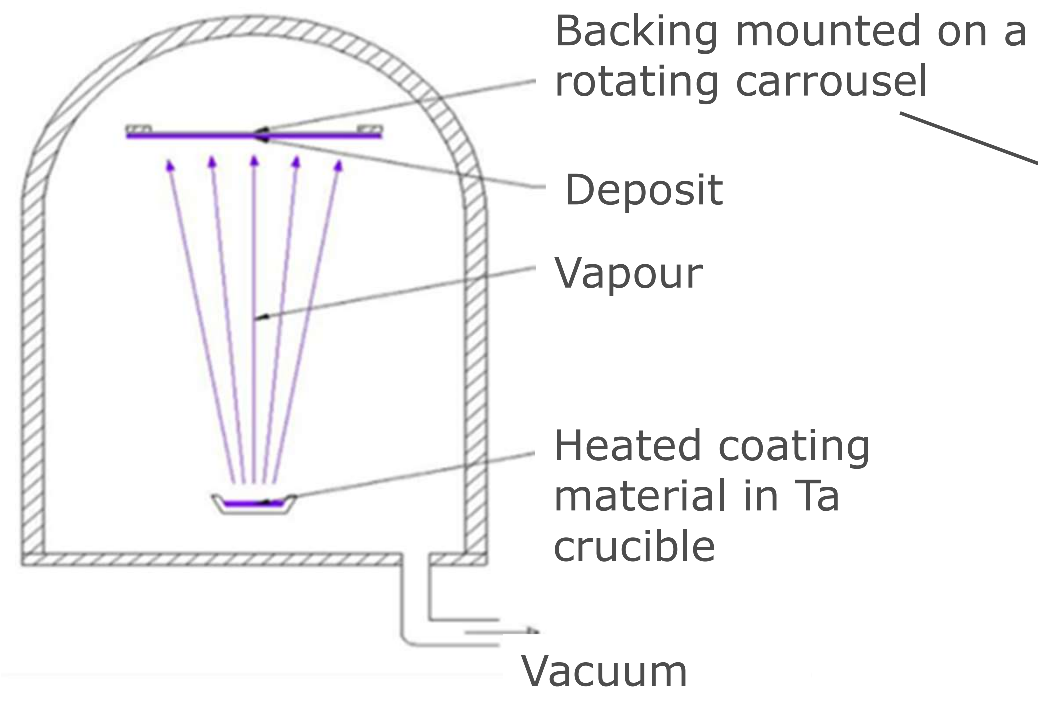
Backing: Al foil 0.025 mm thick glued on 1 mm thick Al-ring $Ø_{out}$ 110 mm, $Ø_{in}$ 100 mm

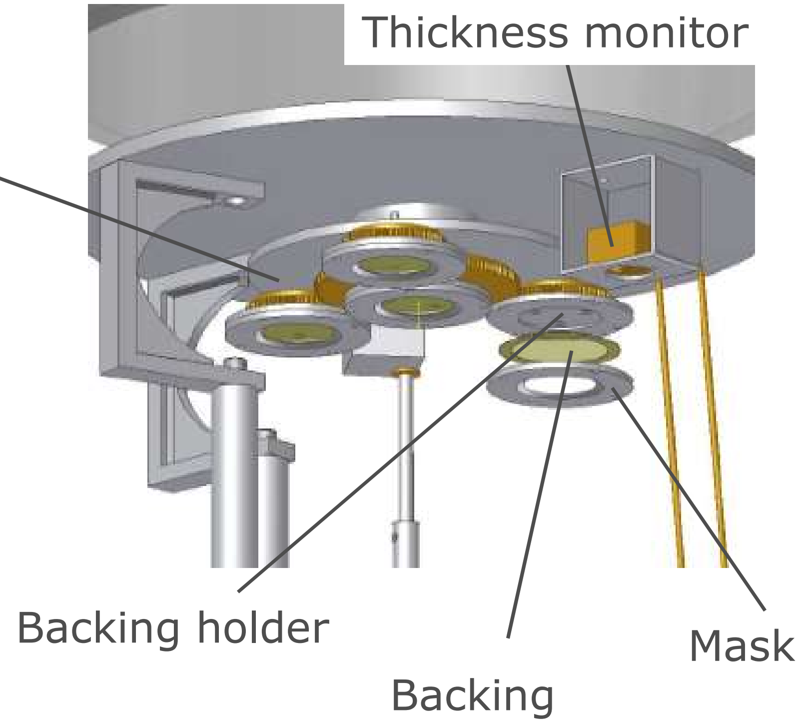


V. Michalopoulou et al., Measurement of the neutron-induced fission cross section of ^{230}Th at the CERN n_TOF facility, PHYSICAL REVIEW C 108, 014616 (2023)

Preparation of actinide targets

Physical vapour deposition, based on the condensation of a vaporized substance onto a backing





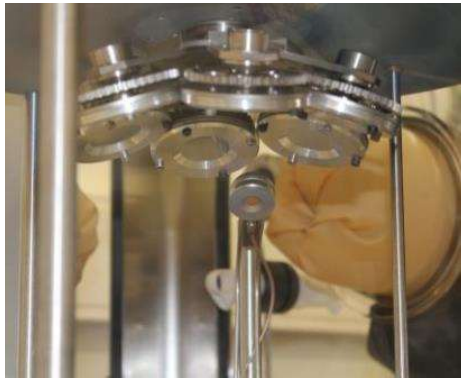
Preparation of actinide targets

Targets - Ions Sources
GANIL, France
6-8 Sept. 2023

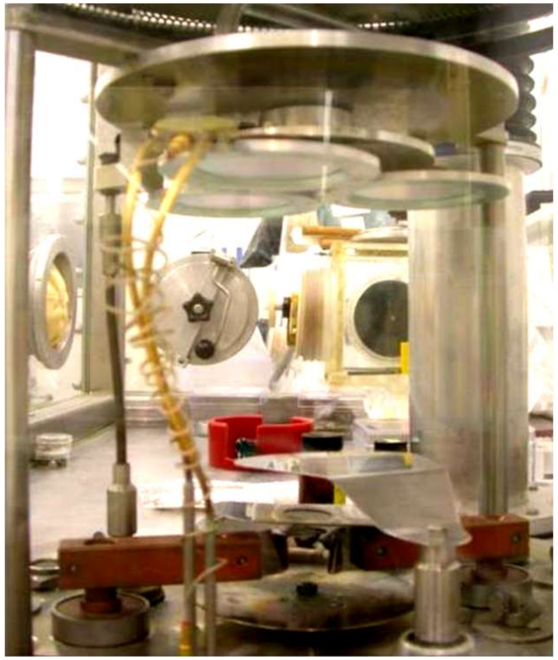
Physical vapour deposition

$^{238}UF_4$ evaporator

Resistance heating in Ta crucible
Sublimation of UF_4 at 1500°C



$^{235}UF_4$ evaporator



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Preparation of actinide targets

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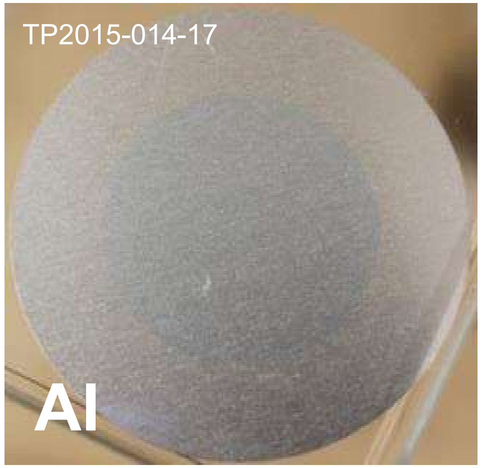
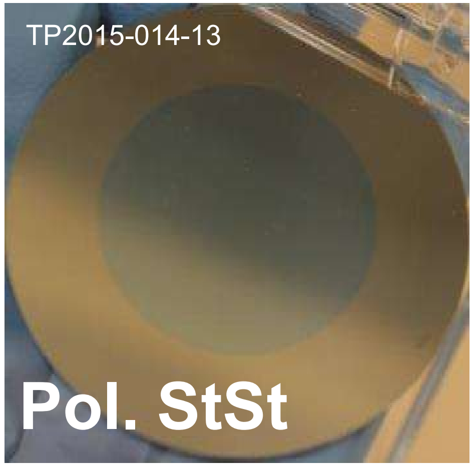
Physical vapour deposition





material: ^{238}U deposit Ø: 30 mm

mass ^{238}U : 0.3 mg areal density ^{238}U : 48 µg cm^{-2}

substrate: 0.25 mm thick, Ø 50 mm

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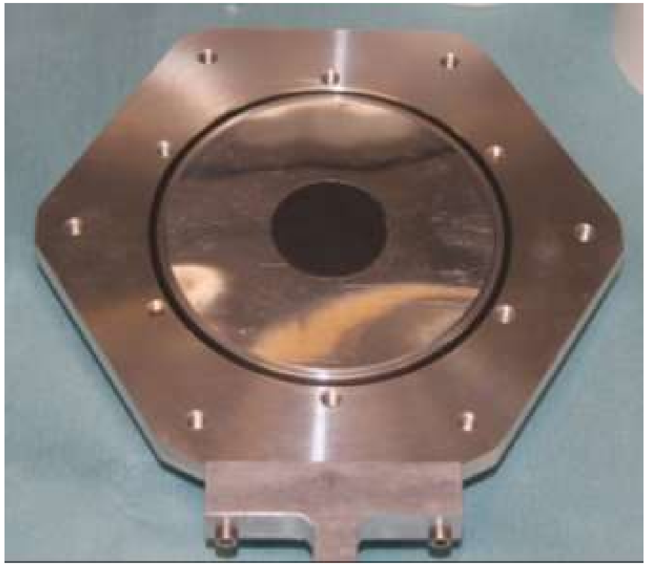
Preparation of actinide targets

^{233}U disc prepared by punching



n inelastic scattering cross-section of ^{233}U
for new generation cycles like the thorium cycle,
important to test and improve predictive power of
theoretical codes

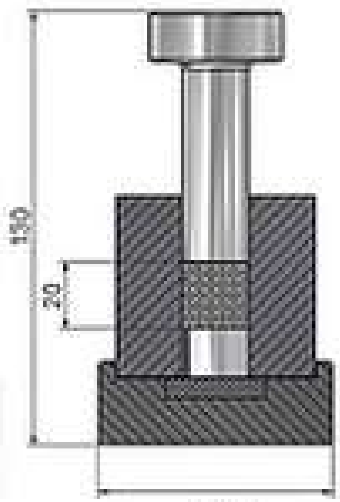
^{233}U disc Ø 30 mm 0.64 mm thick

Prepared by punching in a glove box
Characterized for mass and thickness in argon glove box
Mounted in a measurement holder with 50 µm Al foil

Preparation of actinide targets

Uniaxial pressing

1. Die filling
2. Compaction
3. Ejection



Hydraulic press in a glove box

5 mm Ø pellet of 0.1 g $^{238}U_3O_8$
hydraulic pressing at 10 kN

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Preparation of actinide targets

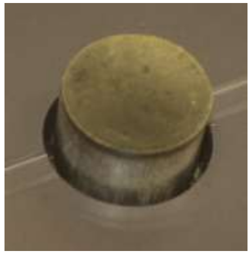
Uniaxial pressing $^{239}PuO_2$ pellets

$^{239}PuO_2$ Batch 716
99.97% ^{239}Pu

Analysed for impurities:
C, CO_2 and Cl

$^{239}PuO_2$ Batch 1756
99.90% ^{239}Pu

$^{239}PuO_2$ **Purified**
Batch 1756(p)
99.90% ^{239}Pu



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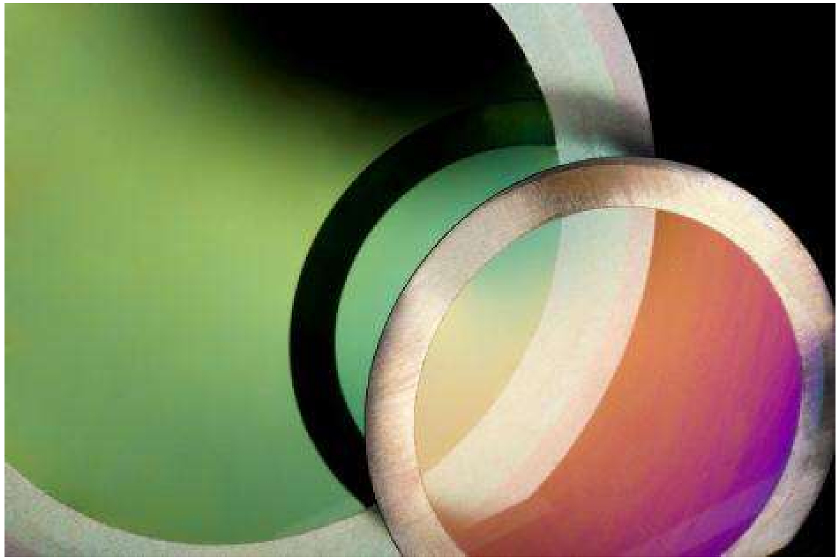
Preparation of substrates

Thin polyimide foil

- Prepared by polymerisation
- Ø 10-80 mm
- Areal density: 20-200 µg/cm2
- Mechanically strong
- Excellent resistance to
  - irradiation with charged particles
  - temperature
  - chemicals
- Not commercially available at thickness of interest



Preparation of substrates

Process to produce thin polyimide foils

Preparation of Amide-Acid (polycondensate) solution in dry atmosphere

1,2,4,5 - benzenetetracarboxylicdianhydrid + 4,4‘ – diaminodiphenylether in N,N' – dimethylformamide

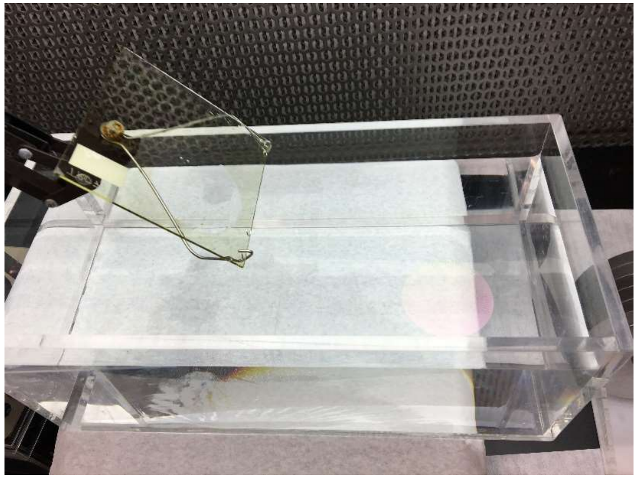
Cleaning of glass plates in oxygen plasma

Coating glass plates with polycondensate solution in Argon box

Polymerization 12 min at 350ºC

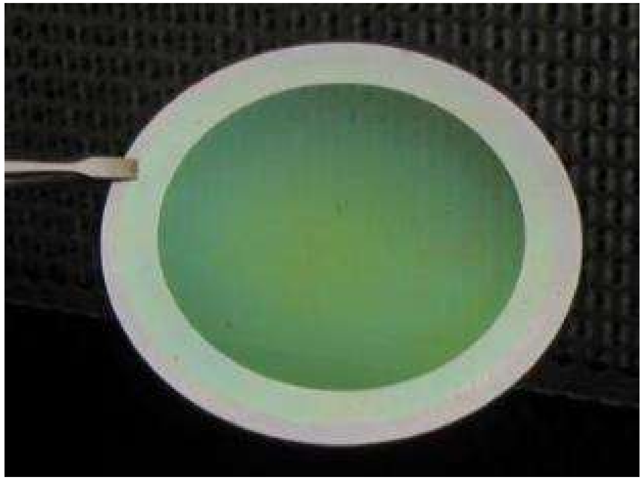
Preparation of substrates

Process to produce thin polyimide foils



Release of polyimide foil from glass plate

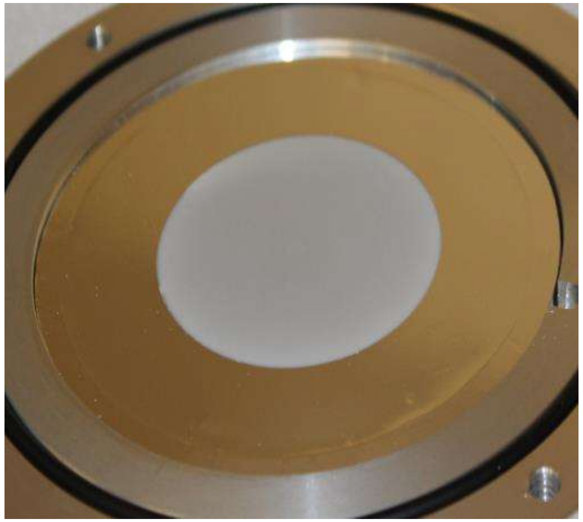
Transfer of polyimide foil onto ring



Polyimide foil on ring

Preparation of substrates

Mounting of Al foil with thickness of 10-30 μm on Al ring



^{241}Am deposit on 25 μm Al foil

Preparation of substrates

Cleaning process

for Al substrates to improve adhesion of the deposited layer

Al discs
- Chemically etching
- In a mixture of 80% H_3PO_4 - 4% HNO_3 - 16%H_2O

Thin Al foils glued on a ring
- Plasma cleaning/soft etching
- In argon-nitrogen

Low-pressure plasma cleaner

for glass plates to increase hydrophilicity

- Plasma cleaning/soft etching
- In oxygen

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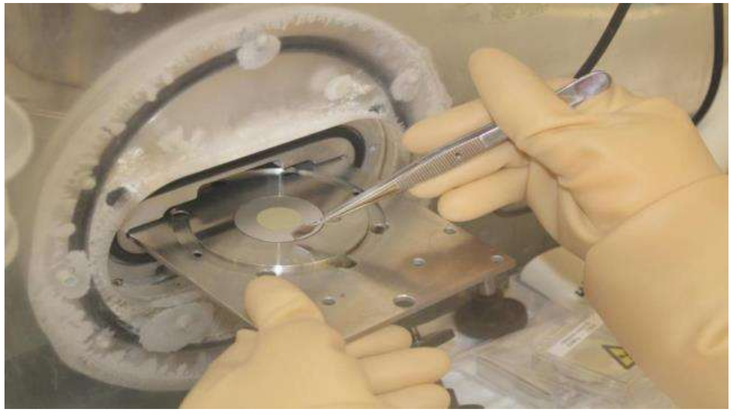
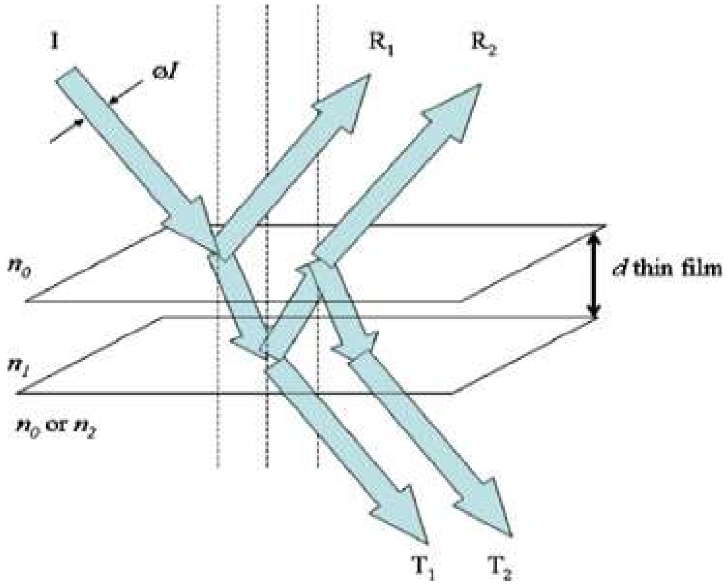


Characterization of actinide targets

Thickness measurement of polyimide foils by photo spectrometry



reflection mode

transmission mode

Characterization of actinide targets

Targets - Ions Sources
GANIL, France
6-8 Sept. 2023

Low-geometry alpha-particle counting: activity, homogeneity, impurity

Detector
diaphragm
Ø 20mm

50 200 mm

Actinide target

Detector
diaphragm
Ø 17mm

1637 mm

Actinide target

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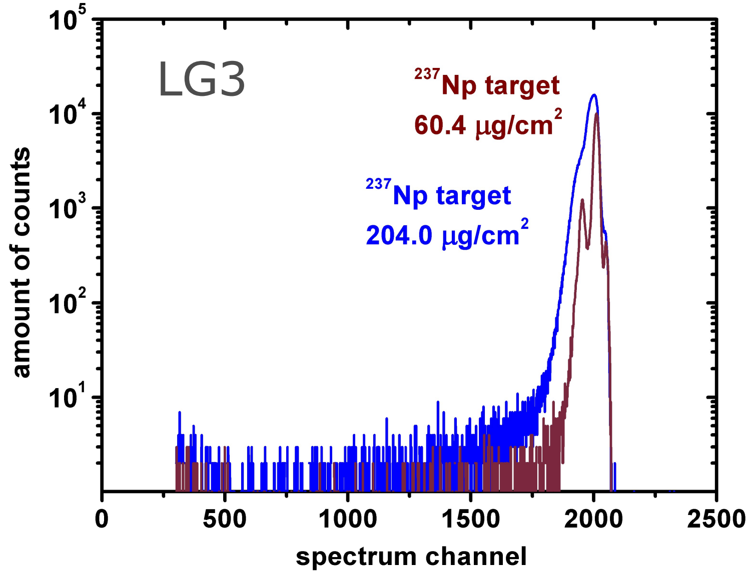
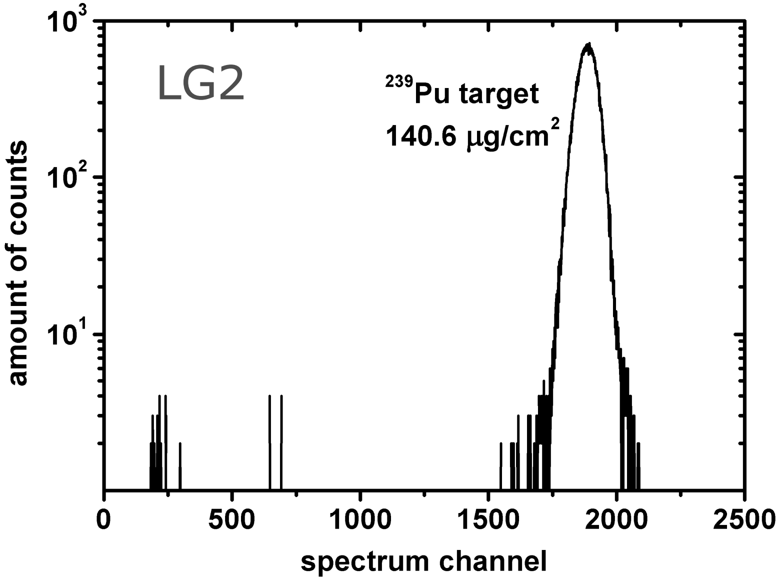
Characterization of actinide targets

Activity measurement by low-geometry alpha-particle counting





Measurement runs of 10000 s
Solid angle: 0.24 % of 4π sr

Measurement runs of 20000 s
Solid angle: 0.0007 % of 4π sr

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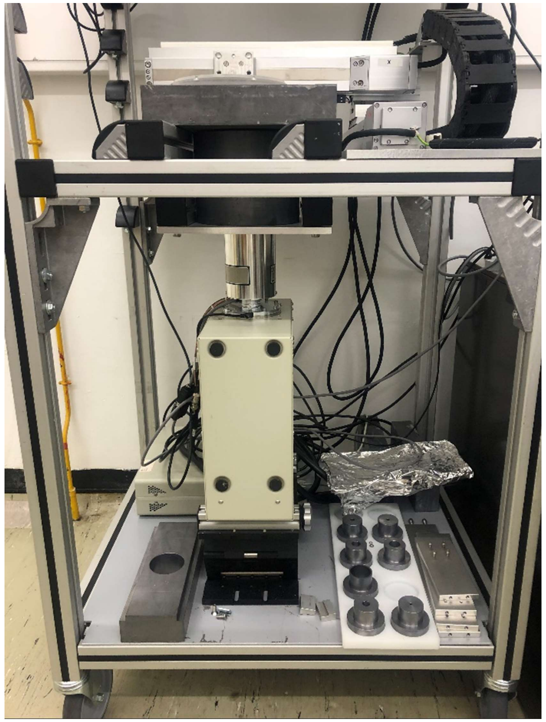
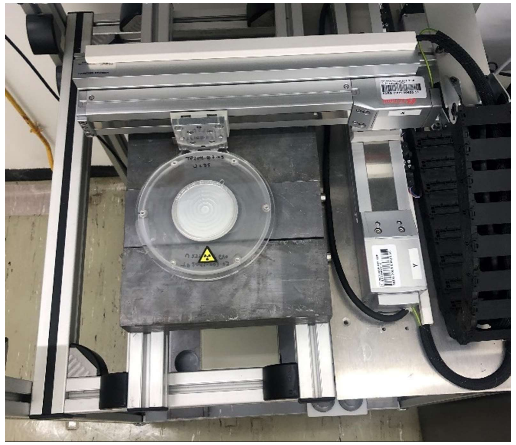
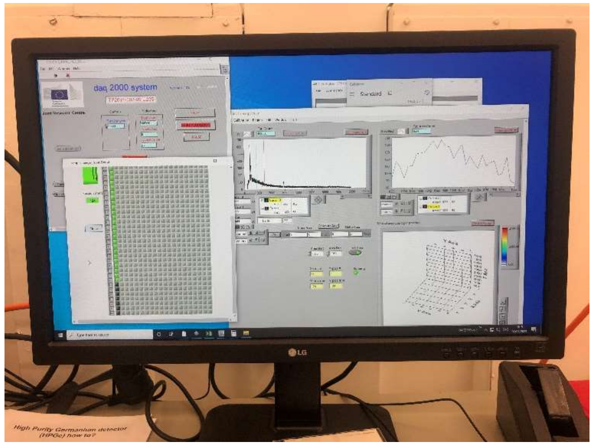
Characterization of actinide targets

Gamma spectrometry: impurity, homogeneity





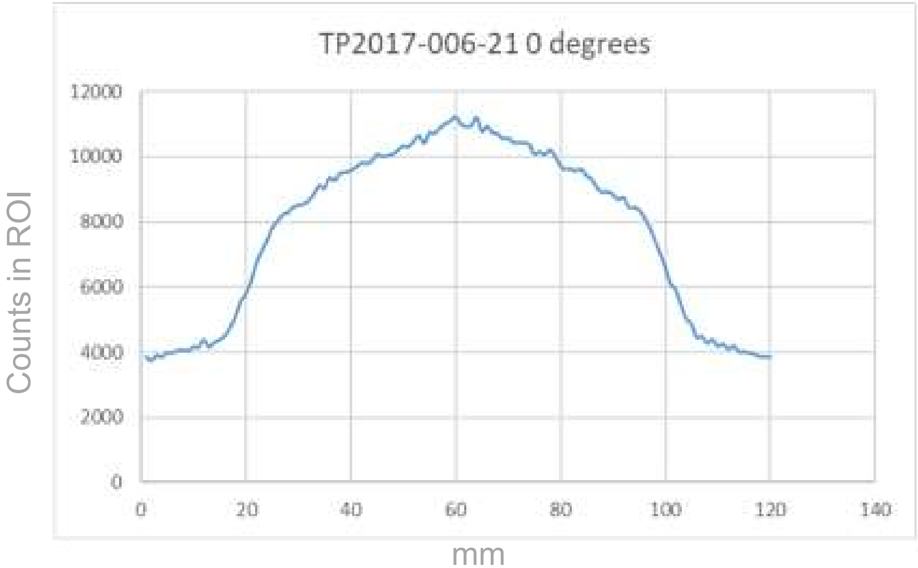


In-house made X-Y scanning system

Ge detector GR4520 cooled with an electrically refrigerated cryostat Cryo-Pulse 5 Plus

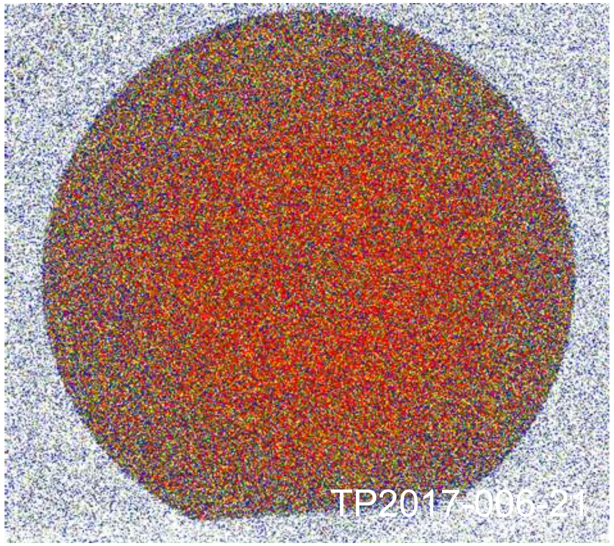
Characterization of actinide targets

Gamma spectrometry: homogeneity



Gamma-scan collimator 8 mm

^{230}Th

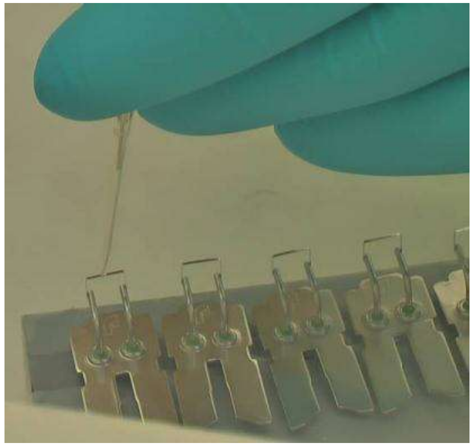


Autoradiograph

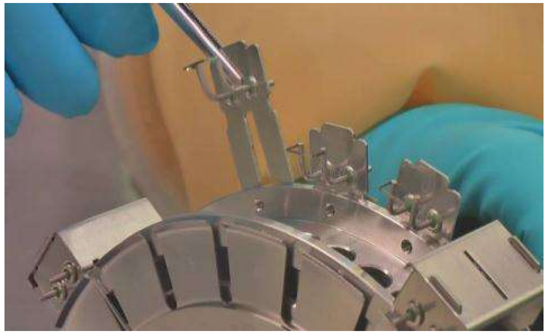
Characterization of actinide targets

Mass spectrometry: Atomic abundances

TRITON Thermal Ionization Mass Spectrometer



loading of the samples
and the standards on the Re filament

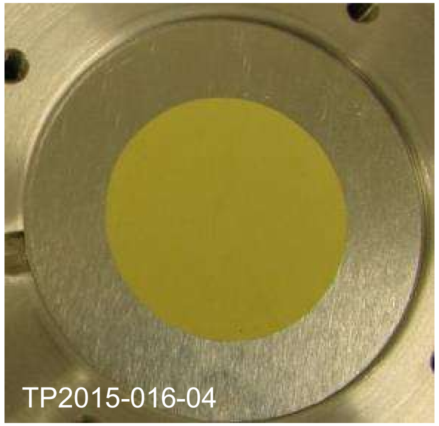


positioning of the filaments
in the magazine

Molecular plating versus physical vapour deposition

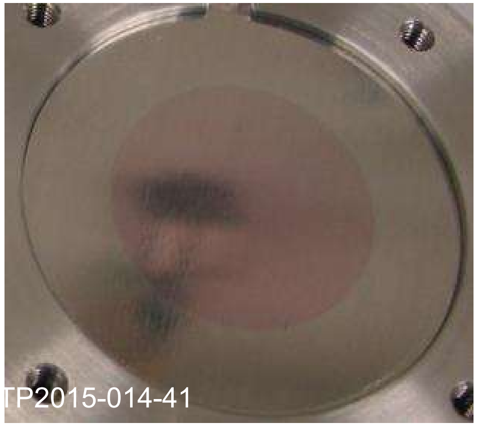
Chemical form

Molecular plating in C_3H_8O
of U_3O_8 in 0.75 M HNO_3
268 µg cm^{-2} ^{238}U on Al
Deposit Ø: 30 mm
Backing: 0.25 mm Al Ø 50 mm



U oxide? layer containing C_3H_8O
Oxidation state U(VI)

Physical vapour deposition
of UF_4
264 µg cm^{-2} ^{238}U on polished Al
Deposit Ø: 30 mm
Backing: 0.25 mm Al Ø 50 mm



UF_4 layer
Oxidation state U(IV)

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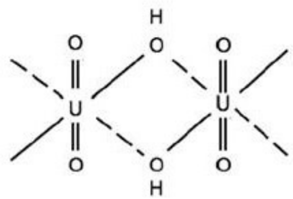
Molecular plating versus physical vapour deposition

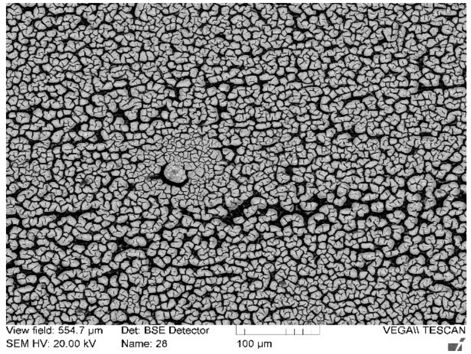
Morphology

Molecular plating 268 µg cm^{-2} ^{238}U on Al

- Maze-like layer
- U(VI)
- Major elements: U, C, O
- Contains isopropanol C_3H_8O



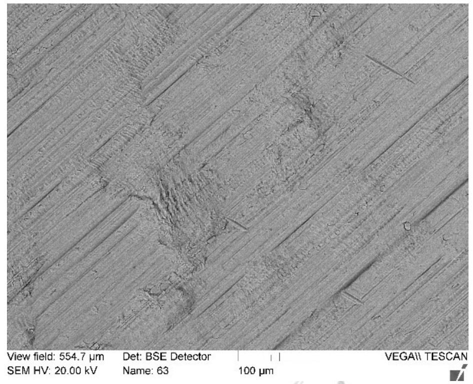
*Sadi et al. proposed the following structure



100 µm SEM Magnification 1.25kx

Physical vapour deposition 264 µg cm^{-2} ^{238}U on Al

- Smooth layer, follows roughness profile of substrate
- U(IV)
- Major elements : U, F
- UF_4 deposit

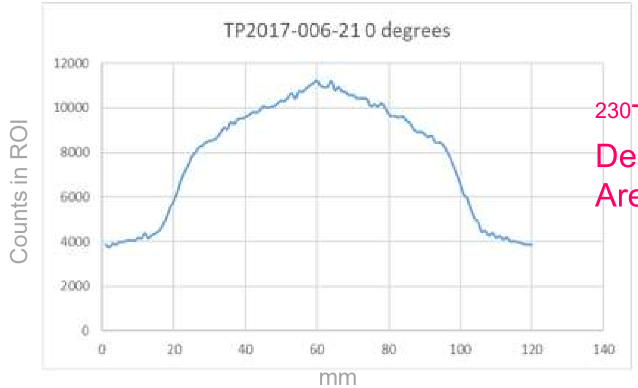


* S. Sadi, et al., Nucl Instr Meth n Physics Research A 655 (2011) 80–84

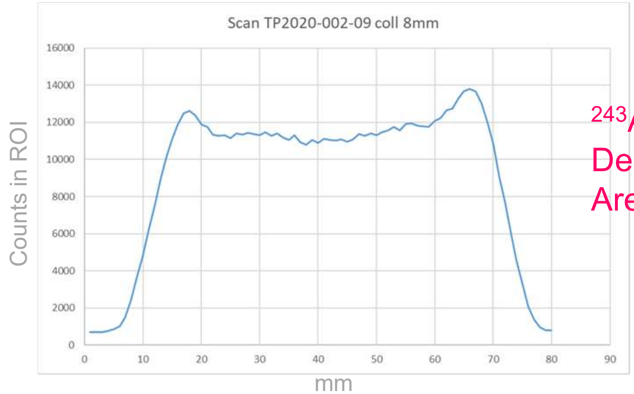
Molecular plating versus physical vapour deposition

Homogeneity

Molecular plating



^{230}Th
Deposit Ø: 80 mm
Areal density: 92 µg/cm^2



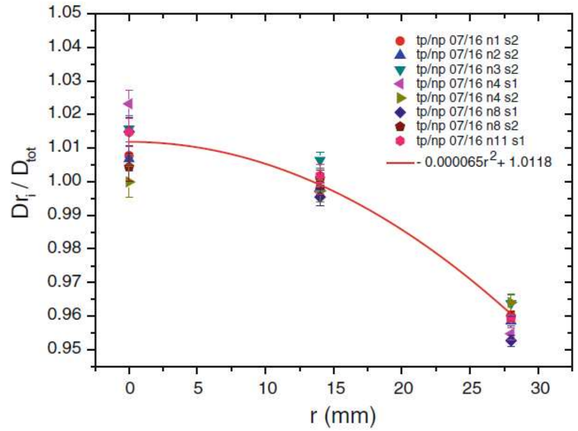
^{243}Am
Deposit Ø: 60 mm
Areal density: 3.3 µg/cm^2

Gamma scan (statistical uncertainty 1%)
About 8% difference in areal density between the centre and the edge of the deposit

Physical vapour deposition

Distance sublimation source to backing: 19.5 cm

^{235}U
Deposit Ø: 70 mm
Areal density: 450 to 600 µg/cm^2



Alpha scan
About 5% difference in areal density between the centre and the edge of the deposit

Molecular plating versus physical vapour deposition

Comparison

Process	Molecular plating	Physical vapour deposition
Yield	75-95%	< 5%
Time	3 hours	1 day (deposition 1 hour)
Chemistry	Preparation of electrolyte (0.5 day)	Conversion of U_3O_8 into UF_4 (1 week)
Equipment	30 kEuro (+ 30 kEuro glove box)	150 kEuro (evaporator integrated in glovebox)

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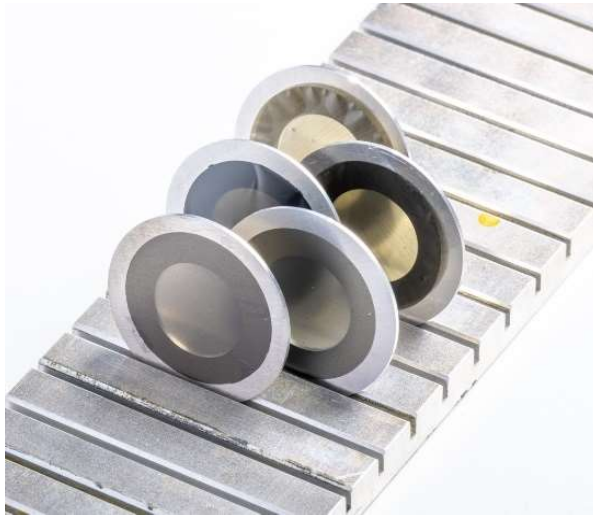


Ongoing R&D

Preparation of ^{235}U deposits by e-beam evaporation

New ^{235}U evaporator integrated in a glove box

- Resistance heating
- E-beam
- In situ RF plasma cleaning
- Movable sample stage (flexibility in yield vs homogeneity)
- Protection tube to prevent cross contamination for use of other elements

Ongoing R&D

Preparation of ^{235}U deposits by e-beam evaporation

New ^{235}U evaporator integrated in a glove box

Advantage e-beam:

- Original U_3O_8 material can be used instead of UF_4
- No time consuming wet chemical precipitation method

Ongoing R&D

Molecular plating on thin conductive polyimide foils

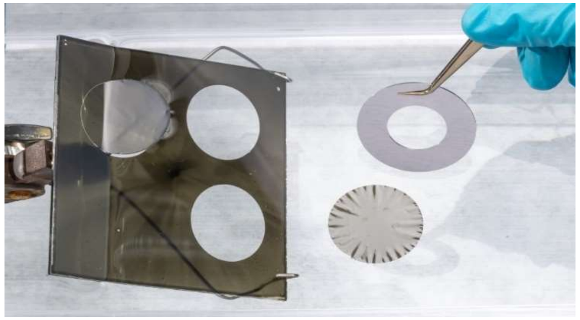
- Purpose: spectroscopic targets to perform fission cross-section and fragment yield measurements with a geometrical efficiency close to 2π.
- Preparation of conductive PI foils
  - Carbon fillers
  - Minimum thickness
  - Conductivity
  - Energy loss of alpha particles
  - Mechanical strength
- Re-design of molecular plating cell
- Molecular plating

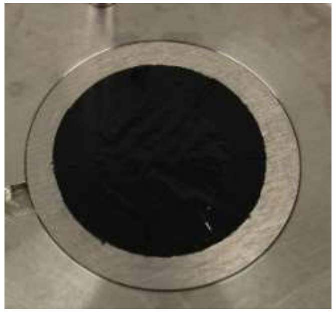
Targets - Ions Sources
GANIL, France
6-8 Sept. 2023

Ongoing R&D

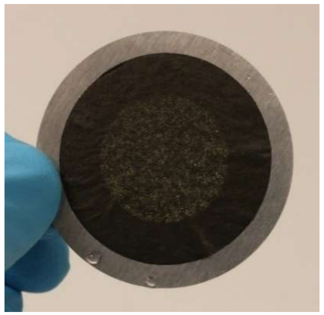
Molecular plating on thin conductive polyimide foils

Transfer of conductive polyimide foil onto ring

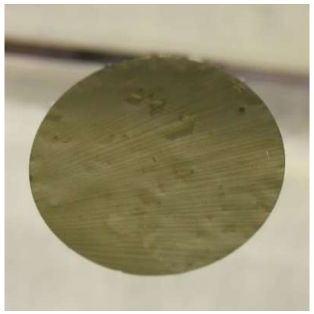




CNT



NPG



SLG

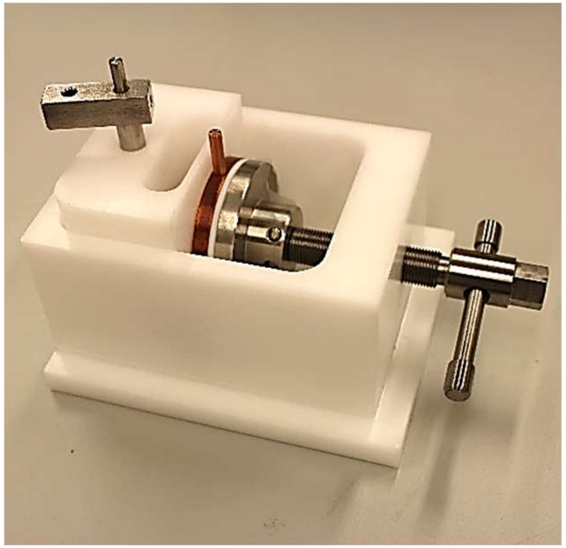
C filler Type	Areal density (μg/cm^2)
PI/CNT (Carbon Nano Tubes)	420 - 645
PI/GNnP (Graphene NanoPlatelets)	868 - 970
PI/SLG (Single Layer Graphene)	195 - 790
PI/E-GN (Customised graphene suspension in DMF)	70 - 230

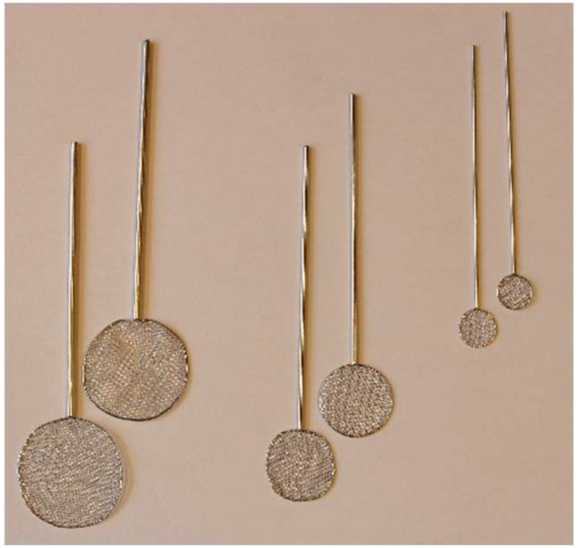
European Commission

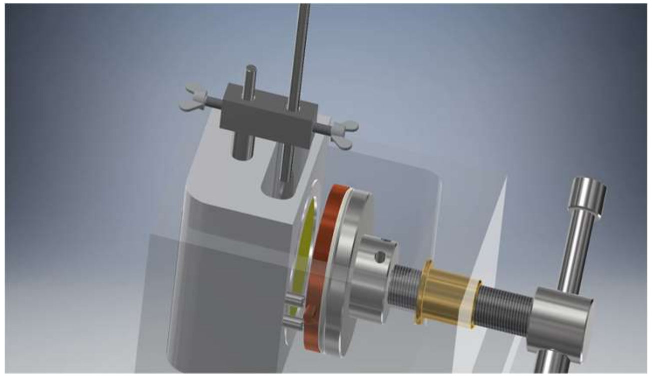
Ongoing R&D

Molecular plating on thin conductive polyimide foils

New Design of Molecular Plating Cell







- Even pressure distribution seal
- No solvent weight on thin backing
- POM base (one piece) also a spill tray
- Ti/Pt coated anode (same size/shape as deposit)
- Can be extended for various sizes of backings

Design based on molecular plating cell described in paper:
M.N. Torrico, R.A. Boll, M. Matos, Electrodeposition of actinide compounds from an aqueous ammonium acetate matrix: Experimental development and optimization., NIM A 790 (2015) 64–69

This presentation

Resources and skills

Key resources for production of actinide targets

- Actinide material:
  - **Supplier of enriched actinide material!**
  - Public procurement procedure
  - Price material: depends on radioisotope, purity, analysis
  - Dispensing/packing costs if applicable
  - Transport: price depends on type and amount of radioactive material

^{243}Am as oxide powder from ORNL, transport UN2915 to JRC Geel

Resources and skills

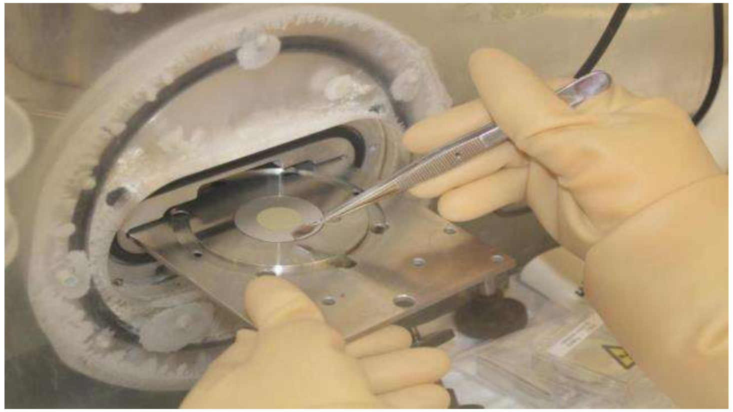
Key resources for production of actinide targets

- Basic laboratory tools (glass ware, pipettes, purification columns etc.)/ solvents, chemicals
- Substrate/vial/container
- Cleaning equipment (e.g. Plasma system)
- Fume hood
- Glovebox in under-pressure
- Molecular plating set-up
- Physical vapour deposition set-up
- Mechanical transformation equipment (rolling, punching, pressing)
- Transport container

Resources and skills

Key resources for characterization of actinide targets

- Alpha counter (incl. scanning)
- Gamma spectrometer (incl. scanning)
- Microbalance
- Micro-meter/calliper
- Mass spectrometer
- Scanning electron microscope with energy dispersive X-ray spectroscopy



Resources and skills

Skills

Scientific and technical competences in

- Basic laboratory work
- Mass metrology
- Nuclear chemistry
- Nuclear physics
- General and nuclear engineering
- Working in a fume hood
- Working in a glove box
- Dexterity

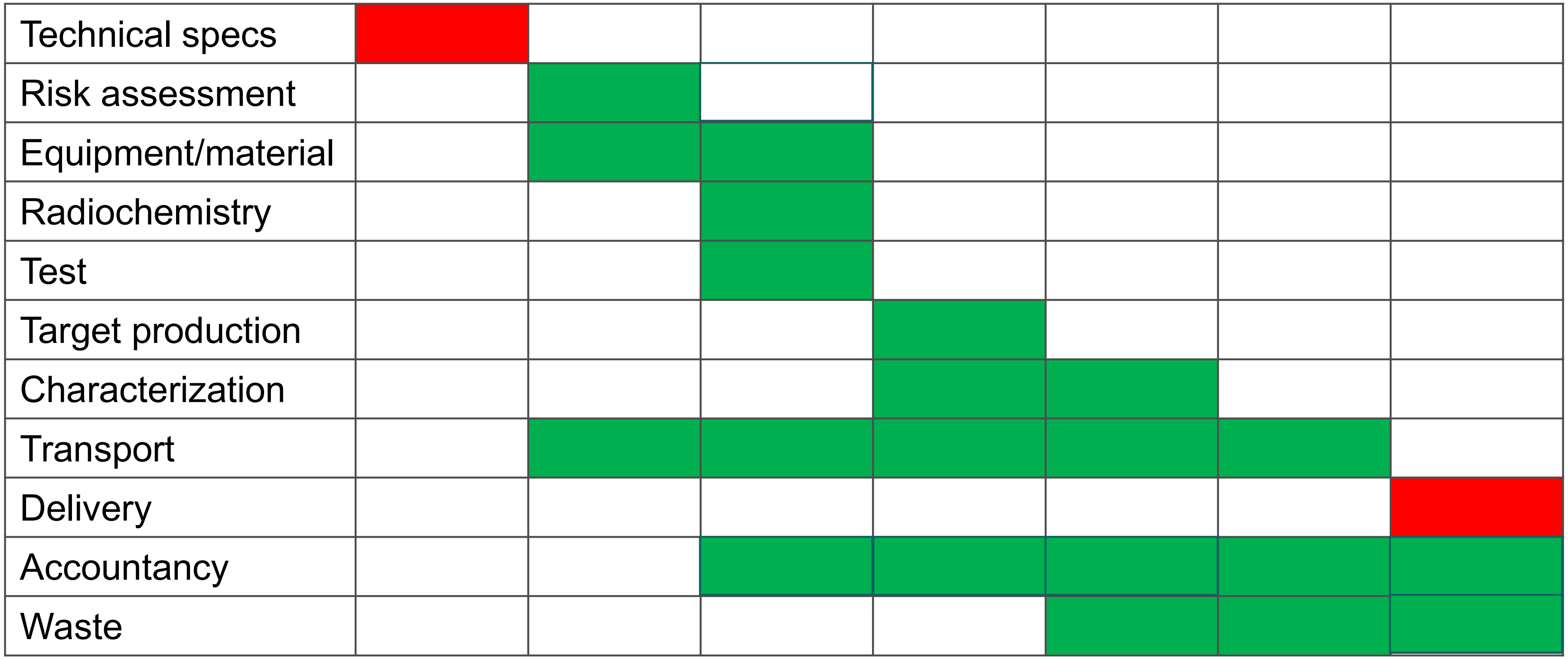
Resources and skills

Skills

- Concerned with nuclear safety
  - Radioprotection
  - ALARA (as low as reasonably achievable), justified
  - Radioactive waste
  - Radioactive transport
- Concerned with nuclear safeguards
  - Accountancy of fissile material
- Concerned with nuclear security
  - Clearance
- Concerned with legal/finance
  - Procurement

Summary

Simplified diagram of a target production planning



Summary

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- G. Sibbens, A. Moens, R. Eykens, D. Vanleeuw, F. Kehoe, H. Kühn, R. Jakopic, S. Richter, A. Plompen, Y. Aregbe, **Preparation of ^{240}Pu and ^{242}Pu targets to improve cross section measurements for advanced reactors and fuel cycles,** J Radioanal Nucl Chem (2014) 299:1093–1098
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- A. Moens, P.A. Celdran, H. Hein, G. Sibbens, D. Vanleeuw, S. Van Winckel, **Production of powder targets for neutron-induced cross section measurements**, EPJ Web of Conferences 285, 04002 (2023)
- J. Karpinska et al., **Preparation of thin conductive polyimide foils for nuclear chemistry**, in preparation

European Commission

Thank you
and keep in touch

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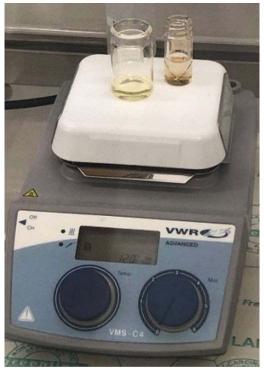
Extra slides with detailed information

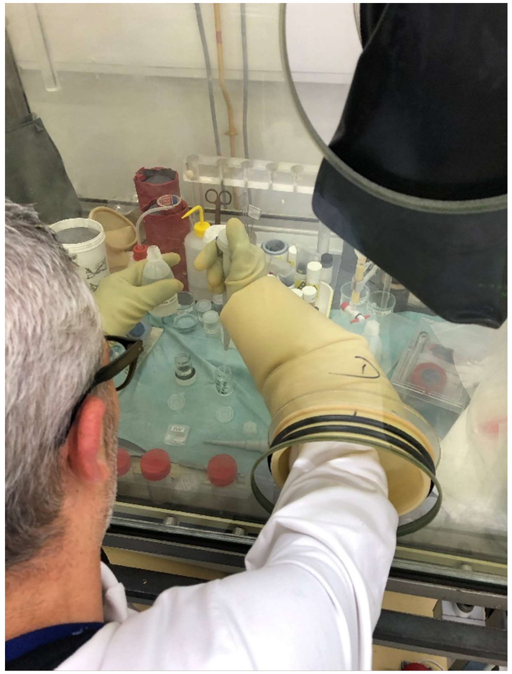
Radiochemistry

Purification

Purification e.g. for Am, for Pu

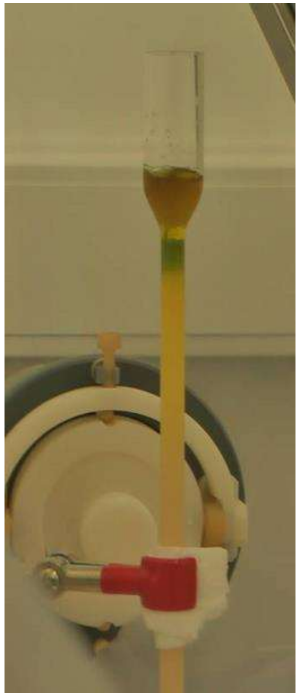
- Dissolution of the actinide oxide powder
  in concentrated HNO_3 and
  if needed small amount of 1% HF at about 100 ºC
- Evaporation near dryness at about 100 ºC
- Conditioning of Pu by a REDOX cycle
  Pu(VI) → Pu(III) by adding 1.25 M $FeCl_2$
  Pu(III) → Pu(IV) by adding 1 M $NaNO_2$
  in HNO_3 (molarity depending on purification)





Radiochemistry

Purification of Pu for Am by ion exchange



Loading Pu
in 8 M HNO_3

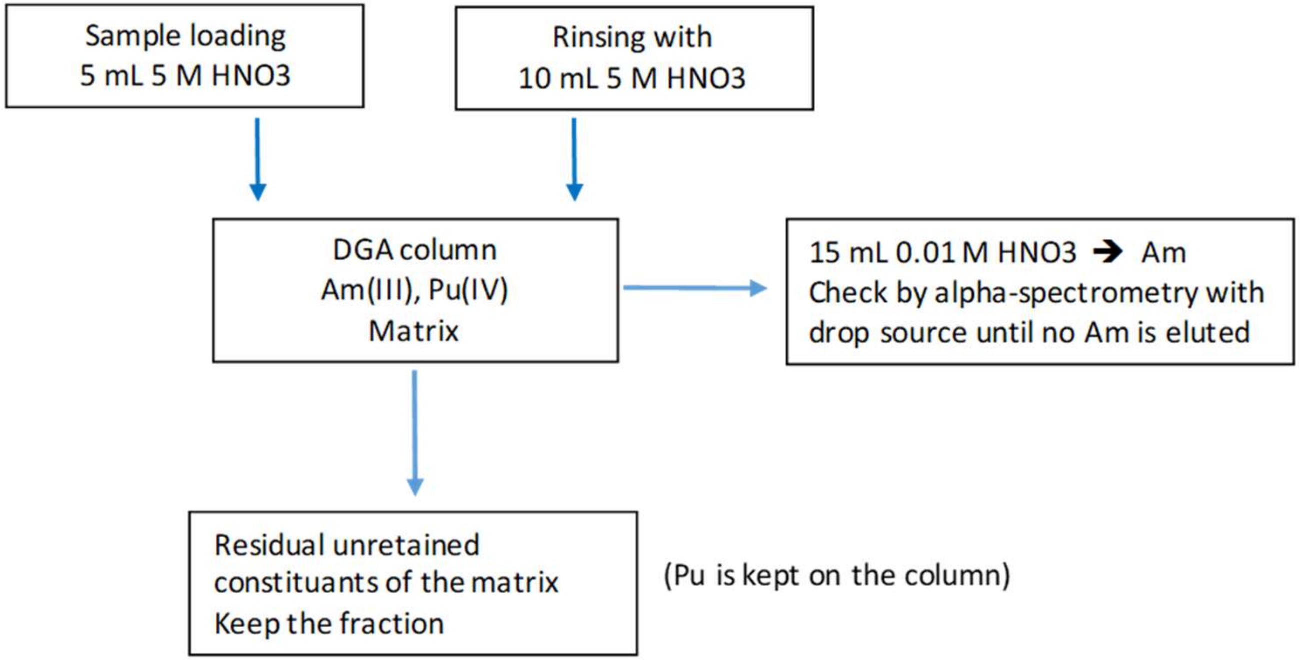
Stripping by adding
8 M HNO_3

Eluting Pu by adding
0.35 M HNO_3

anion exchange resin: Dowex 1-X8
particle size: 0.09 - 0.25 mm

Radiochemistry

Purification of Am for Pu by extraction chromatography



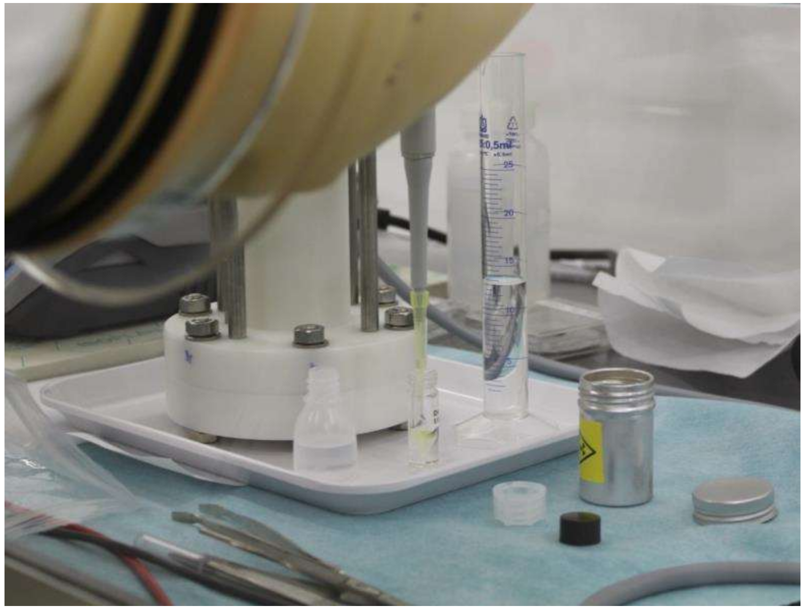
DGA resin, normal, 12 mg/mL, 100-150 µm particle size
Great affinity for americium

Radiochemistry

Electrolyte for molecular plating

Electrolyte for molecular plating: isopropanol + actinide dissolved in 0.75 M HNO_3

- Dissolution of the actinide oxide powder
  in concentrated HNO_3 and
  if needed small amount of 1% HF at about 100 ºC
- Evaporation near dryness at about 100 ºC
- Dissolution in 0.75 M HNO_3



Radiochemistry

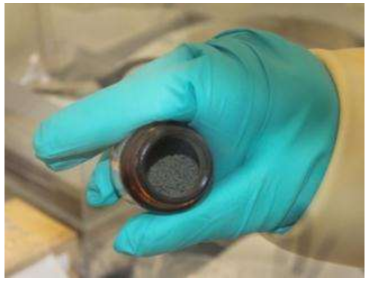
UF_4 for physical vapour deposition

Sublimation in vacuum of

- U_3O_8 requires T >2500°C which is not possible via resistance heating
- UF_4 possible at around 1500°C

Conversion of U_3O_8 into UF_4 via wet chemical precipitation method

Starting material U_3O_8

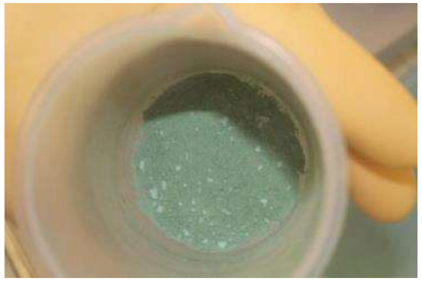


$$U_3O_8 + 6HCl \rightarrow 3UO_2Cl_2 + 2H_2O + H_2\uparrow$$

$$U(VI)O_2Cl_2 \cdot xH_2O + SnCl_2 \rightarrow U(IV)Cl_4 + Sn(OH)_2 \cdot yH_2O$$

$$UCl_4 + 4HF \rightarrow UF_4\downarrow + 4HCl$$

Resulting material UF_4



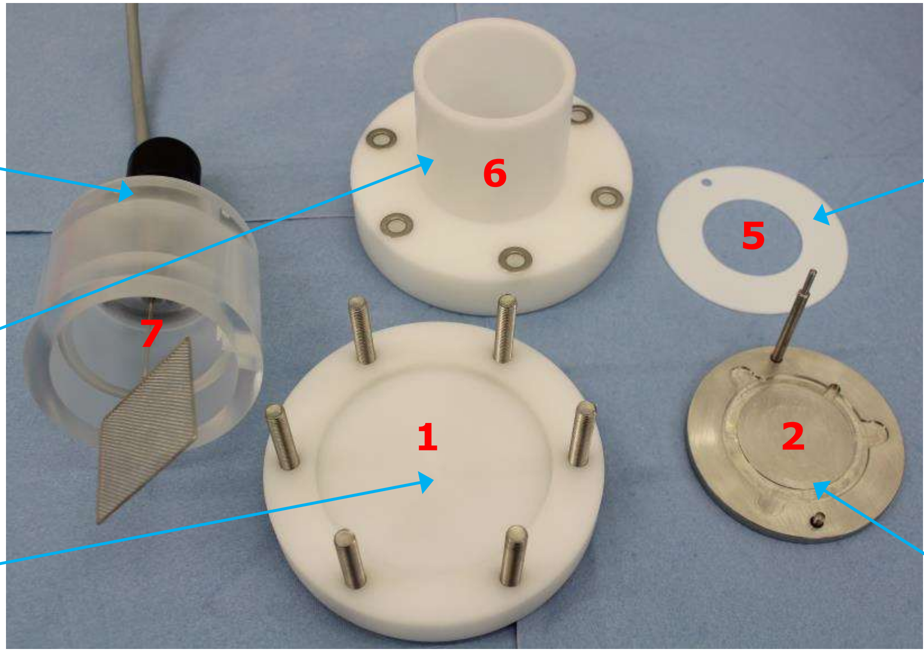
Preparation of actinide targets

Molecular plating cell

7 PMMA upper part of molecular plating cell with motor and rotating Pt anode

6 POM middle part of molecular plating cell

1 POM bottom part of molecular plating cell



5 PTFE compression ring

Mask defining the diameter of the deposit **4**

Substrate Al disc **3**

2 Stainless steel substrate holder (cathode)

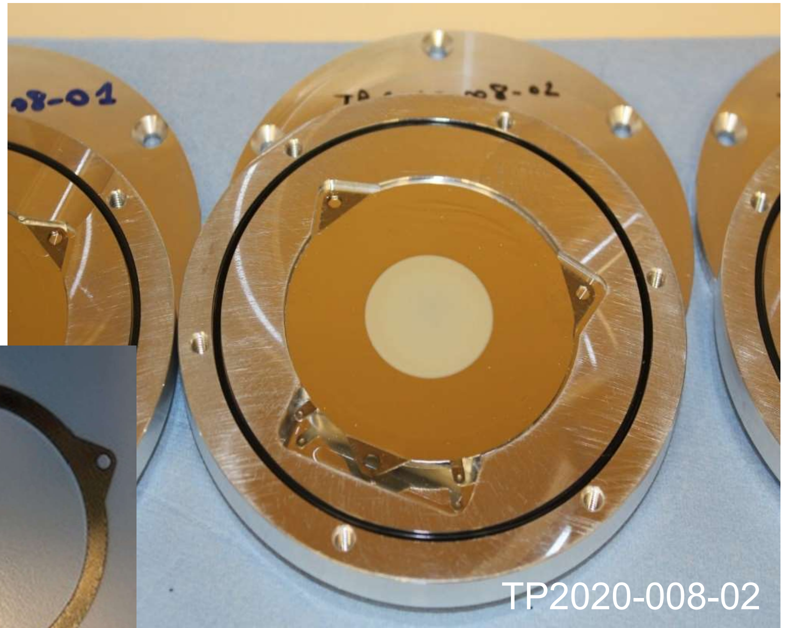
Preparation of actinide targets

Molecular plating

Material: 99.8921 % ^{240}Pu

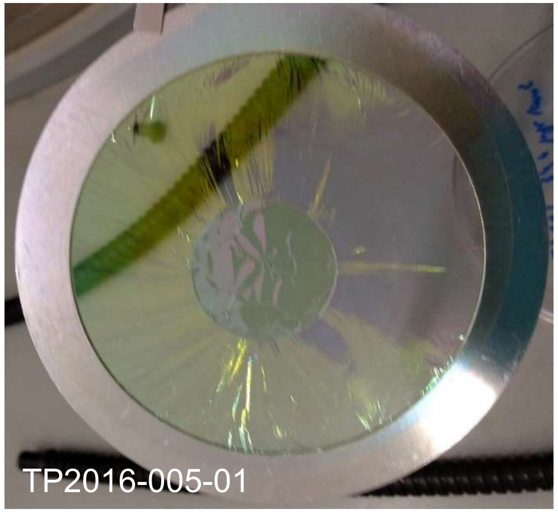
Activity ^{240}Pu : 4.1 MBq
Mass ^{240}Pu: 487.8 µg
Areal density ^{240}Pu : 57.04 µg/cm^2

Deposit Ø: 33 mm
Backing: 18 µm Aluminium stretched and glued on 0.3 mm thick stainless steel frame $ø_{out}$ 74 mm $ø_{in}$ 64 mm



Preparation of actinide targets

Physical vapour deposition



Material: 99.998% ^{238}U

Mass ^{238}U : 1.84 mg

Areal density ^{238}U: 377 μg·cm^{-2}

Deposit diameter: 20 mm

Backing: 34 μg/cm^2 polyimide foil
on 1 mm thick Al ring $Ø_{out}$ 90 mm $Ø_{in}$ 70 mm

For the development of innovative techniques and instrumentation for fission cross section measurements

Preparation of actinide targets

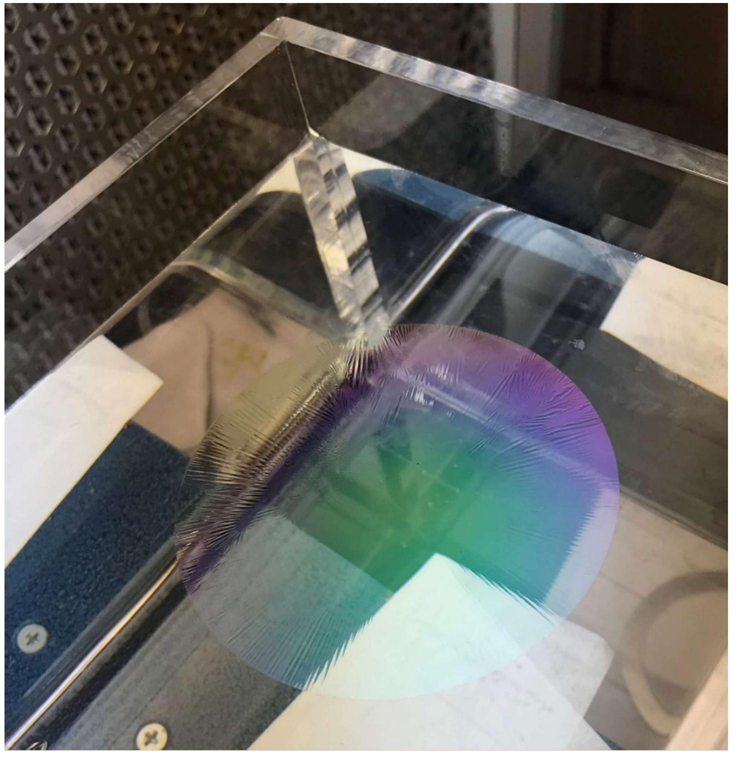
Uniaxial pressing

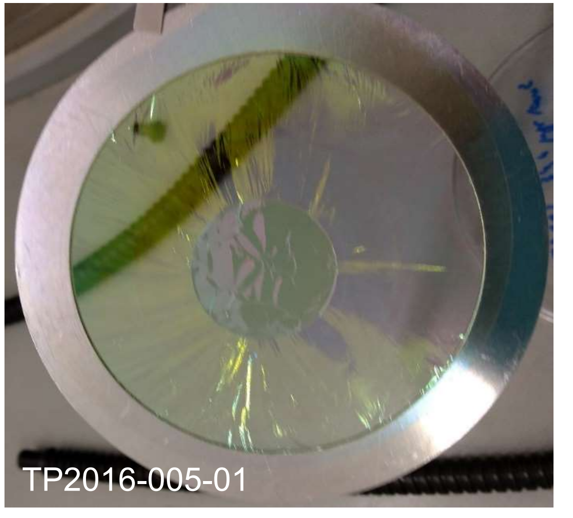
- 5 mm Ø pellet of 0.1 g $^{238}U_3O_8$
- low $^{236}U/^{238}U$ ratio of $7 \cdot 10^{-12}$
- Produced by hydraulic pressing at 10 kN in glove box
- Characterized for mass by weighing in glove box

Pellet is used in a campaign to measure the $^{235}U(n,\gamma)$ cross-section deduced by quantify the number of ^{236}U nuclei produced after neutron irradiation

Preparation of substrates

Process to produce thin polyimide foils





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on 1 mm thick Al ring $Ø_{out}$ 90 mm $Ø_{in}$ 70 mm

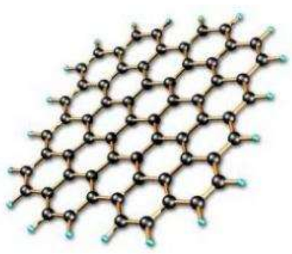
Ongoing R&D

Targets - Ions Sources
GANIL, France
6-8 Sept. 2023

Molecular plating on thin conductive polyimide foils

Carbon Fillers (PI/C) used as conductive fillers

SLG
Single Layer Graphene

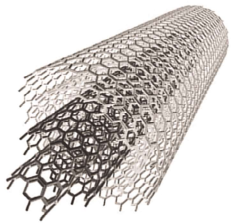


1-5 atomic layer graphene nano sheets
650 to 750 m^2/g
500 to 700 S/m

GNnP
Graphene Nano Platelets

2–10 nm
5 µm diameter
20 to 40 m^2/g
80000 S/m

CNT
Carbon Nanotubes



10-20 nm diameter
5-15 mic length

CB
Superconductive Carbon Black

E-GN
Customised graphene suspension in DMF

~5mg/ml

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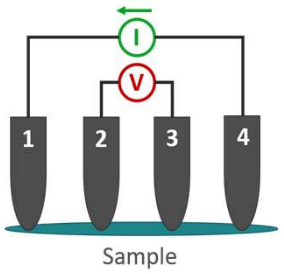
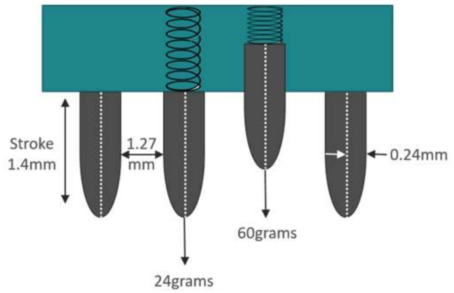
Ongoing R&D

Targets - Ions Sources
GANIL, France
6-8 Sept. 2023

Molecular plating on thin conductive polyimide foils

Conductivity of foils





Four Point Probe
- Easy to use
- Wide Current Range
- Non- Destructive
- Rapid Material Characterization

Id	Areal density µg/cm2	Conductivity Siemens/m
Au/PI	-	2.384
C (GSI)	121.43	782.9
CNT/PI	423.95	44.44
GNnP/PI	868.12	26.67
SLG/PI	148.25	0.6453
	309.95	0.5864
	590.55	0.142
	647.73	0.4066
E-GN/PI	58.07	1.624
	100.56	0.7351
	171.32	0.3504
	213.01	0.1939

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Ongoing R&D

Molecular plating on thin conductive polyimide foils