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14th International Exchange Meeting on Partitioning & Transmutation

Overview of Post-Irradiation Examination results on Minor Actinide bearing fuels within the FUTURIX-FTA international collaboration

D. Freis, F. Delage**, G. Cécilia**, J. Lamontagne**, L. Loubet**, C. Sabathier**, R. Nasyrow*, D. Papaioannou*, G. Paperini*, W. de Weerd*, J. Somers*, R. Konings**

**European Commission, JRC, Directorate G - Nuclear Safety and Security*

***CEA/DEN/DEC, Cadarache Centre, Fuel Study Department, France*

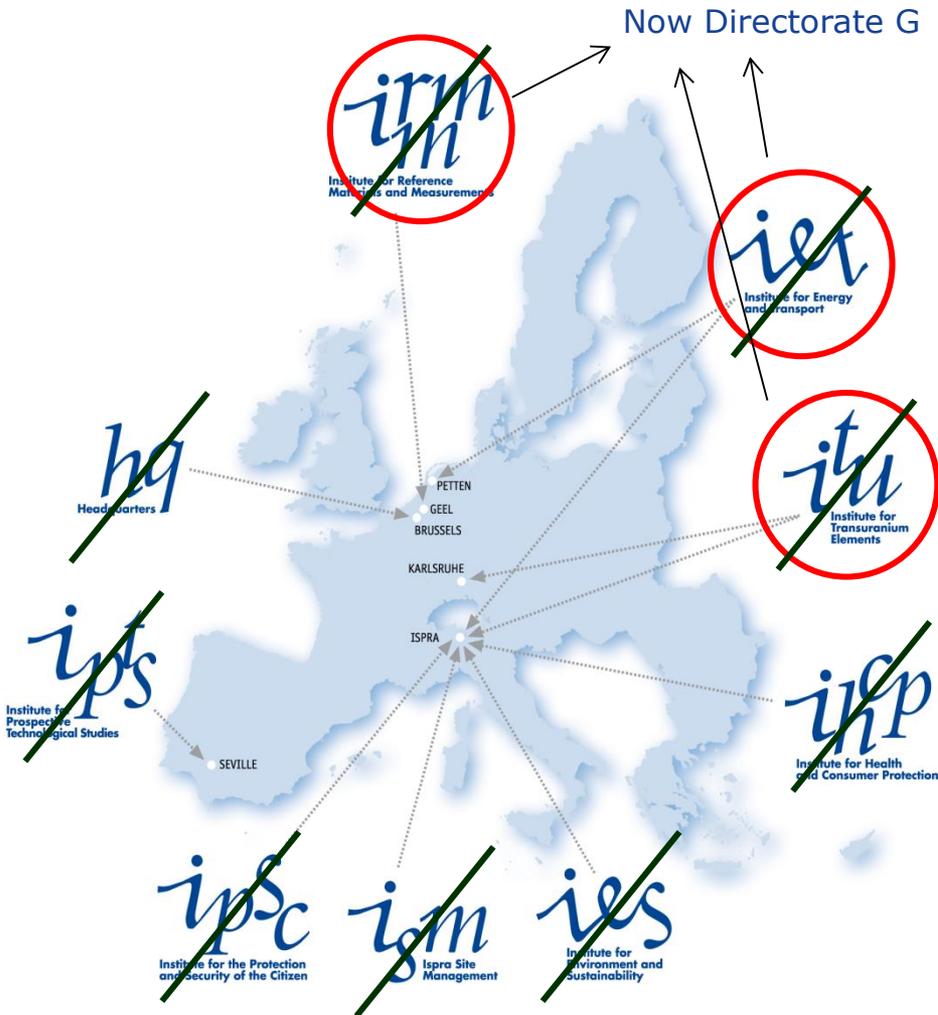


European
Commission

Overview

1. The Joint Research Centre – Directorate G
2. The FUTURIX-FTA Irradiation Experiment
3. Fuel Preparation (FX 5 & 6) / Fuel Characteristics
4. Irradiation Conditions
5. Post Irradiation Examination of CerCer & CerMet Fuels
6. Summary

The Joint Research Centre (JRC)



The JRC is the European Commission's science and knowledge service.

JRC has 10 Directorates, is located across 5 EU Member States and employs over 3000 people.

JRC's nuclear work programme, funded by EURATOM is carried out by Directorate G – Nuclear Safety and Security, which employs more than 500 people at sites in Karlsruhe (former ITU), Petten, Geel and Ispra (red circles).

Directorate G contributes to the scientific foundation for the protection of the European citizen against risks associated with the handling and storage of highly radioactive material.

The FUTURIX-FTA Irradiation Experiment

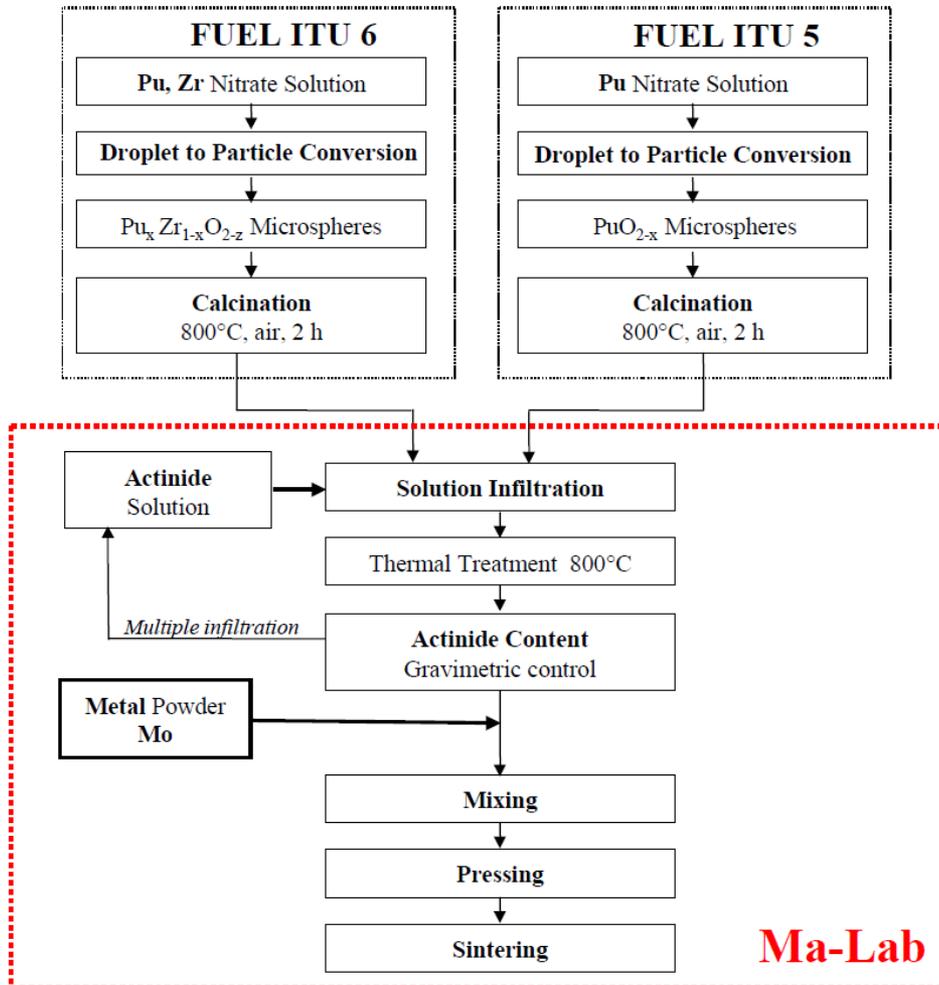
- **Title:** FUels for Transmutation of transURanium elements in phenIX / Fortes Teneurs en Actinides [high actinide content]
- **Objectives:** Comparison of irradiation behavior of oxides, nitrides & metallic MA-bearing fuels (U-free fuels for ADS and metallic low fertile fuels for SFR burner) in representative conditions -> 8 pins irradiated in PHÉNIX
- **Participants:** CEA, DOE, JAEA & JRC-Karlsruhe through bilateral agreements, FP-6 EUROTRANS (2005-2010) & FP-7 FAIRFUELS (2009-2015)
- **Key dates:**
 - Experimental grid: 2003
 - Transportations of fresh fuels from JRC-Karlsruhe and INL to ATALANTE facility: 2006
 - Irradiation in PHÉNIX: May 2007 till March 2009 for 235 EFPD (except nitrides)
 - Pins transportations from PHÉNIX to Cadarache (June 2013), INL (summer 2014), JRC-Karlsruhe (February 2015)
 - PIE for JRC-Karlsruhe & CEA pins firstly in FP-7 FAIRFUELS and in national programs since mid. 2015

FUTURIX-FTA Fuels

Pin	Fuel type	Composition ^[1]	Am content
DOE 1	Metal / low-fertile	35U-29Pu-4Am-2Np-30Zr (wt %)	0.5 g/cm ³
DOE 2	Metal / U-free	U 48Pu-12Am-40Zr (wt %)	1.2 g/cm ³
DOE 3	Nitride / low-fertile	(U _{0.5} ,Pu _{0.25} ,Am _{0.15} ,Np _{0.10})N	1.7 g/cm ³
DOE 4	Nitride / U-free	(Pu _{0.21} ,Am _{0.21} ,Zr _{0.58})N	2.7 g/cm ³
ITU 5	Cer-Met / U-free	(Pu_{0.8},Am_{0.2})O₂ + 86 vol% Mo	0.3 g/cm³
ITU 6	Cer-Met / U-free	(Pu_{0.23},Am_{0.25},Zr_{0.52})O₂ + 60 vol% Mo	1.0 g/cm³
CEA 7	Cer-Cer / U-free	(Pu _{0.5} ,Am _{0.5})O ₂ + 80 vol% MgO	1.0 g/cm ³
CEA 8	Cer-Cer / U-free	(Pu _{0.2} ,Am _{0.8})O ₂ + 75 vol% MgO	2.0 g/cm ³

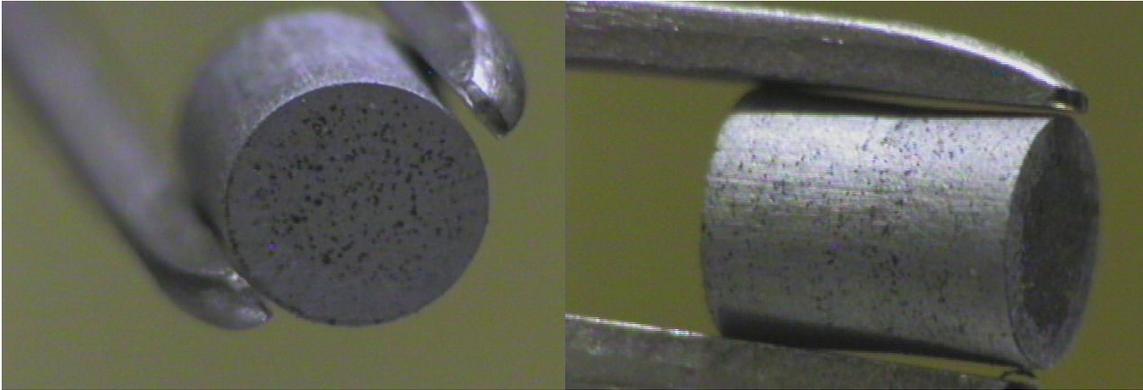
PIE on CEA 7 & 8 was already presented at Global 2015^[2]. PIE results on pins ITU 5 & 6 are newly presented^[3].

Fuel Preparation – FX 5 & 6

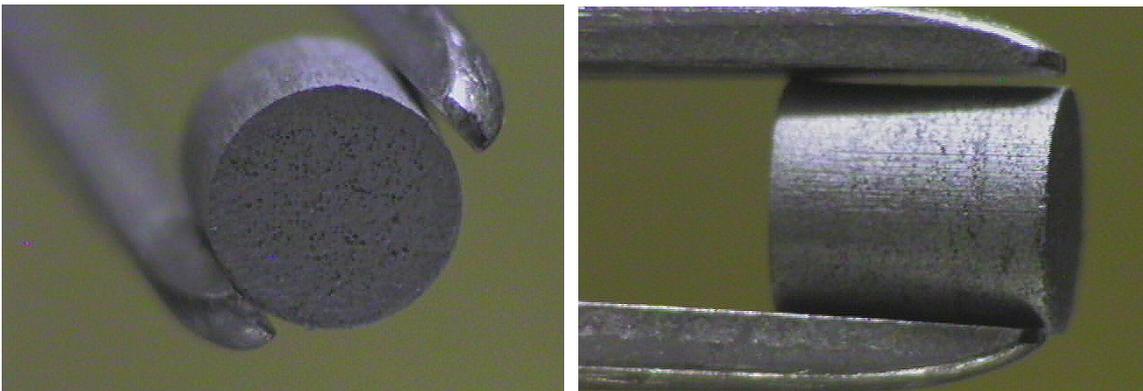


- Particle synthesis by external gelation
- Infiltration of Am into porous $(\text{Pu,Zr})\text{O}_{2-x}$ & PuO_{2-x} particles
- Mixing of fuel particles and Molybdenum matrix by conventional powder blending
- Bi-axial pressing (500 MPa)
- Sintering (1600°C, 8h)

Visual Aspect of ITU 5 & 6

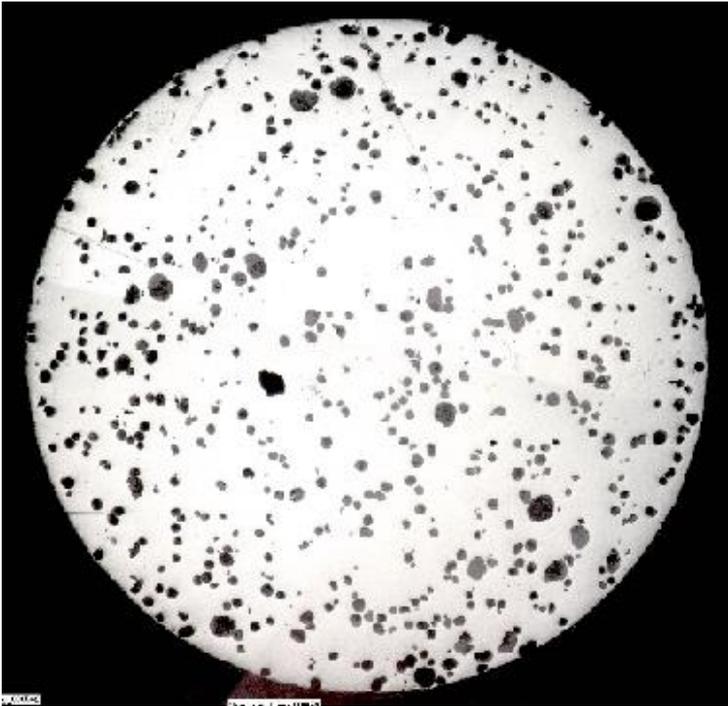


ITU 5
86% Mo

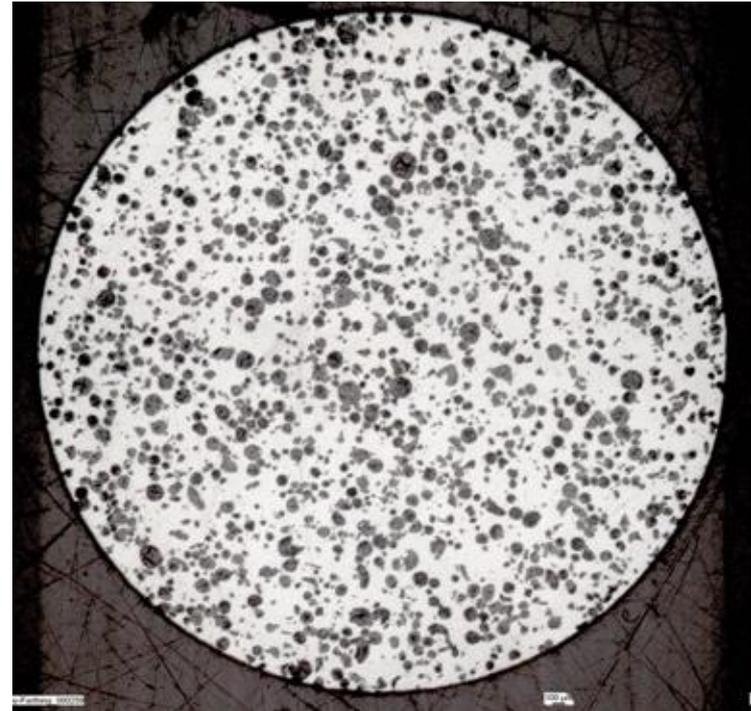


ITU 6
60% Mo

Ceramographs

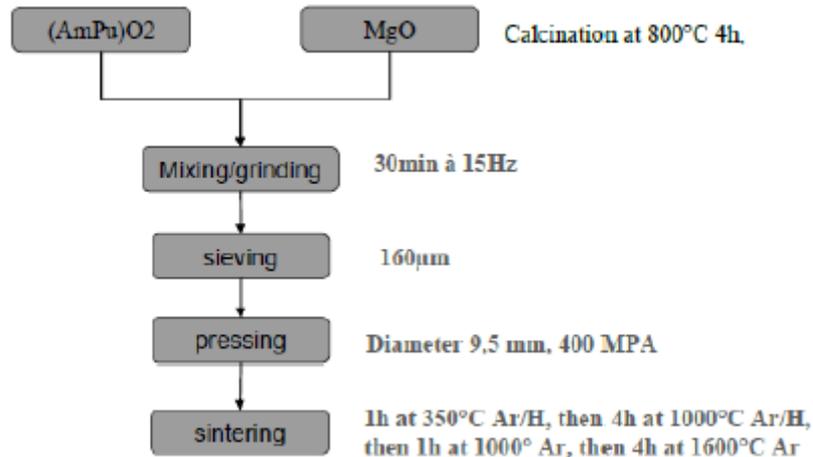


ITU 5
86% Mo

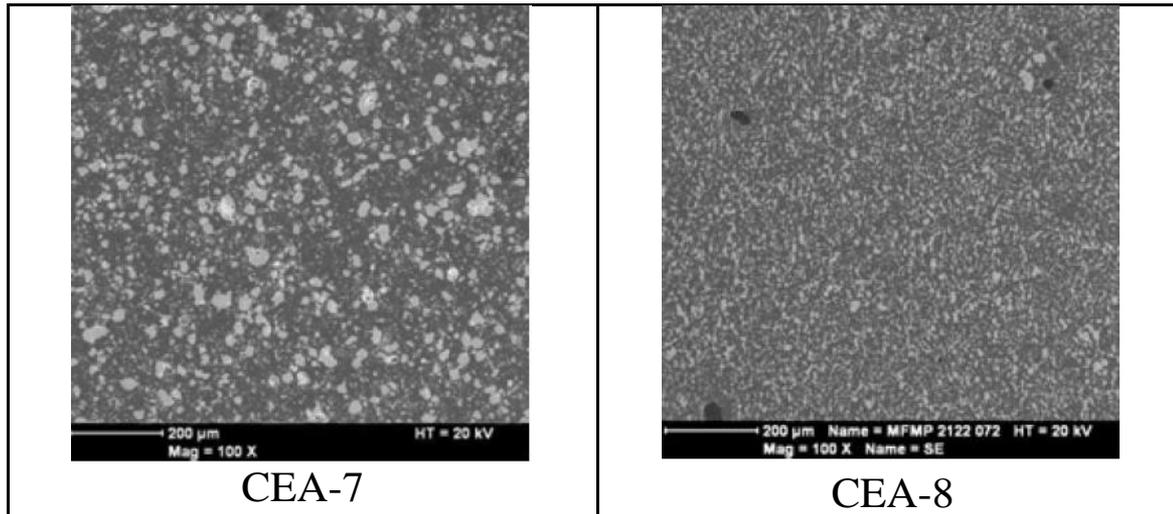


ITU 6
60% Mo

Fuel Preparation – FX 7 & 8



- Preparation in shielded cells (ATALANTE facility)
- Synthesis of (Pu,Am)O_{2-x} particles by oxalic co-conversion
- Particles dispersion into inert magnesia matrix by conventional powder metallurgy process
- Sintering

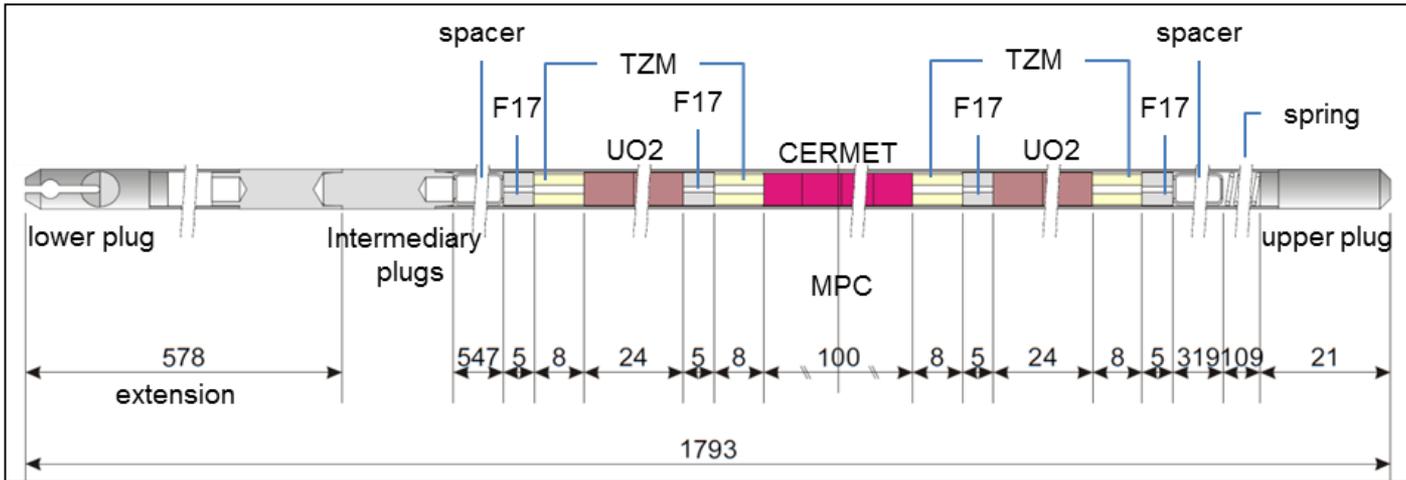


Fuel Characteristics

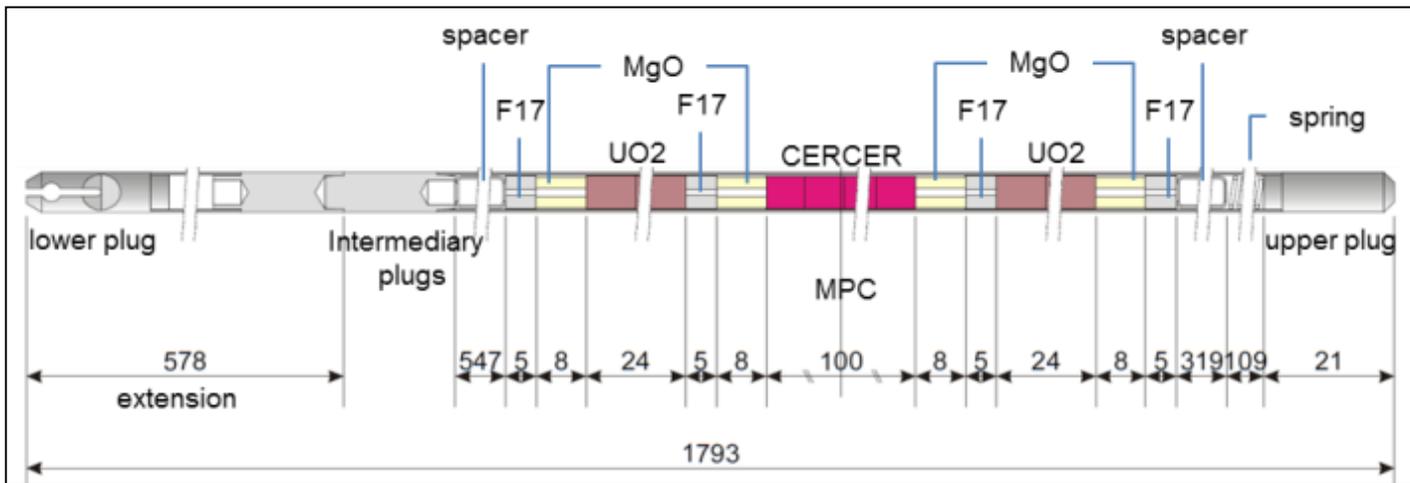
Fuel ID	ITU-5	ITU-6	CEA 7	CEA 8
Pu content (wt%)	10.17	9.40	19.9	9.1
Am content (wt%)	2.68	10.33	19.6	35.6
Mean diameter (mm)	5.18	5.19	5.19	~5.14
Average Height (mm)	6.00	5.80	6.66	6.49
Density (g.cm⁻³)	10.37	9.50	4.88	5.12
Total porosity (%)	4.1	4.8	4.5	7.5
Open porosity (%)	0.5-0.6	0.7-0.8	0.3*	0.4*
O/M (particle)	1.896	1.786	1.88	1.73
Particle size (µm)	< 225	< 225	< 50	< 50
Particle fraction	14 (volume %)	40 (volume %)	20 (volume %)	25 (volume %)

*estimated

FUTURIX CerMet & CerCer Fuel Pins



CerMet
ITU 5&6



CerCer
CEA 7&8

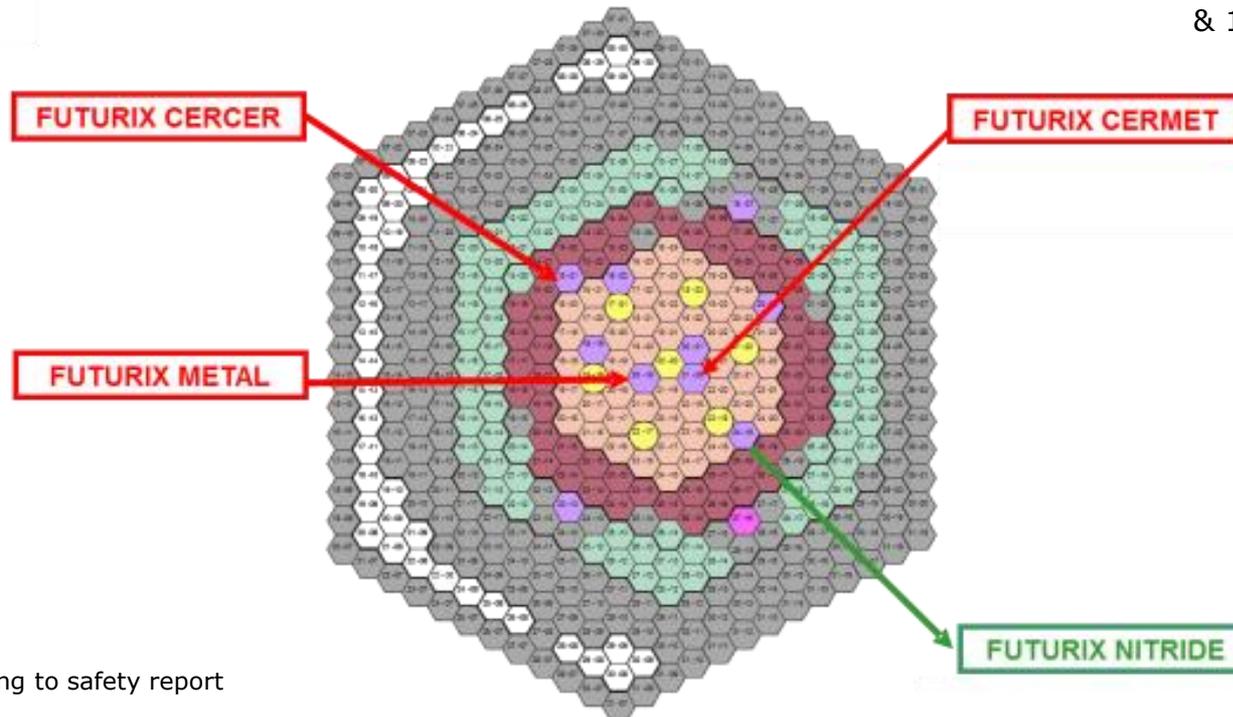
FUTURIX-FTA Position in Phénix Core

CERCER (CEA 7&8)

- Cumulated fluence: $0.93 \times 10^{23} \text{ n.cm}^{-2}$
- Fluence $>0.1 \text{ MeV}$: $0.55 \times 10^{23} \text{ n.cm}^{-2}$
- Max. dose: 28.7 dpa
- Max. temperature*: 1430°C (CEA 7) & 1270°C (CEA 8)

CERMET (ITU 5&6)

- Cumulated fluence: $1.46 \times 10^{23} \text{ n.cm}^{-2}$
- Fluence $>0.1 \text{ MeV}$: $0.88 \times 10^{23} \text{ n.cm}^{-2}$
- Max. dose: 38.6 dpa
- Max. temperature*: 1580°C (ITU 5) & 1480°C (ITU 6)

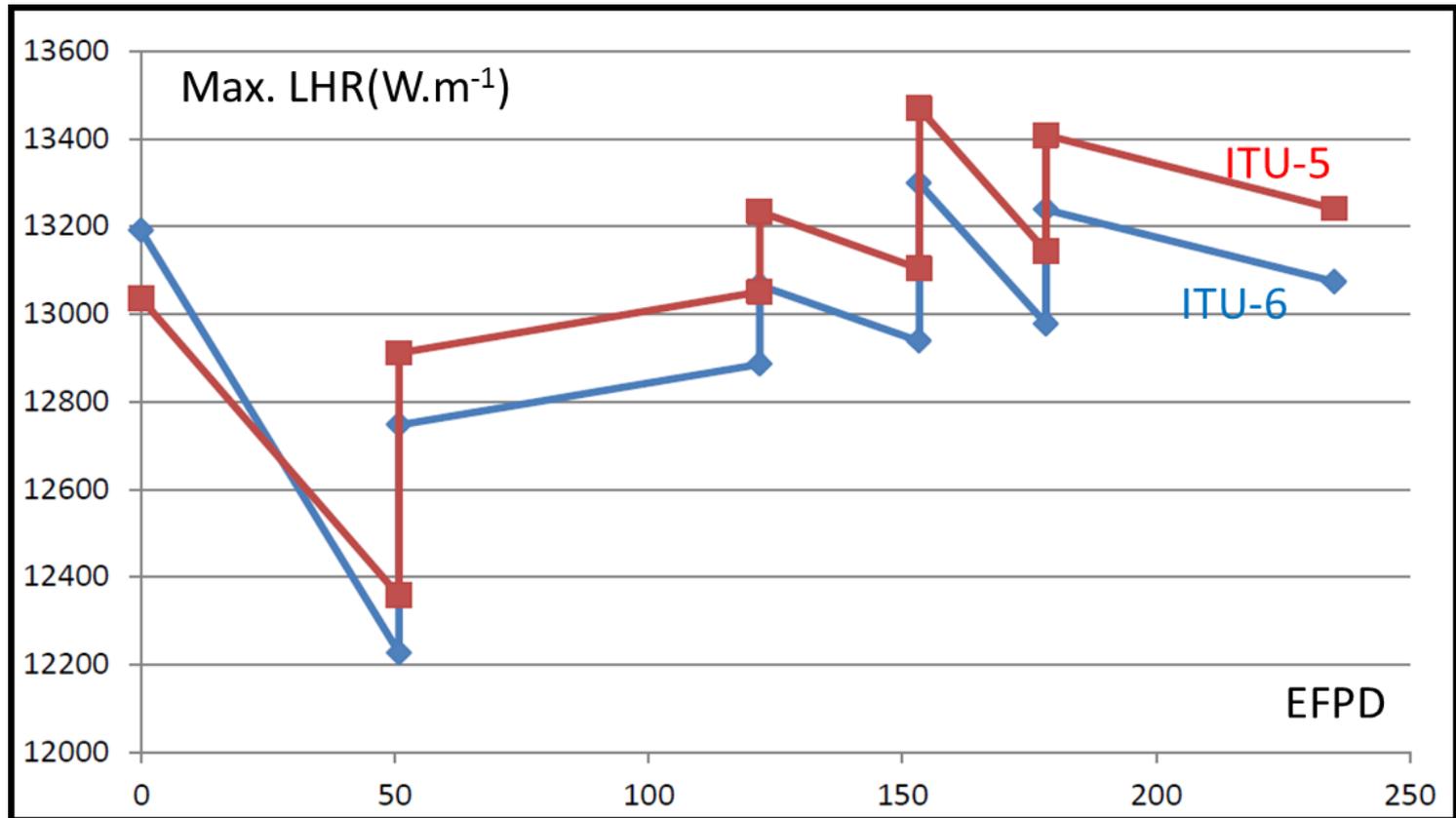


*according to safety report

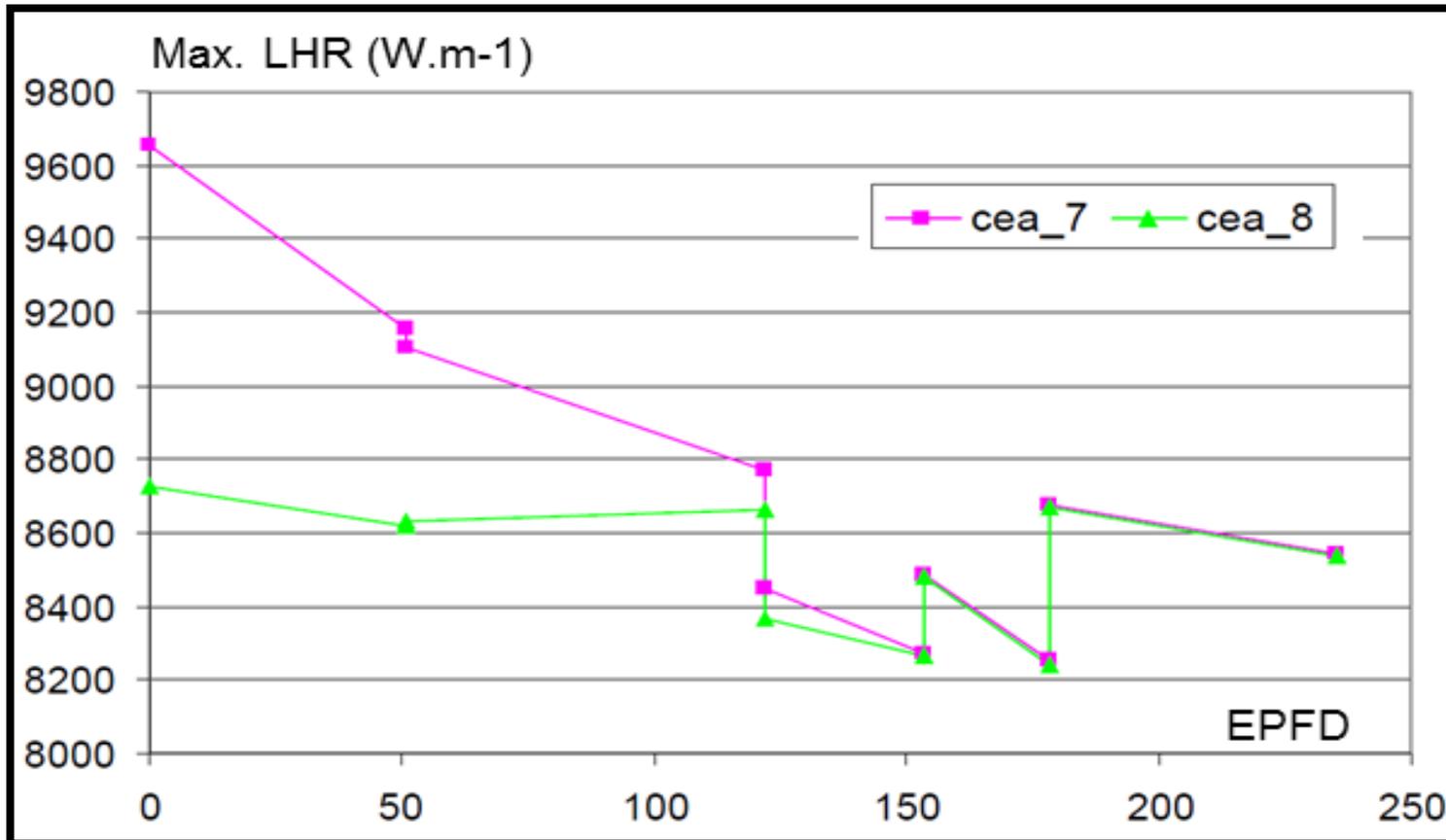
FUTURIX-FTA Irradiation Conditions

Pin	Burn-up (at%)	Transmutation rate (at%)	Linear Heat Rate (W/cm)	Neutron fluence	Dose (dpa)
ITU 5	17.81	27.0	122 - 133	14.59 x 10^{26} n/m ²	38.58
ITU 6	12.88	27.1	124 - 135		
CEA 7	8.6	17.6	82 - 96	9.33 x 10^{26} n/m ²	28.69
CEA 8	5.75	19.8	82 - 87		

Power Evolution ITU 5 & 6



Power Evolution CEA 7 & 8



Post Irradiation Examinations

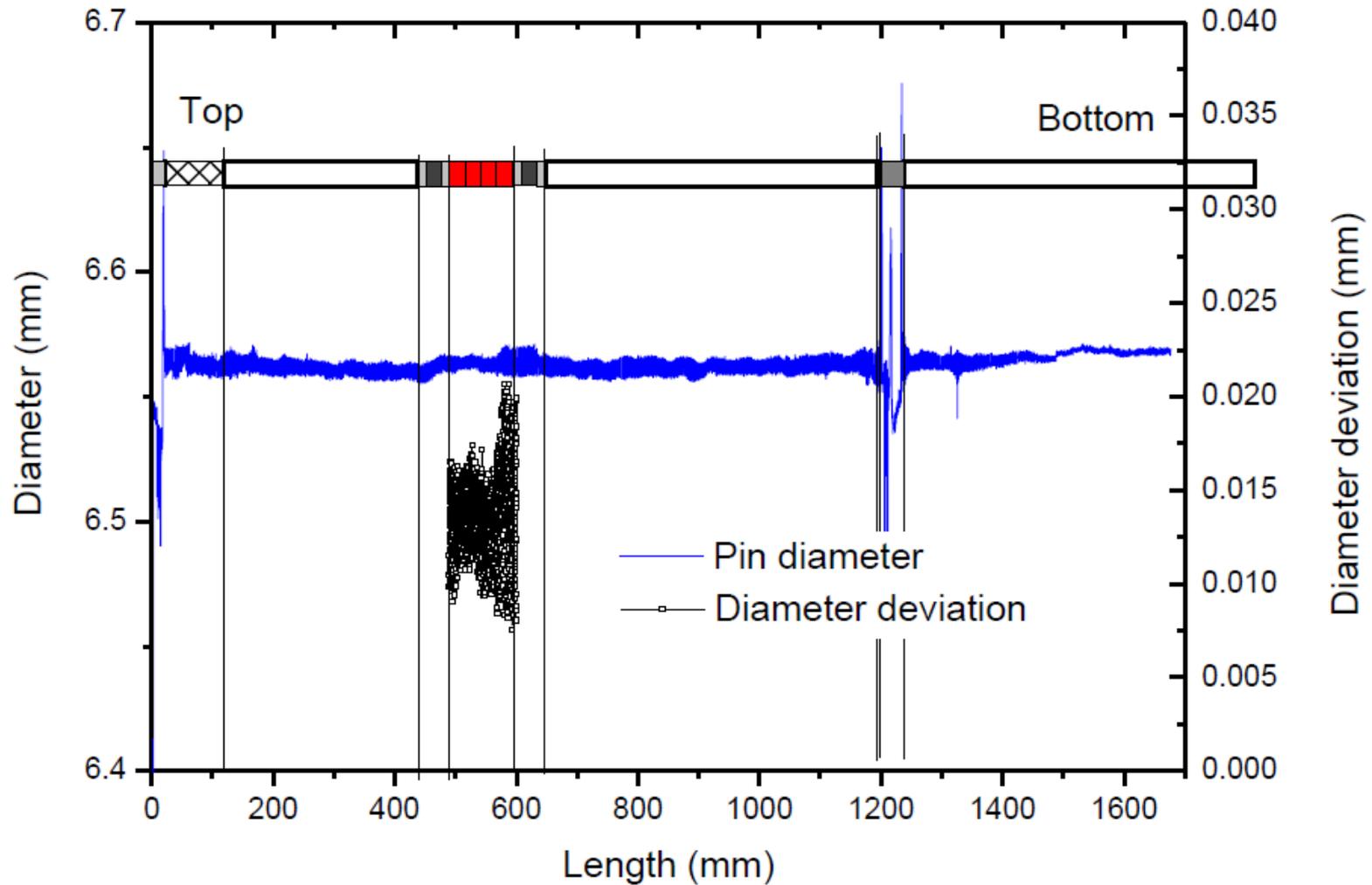
- CerMet Fuel (ITU 5 & 6): JRC-ITU hot cells
 - **Pin length & profilometry** ✓
 - **Gamma spectrometry** ✓
 - **Gas puncturing** ✓
 - **Ceramography** ✓
 - **Pellet Metrology and Hydrostatic Density (ongoing)**
 - **SEM, EPMA (to be done)**
 - **LaserFlash (to be done)**
- CerCer Fuel (CEA 7 & 8): LECA facility (CEA-Cadarache)
 - **Pin length & profilometry** ✓
 - **Gamma spectrometry** ✓
 - **Gas puncturing** ✓
 - **Pellet Metrology and Hydrostatic Density** ✓
 - **Ceramography** ✓
 - **SEM, EPMA (to be done)**

Pin Length Measurement & Profilometry showing no significant changes

FUTURIX-FTA Pins:	ITU-5	ITU-6	CEA-7	CEA-8
Length before irradiation (mm):	1793	1793	1793	1793
Length after irradiation (mm):	1793.0	1793.1	1792.2	1792.0

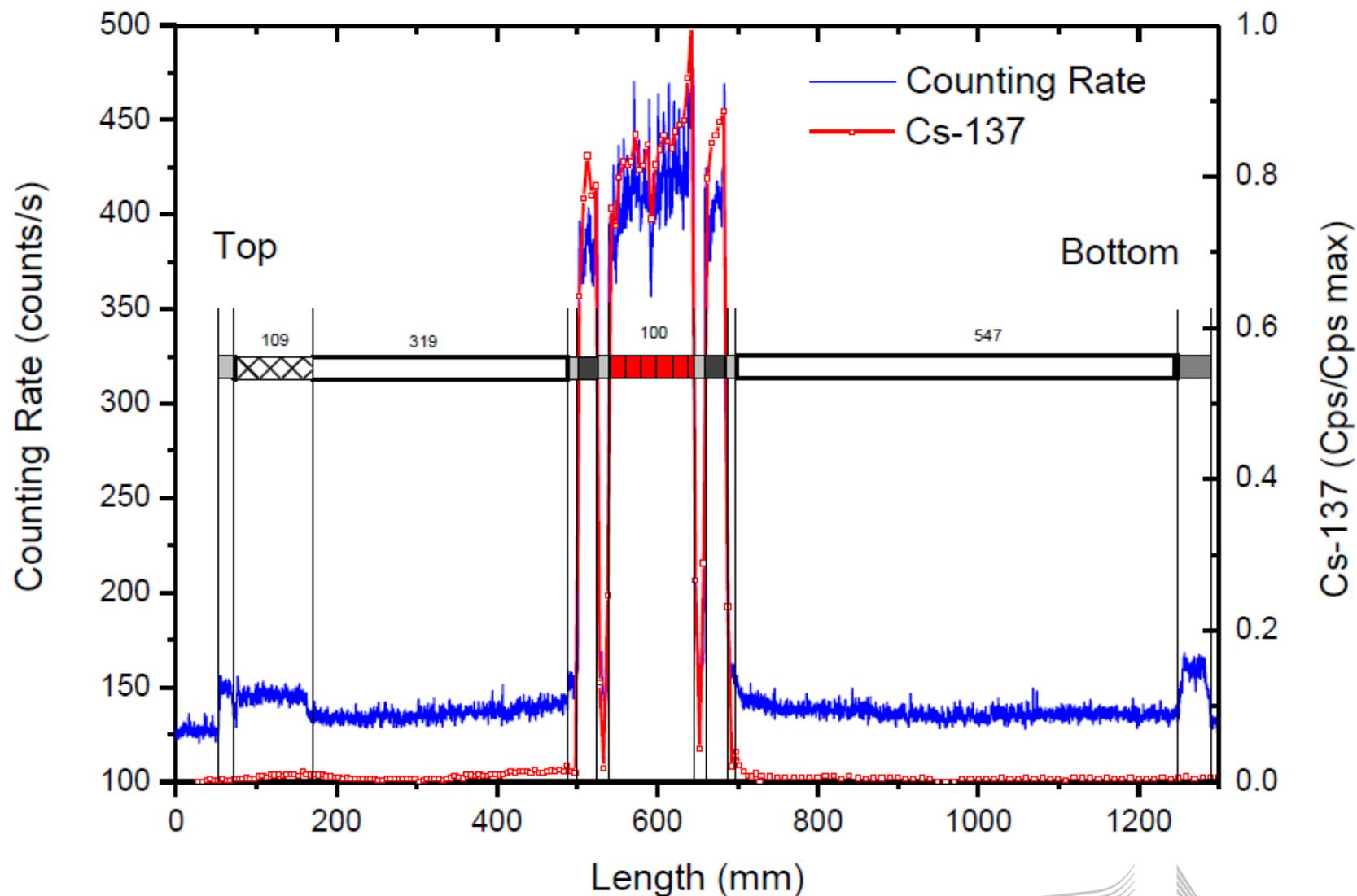
FUTURIX-FTA Pins:	ITU-5	ITU-6	CEA-7	CEA-8
Maximum diameter at experimental column after irradiation (mm)	6.571	6.558	6.562	6.557
Change in % (relative to 6.55 mm)	0.3	0.1	0.2	0.1

Profilometry ITU-5



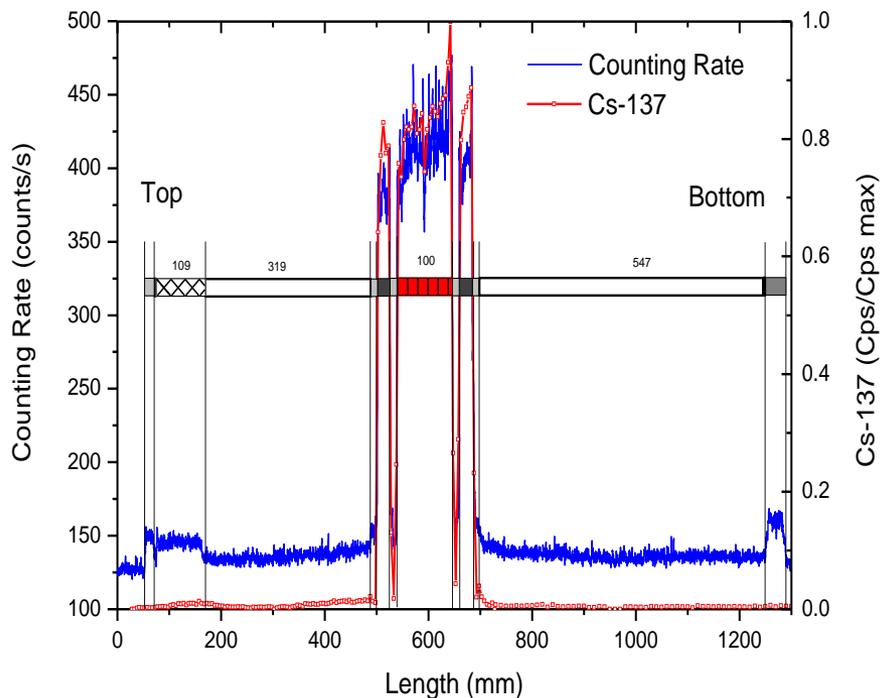
Gamma spectrometry

Gamma Spectrometry of ITU-5 (^{137}Cs)

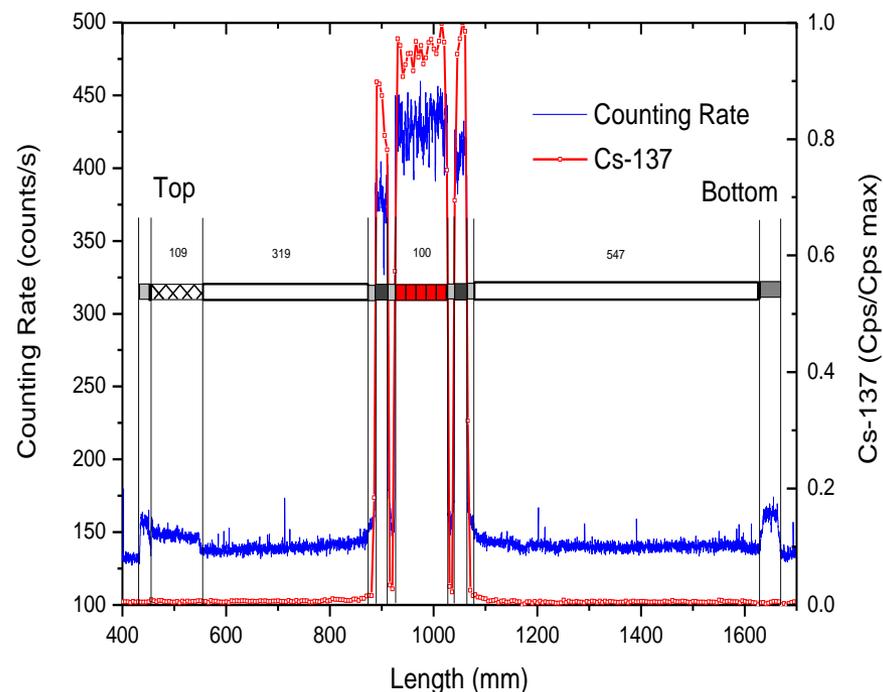


Gamma Spectrometry of ITU 5&6 (^{137}Cs)

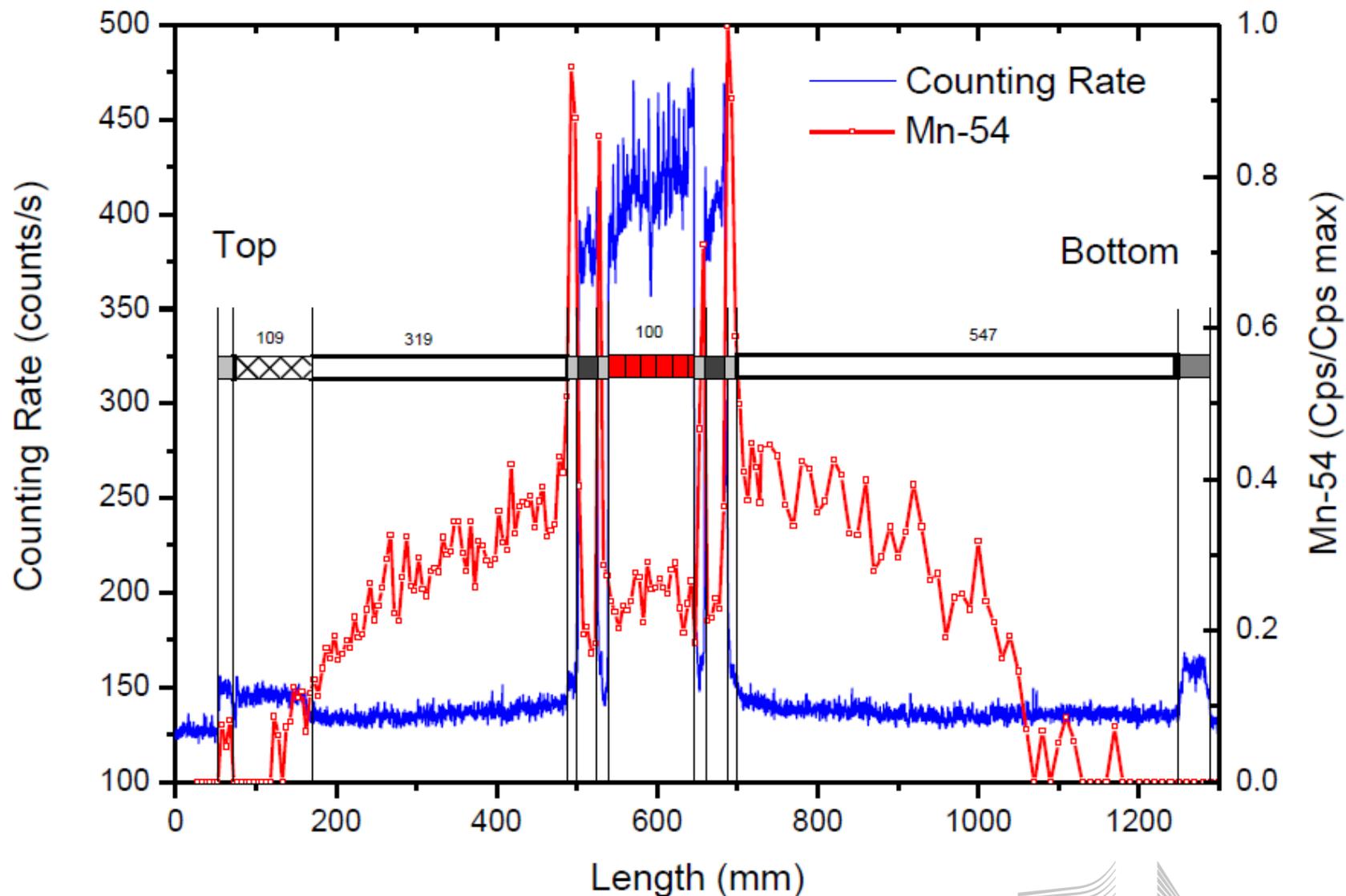
Gamma scanning of pin 5
(total & Cs-137)



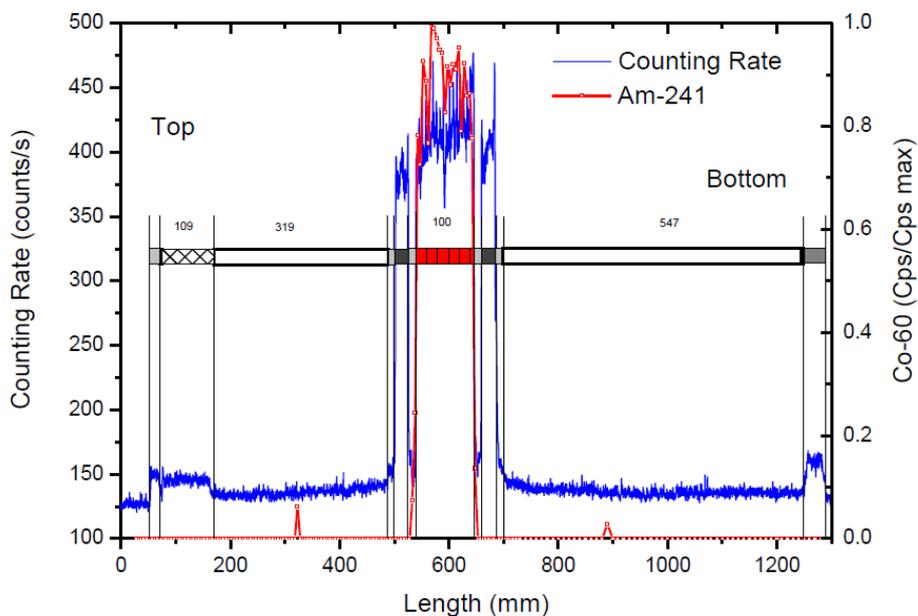
Gamma scanning of pin 6
(total & Cs-137)



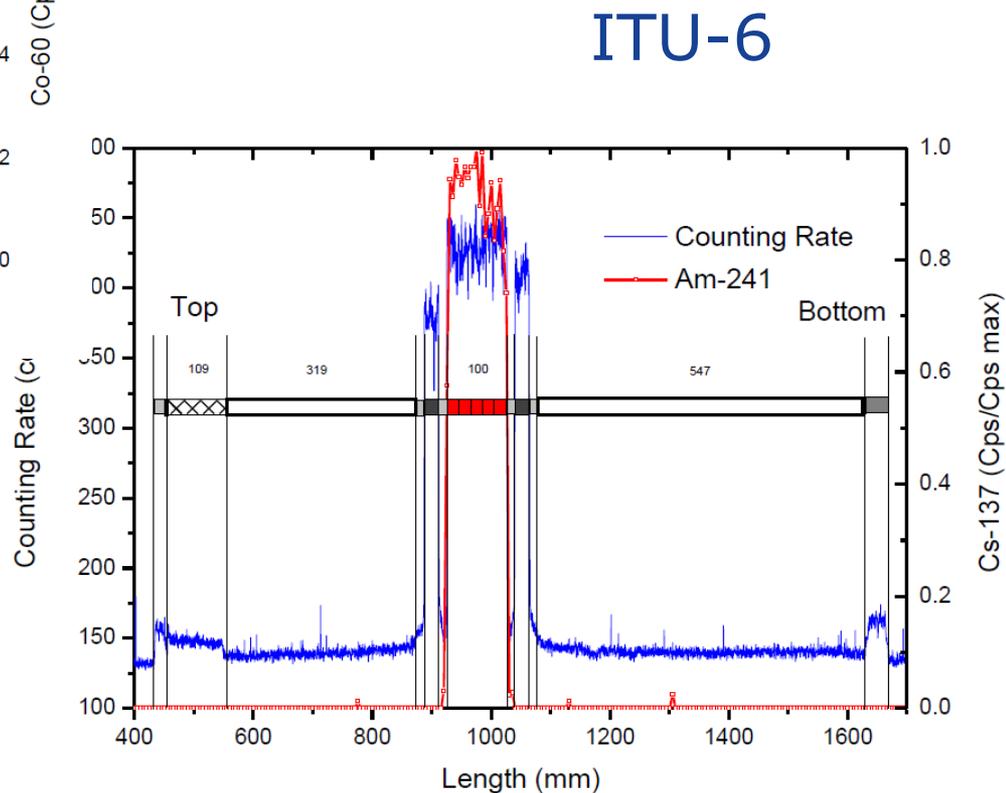
Gamma Spectrometry of ITU-5 (^{54}Mn)



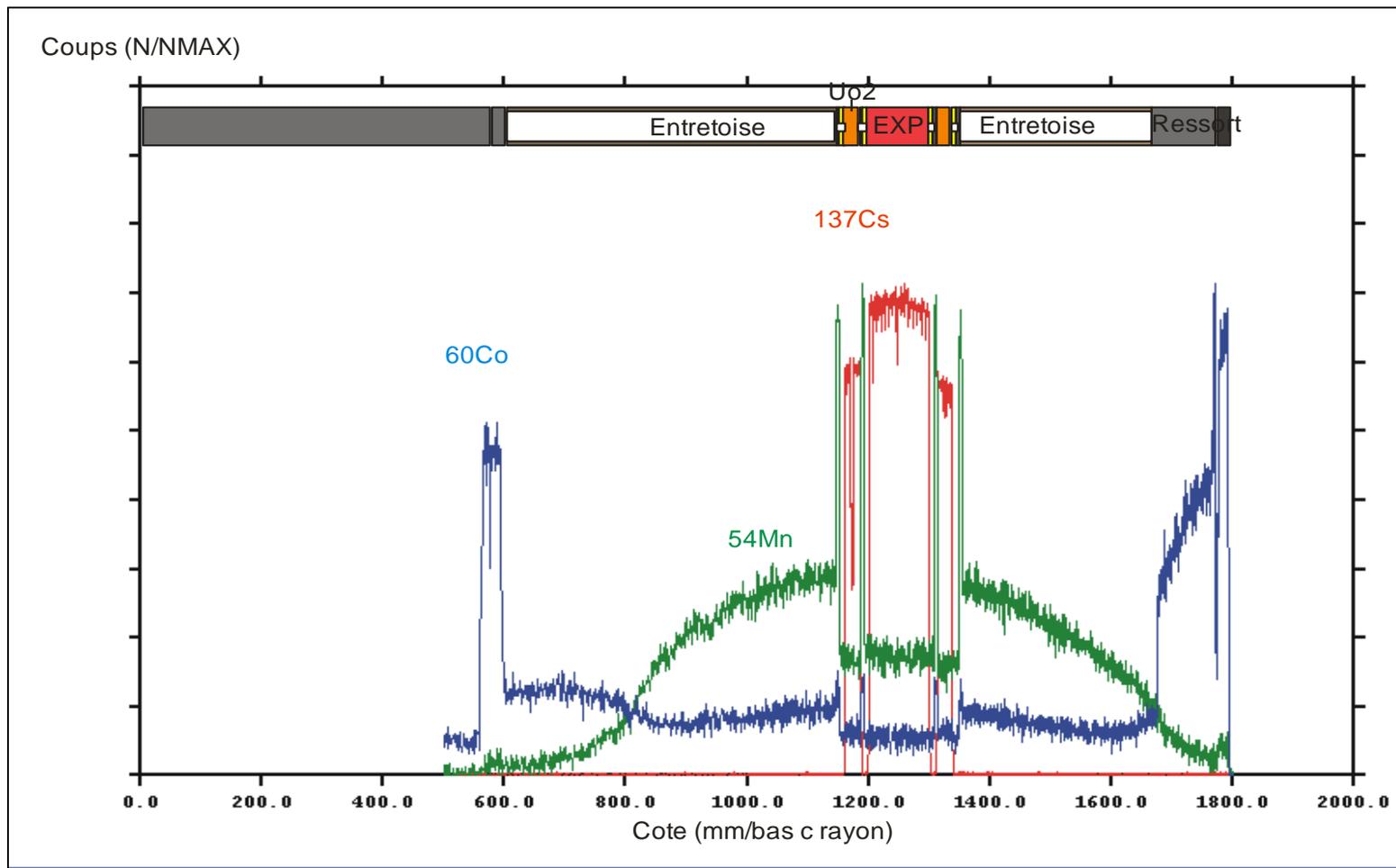
Gamma Spectrometry of ITU 5&6 (^{241}Am)



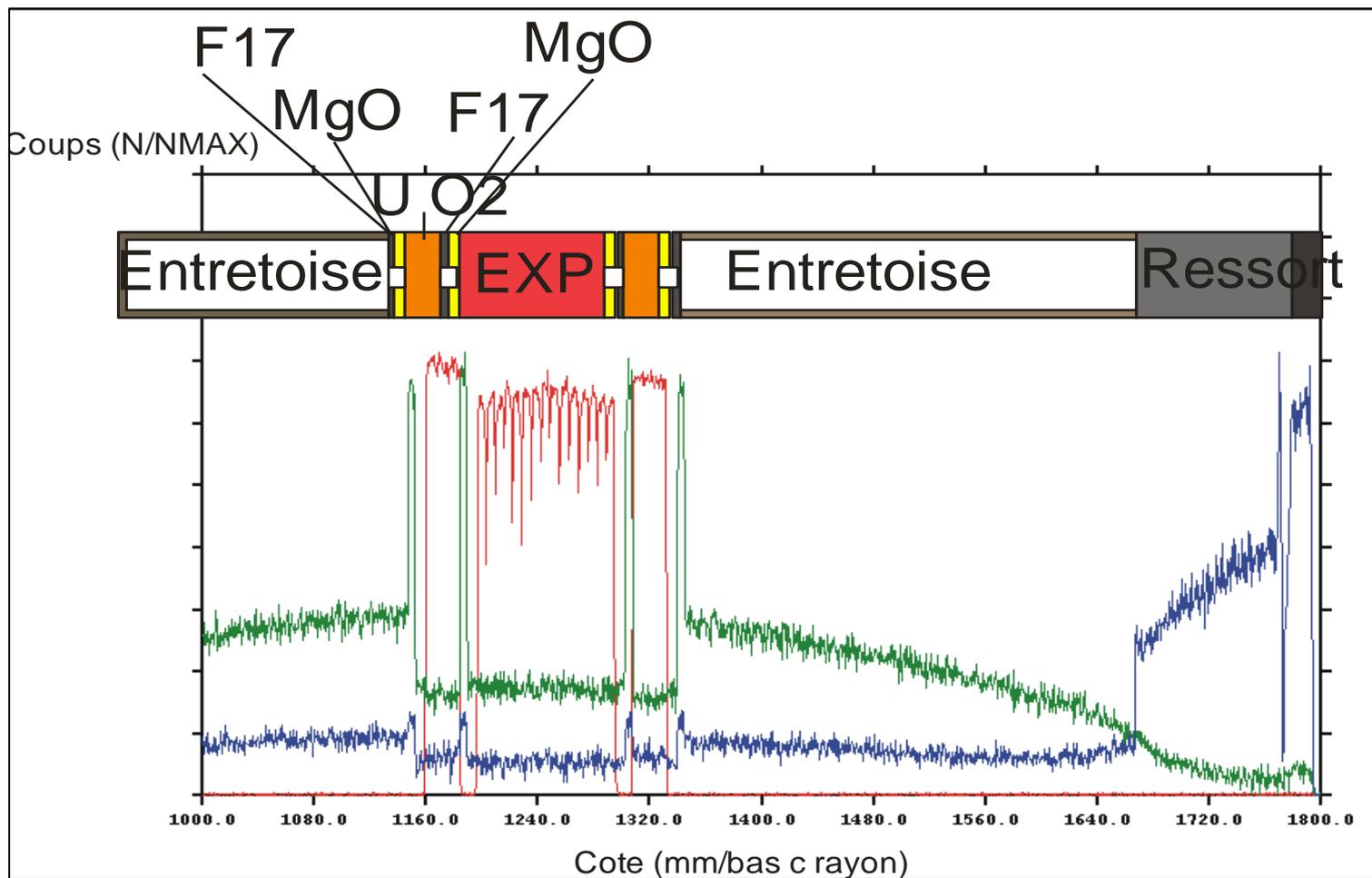
ITU-5



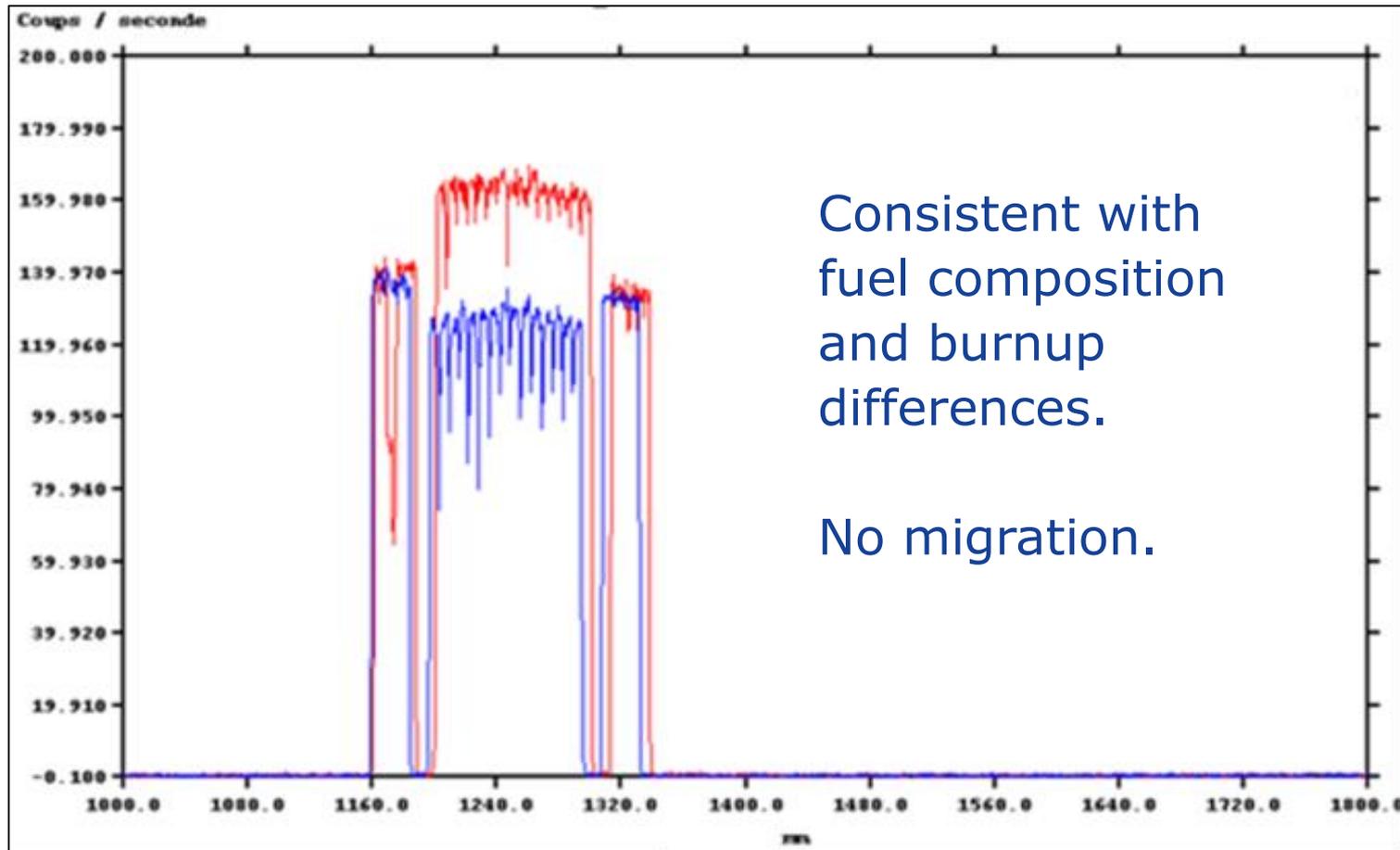
Gamma Spectrometry of CEA 7 (^{137}Cs (red), ^{54}Mn (green) and ^{60}Co (blue))



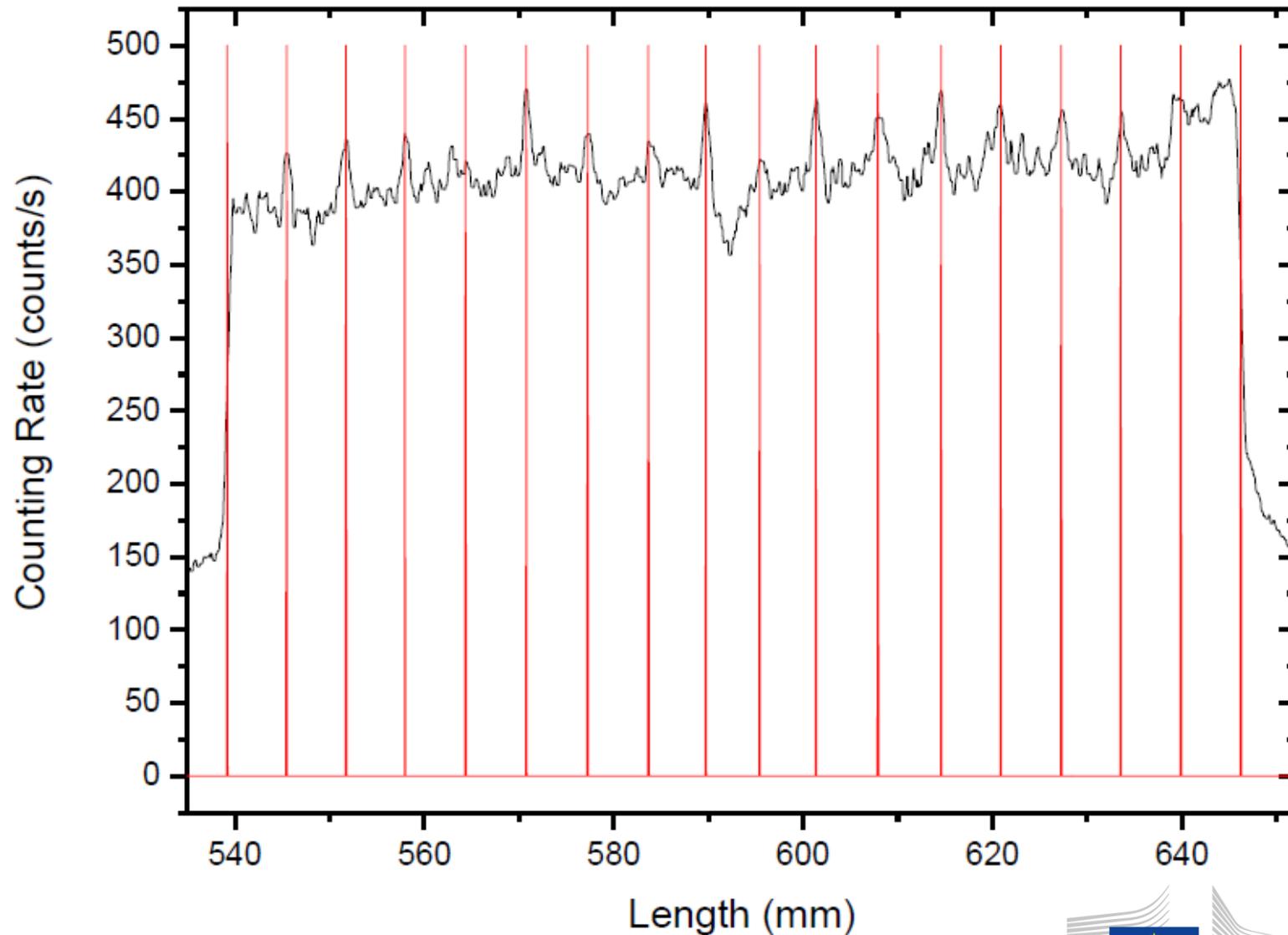
Gamma spectrometry of CEA 8 (^{137}Cs (red), ^{54}Mn (green) and ^{60}Co (blue))



Comparison of the ^{137}Cs distribution between pins CEA 7 (blue) and CEA 8 (red)



Determination of Pellet Positions ITU-5



Estimation of Pellet Elongation from Gamma Spectrometry for ITU-5

Pellet No.	Measured length (mm)	Original length (mm)	Change (%)
1	6.28	6.15	2.05
2	6.28	5.89	6.55
3	6.21	6.04	2.80
4	6.41	6.07	5.59
5	6.41	6.16	4.05
6	6.48	6.04	7.22
7	6.41	6.14	4.39
8	6.08	5.7	6.59
9	5.67	5.5	3.18
10	5.94	5.62	5.73
11	6.54	6.2	5.53
12	6.68	6.24	6.99
13	6.28	6.04	3.91
14	6.41	6.17	3.88
15	6.28	6.01	4.42
16	6.34	6.04	5.01
17	6.34	6.06	4.66
Total column:	107.02	102.07	4.85

Estimation of Pellet Swelling from Gamma Spectrometry

Pin ID:	ITU 5	ITU 6	CEA 7	CEA 8
Fuel column length before irradiation (mm):	102.07	98.68	99.21	97.35
Fuel column length after irradiation (mm):	107.02	101.70	100.1	98.0
Relative elongation (%):	4.85	3.06	0.9	0.7
Equivalent to volume swelling of (%)*:	15.3	9.5	2.7	2.1

Results indicate that there might have been no gap closure (together with profilometry).

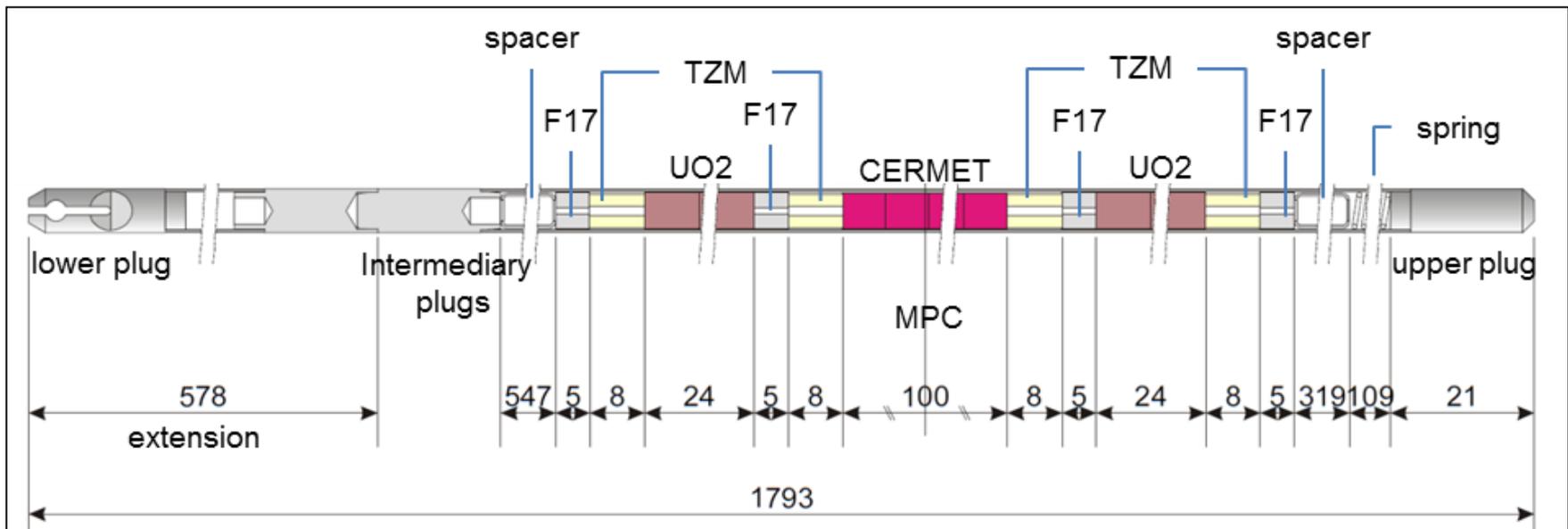
Gas Puncturing Results

Low Fractional Releases in Both Pins

		He(mol)	Kr(mol)	Xe(mol)	Total (mol)
ITU-5	produced	4.68×10^{-4}	7.00×10^{-5}	7.32×10^{-4}	1.27×10^{-3}
	FR (%)	43.23	33.40	22.44	31.06
ITU-6	produced	1.55×10^{-3}	6.95×10^{-5}	7.20×10^{-4}	2.34×10^{-3}
	FR (%)	35.87	43.17	21.67	30.10
CEA-7	produced	1.20×10^{-3}	4.93×10^{-5}	5.04×10^{-4}	1.75×10^{-3}
	FR (%)	~0	0.75		<1
CEA-8	produced	2.32×10^{-3}	4.42×10^{-5}	4.25×10^{-4}	2.79×10^{-3}
	FR (%)	~0	0.22		<1

Recovery of CerMet Pellets

- All pellets were found loose inside pin, no indication of gap closure
- All pellets were completely intact and could be extracted
- Confirmation of gamma spectrometry

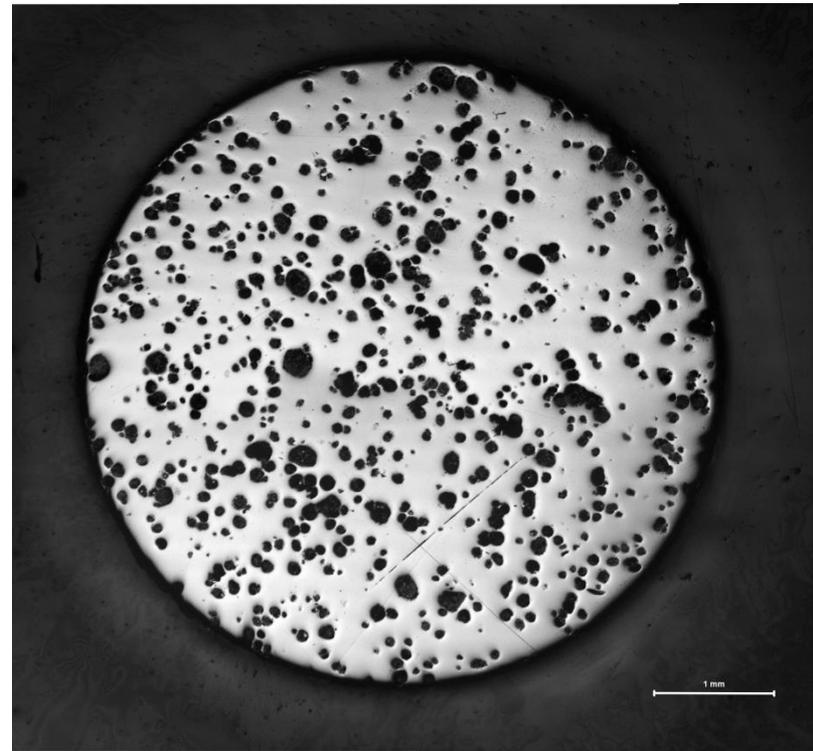


Ceramography of ITU 5 ($\text{Pu}_{0.8}\text{Am}_{0.2}\text{O}_{1.9}$ + 86 vol% Mo)

Fresh fuel



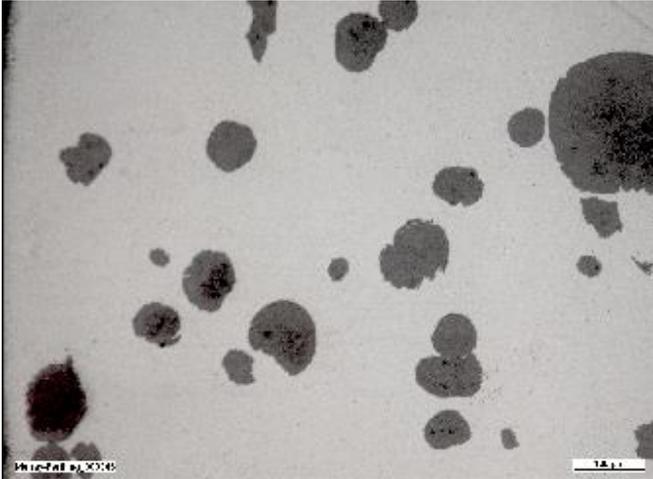
Irradiated fuel



- No cracks in Mo matrix
- Particle at pellet boundary formed open porosity -> gas release

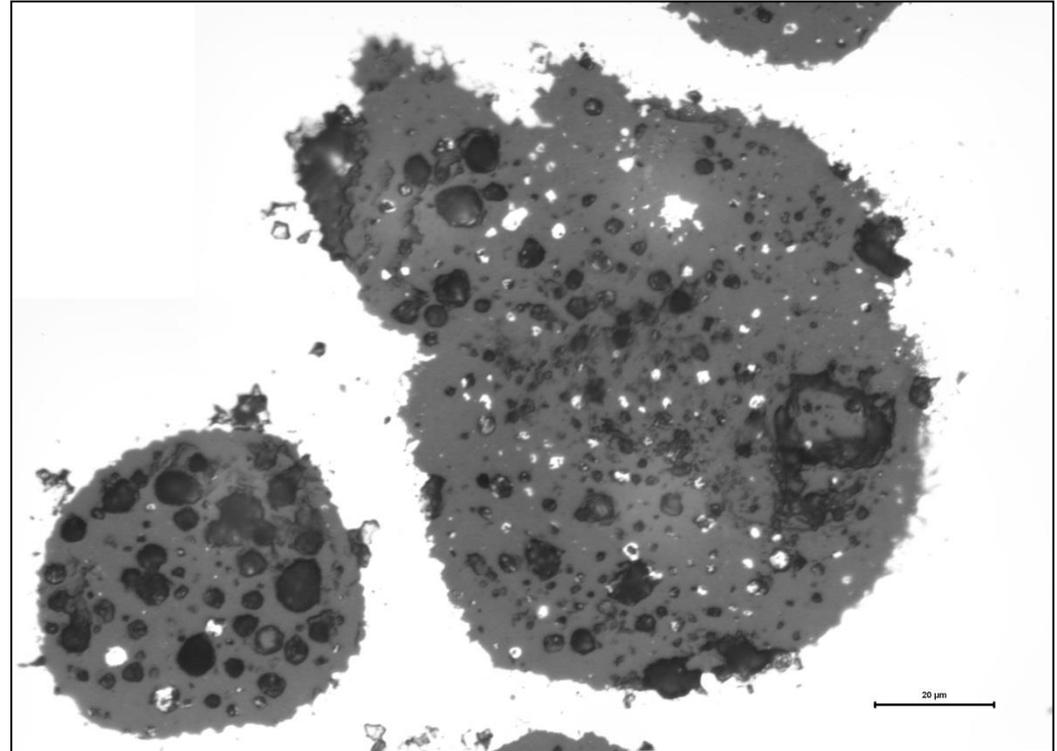
Ceramography of ITU 5 ($\text{Pu}_{0.8}\text{Am}_{0.2}\text{O}_{1.9}$ + 86 vol% Mo)

Fresh fuel



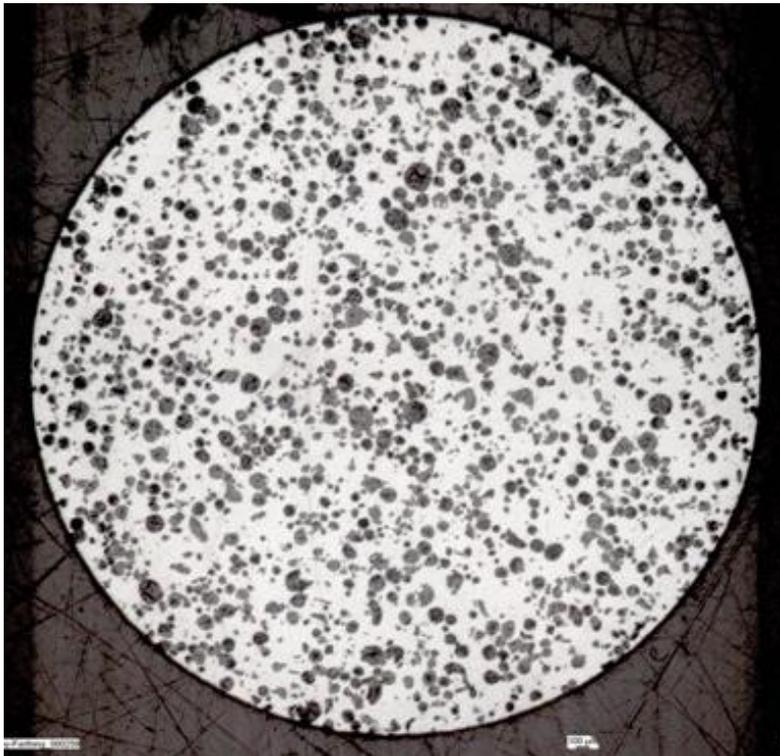
- No cracks in Mo matrix
- Metallic precipitates in particles
- Gas bubbles mainly located in particles
- "Finger"-like particle-matrix transition
- Halo around particles

Irradiated fuel

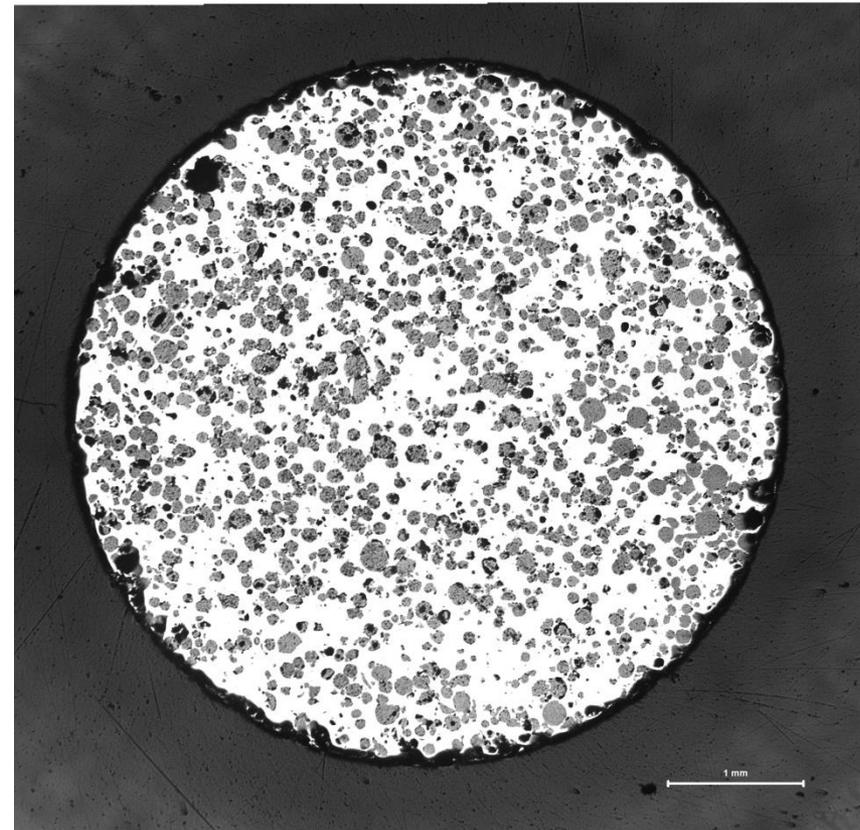


Ceramography of ITU 6 ($\text{Pu}_{0.23}\text{Am}_{0.2}\text{Zr}_{0.52}\text{O}_{1.8}$ + 60 vol% Mo)

Fresh fuel



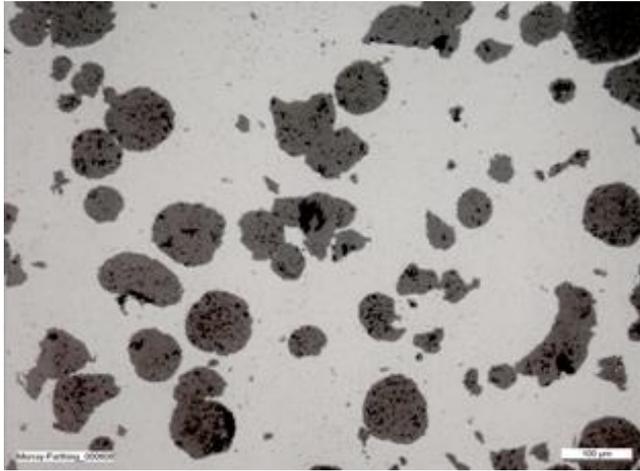
Irradiated fuel



- No cracks in Mo matrix
- Particle at pellet boundary formed open porosity -> gas release

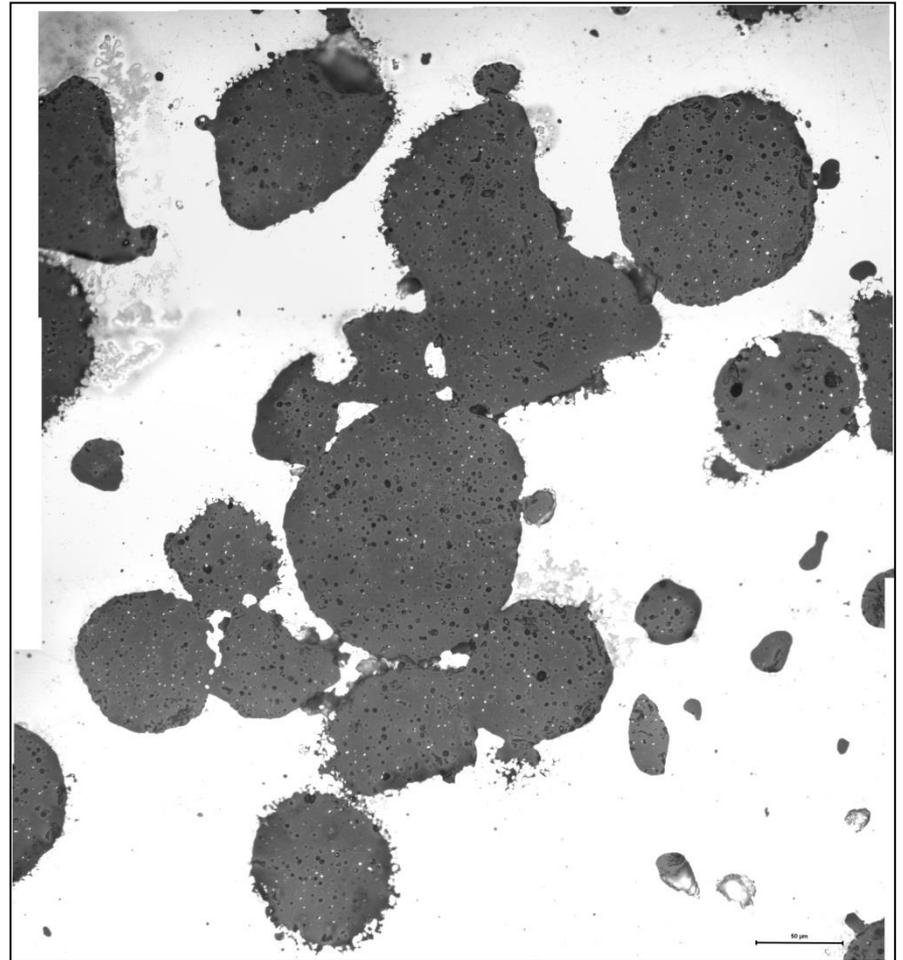
Ceramography of ITU 6

Fresh fuel



- No cracks in Mo matrix
- Metallic precipitates in particles
- Gas bubbles mainly located in particles
- "Finger"-like particle-matrix transition
- Halo around particles

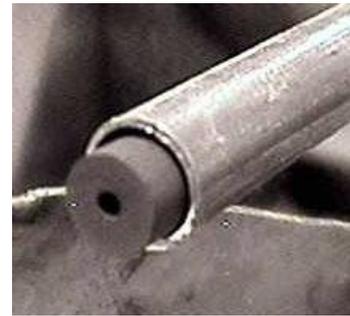
Irradiated fuel



Recovery of CerCer Pellets

- Pellets were found loose inside pin, no indication of gap closure
- CEA 7
 - **8 of 15 pellets recovered in one piece**
- CEA 8
 - **5 of 15 recovered in one piece**
- Other pellets were extracted in relatively large fragments

MgO pellet being extracted (a) and F17 spacer (b), from pin CEA-8

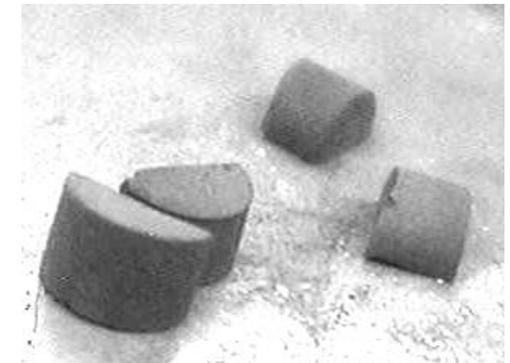


a



b

extracted Am-bearing CERCER fuels from CEA-7



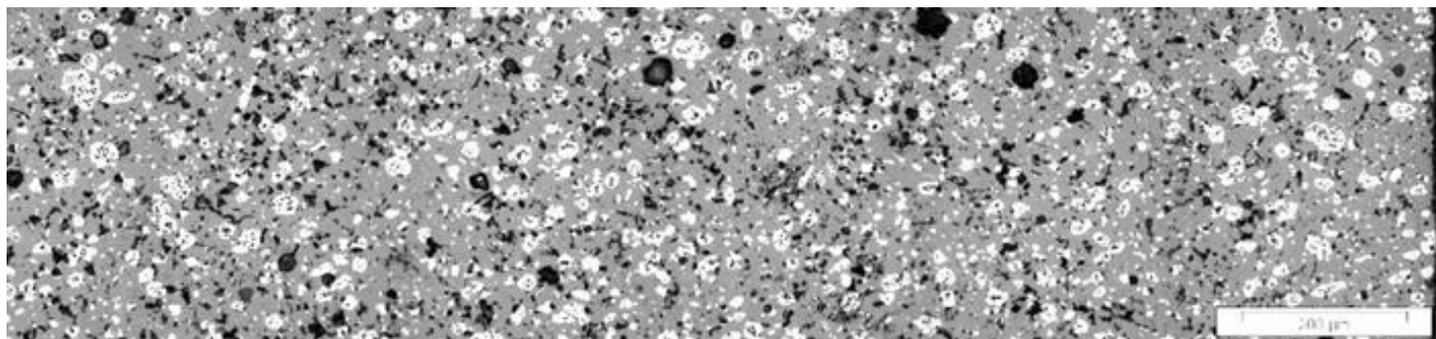
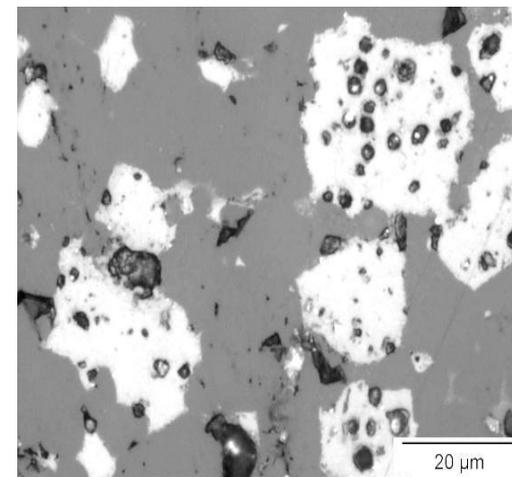
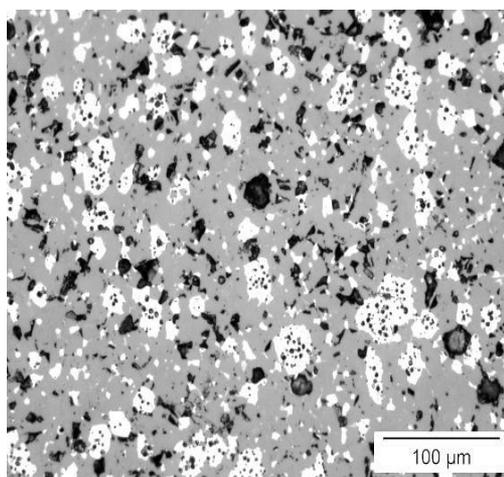
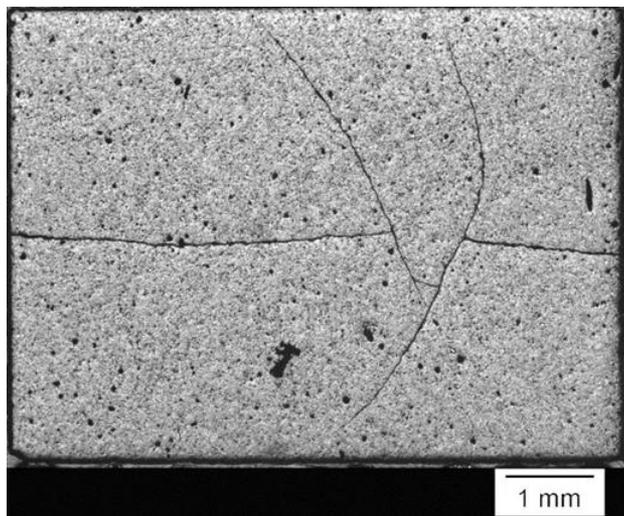
Pellet Metrology of CEA 7 & 8

Target #	Δ Height (%)	Δ Diameter (%)	Δ Geometrical density (%)
Futurix FTA 7			
5114	0,7	1,5	-3,6
5119	0,6	1,3	-3,1
5120	0,6	1,5	-3,4
5121	1,2	1,2	-3,6
5126	0,8	1,3	-3,3
5127	0,7	0,9	-2,5
5128	0,9	1,3	-3,4
5129	0,7	1,1	-2,8
Futurix FTA 8			
2112	1,1	1,1	-3,2
2113	0,9	1,2	-3,2
2117	0,7	1,4	-3,5
2124	0,8	0,7	-2,2
2120	0,3	1,5	-3,2

Consistent with results from gamma spectrometry, indicating circa 3% volume swelling.

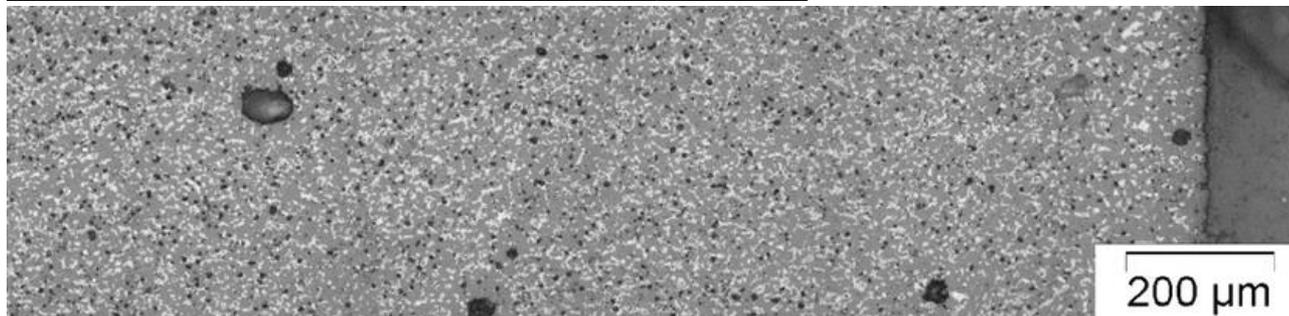
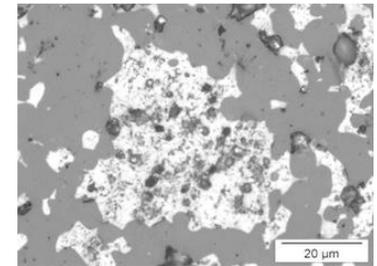
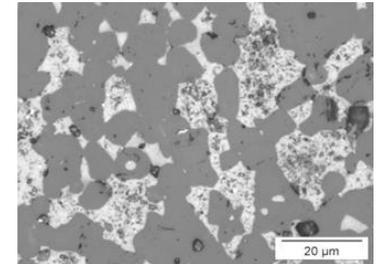
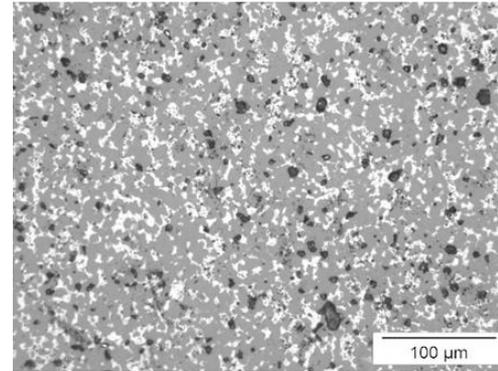
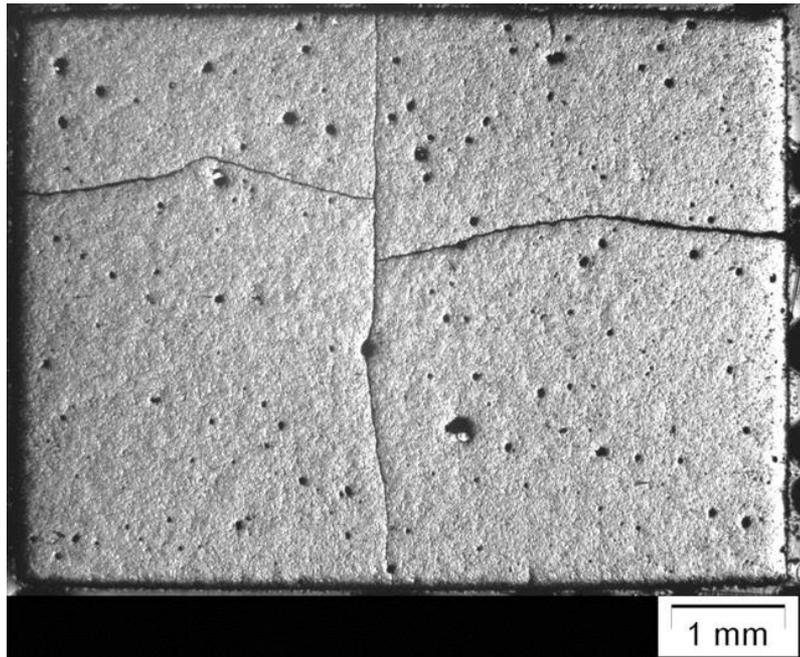
Ceramography of CEA 7 ($\text{Pu}_{0.5}\text{Am}_{0.5}\text{O}_{1.88} + 80\text{vol}\% \text{MgO}$)

No significant evolution of the diameter of the pellets along the cuts; rounded shaped bubbles inside $(\text{Pu},\text{Am})\text{O}_{2-x}$ particles that probably host gases.



- > some cracks (consistent with broken pellets shapes)
- > metallic precipitates
- > gas bubbles mainly located in particles (consistent with low gas release rate)
- > secondary phase at MgO grain boundaries

Ceramography of CEA 8 ($\text{Pu}_{0.2}\text{Am}_{0.8}\text{O}_{1.73}$ + 75 vol% MgO)



few cracks

- > gas bubbles mainly located in particles (but features different from CEA-7)
- > secondary phase at MgO grain boundaries ?

Summary 1/2

- Within the frame of the international collaboration **FUTURIX-FTA** as well as the European research programmes **EUROTRANS (FP6)** and **FAIRFUELS (FP7)** **CEA and JRC** have conducted together preparation, irradiation and **PIE** of **2 CerCer** and **2 CerMet** fuels for **transmutation of Am** in the **Phénix fast reactor**.
- CEA has achieved promising PIE results regarding CerCer fuels based on MgO matrix (CEA 7 & CEA 8):
 - **Non-significant elongations and deformation of the pins**
 - **No ^{137}Cs migration in the fuel stacks**
 - **Low Helium and Fission Gas release rates**
 - **A significant fraction of fuel pellets recovered as one piece**
 - **Moderate and similar pellet swelling ($\sim 3\%$) for both fuels (0.3–0.5 vol.%/at%)**
 - **Low hydrostatic density changes ($\sim 0,4\%$)**

Summary 2/2

- ITU has achieved promising PIE results regarding CerMet fuels based on Mo matrix (ITU 5 & ITU 6):
 - **No significant pin length elongation**
 - **No significant pin diameter change at position of the CerMet columns**
 - **No or very low fission product migration**
 - **No or very low Cs release from CerMet pellets**
 - **Pellet length changes ~5% & ~3% for ITU 5 & 6, respectively**
 - **Volume swelling circa 15% & 9% (0.7-0.9 vol.%/at%)**
 - **A fractional gas release of circa 30 % was measured for both pins**
 - **All pellets were recovered completely intact**
 - **No cracks inside the Mo matrix, Bubbles located inside particles**
 - **Outer particles formed open porosity during irradiation (improve synthesis in the future)**

Thank you for your attention!



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References

1. L. Donnet et. al., The FUTURIX-FTA Experiment in PHENIX: Status of fuel fabrication, Proceedings of GLOBAL 2005 Tsukuba, Japan, Oct 9-13, 2005, Paper No. 258
2. G. Cécilia, F. Delage, J. Lamontagne, L. Loubet, Post-Irradiation Examination results for MgO-CERCER Am-bearing fuels irradiated in PHENIX within the frame of the FUTURIX-FTA experiment, Proceedings of Global 2015, September 20-24, 2015 - Paris (France)
3. D. Papaioannou, R. Nasyrow, W. de Weerd, D. Freis, J. Somers, FAIRFUELS Deliverable 3.2.3, FUTURIX-FTA ITU-5 & ITU-6 Cer-Met PIE, JRC-ITU, Karlsruhe, Germany, 2015

Back-up slides

Material Data

Mo

- Molar mass 95.95 g/mol
- Density 10.28 g/cm³
- Thermal expansion 4.8 $\mu\text{m}/(\text{m}\cdot\text{K})$ (at 25 °C)
- Thermal conductivity 138 W/(m·K)
- Specific heat 250 J/kg.K
- Melting point 2896 K (2623 °C, 4753 °F)

MgO

- Molar mass 40.30 g/mol
- Density 3.58 g/cm³
- Thermal expansion 9-12 $\mu\text{m}/(\text{m}\cdot\text{K})$
- Thermal conductivity 45 - 60 W/(m·K)
- Specific heat 880 – 1030 J/kg.K
- Melting point 3125 K (2852 °C, 5166 °F)



FX 5 & 6

Table 1.- Impurity analysis of molybdenum metal

Element µg/g	Concentration	Element	Concentration µg/g
Na	22.99 ± 2.76	Ce	3.91 ± 0.47
Mg	9.14 ± 1.10	Re	2.83 ± 0.34
Al	19.29 ± 2.32	W	80.80 ± 9.70
Rb	0.72 ± 0.09	Pb	20.22 ± 2.43
Y	0.12 ± 0.01	Bi	0.18 ± 0.02
Ru	2.78 ± 0.33	V	0.47 ± 0.06
Cd	1.19 ± 0.14	Cr	8.03 ± 0.96
Ag	0.17 ± 0.02	Mn	1.16 ± 0.14
Sb	0.67 ± 0.08	Fe	93.21 ± 11.19
Cs	0.89 ± 0.11	Co	0.43 ± 0.05
Ba	1.00 ± 0.12	Cu	3.90 ± 0.47
La	0.05 ± 0.01	K	30.03 ± 3.60

Table 1.-Am and Pu content in the beads

	FX5	FX6
Beads	PuO ₂	(Zr,Pu)O ₂
Analysis number	3458	3459
Pu (wt%)	68.65 ± 0.89	28.42 ± 0.37
Am (wt%)	17.59 ± 0.14	30.05 ± 0.24
Zr (wt%¹)		25.31
Am/ΣM	20.4	35.9

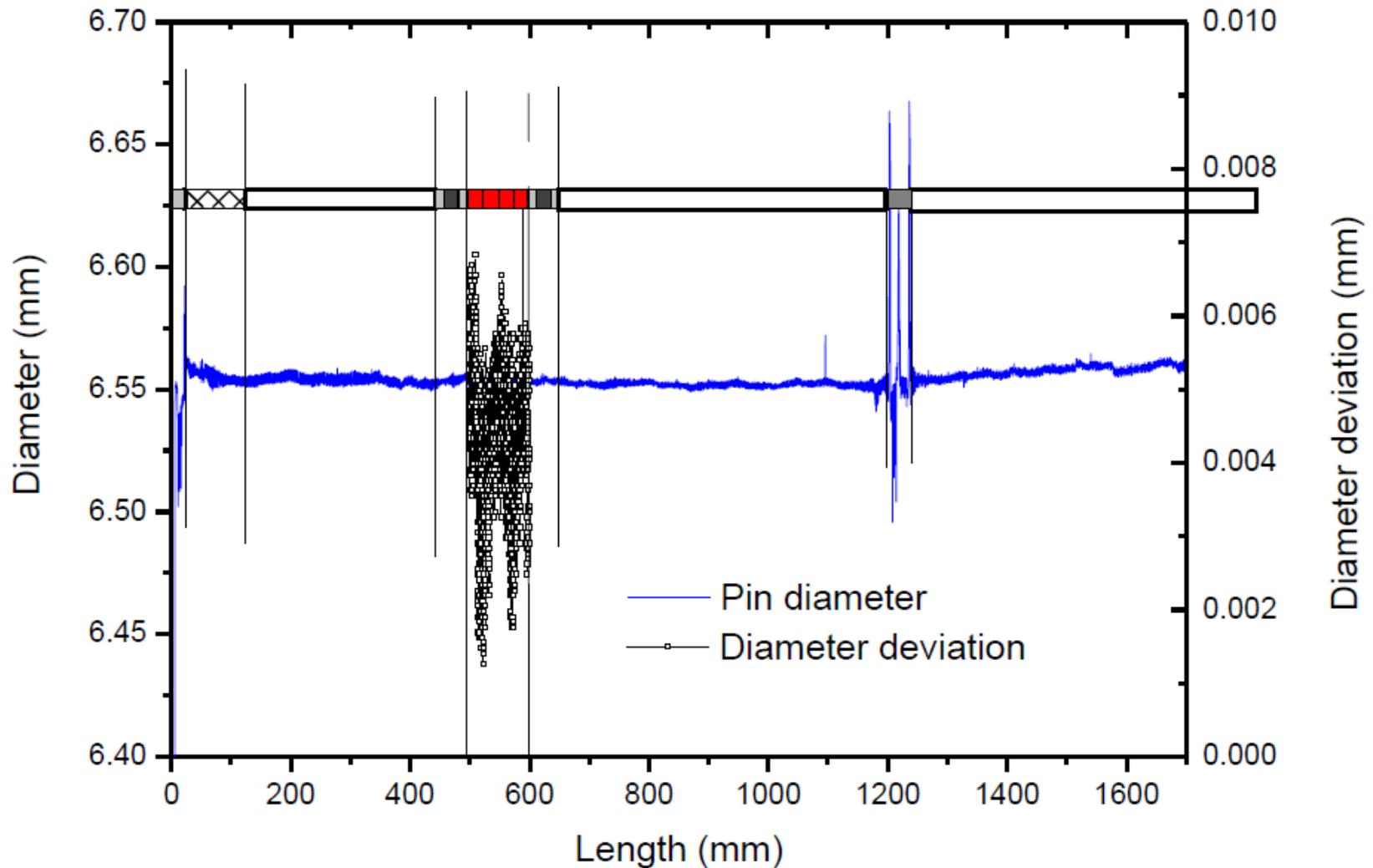
Table 2.- Isotopic composition of plutonium (analysis 08.08.2005)

TIMS	
²³⁸ Pu	0.01535
²³⁹ Pu	90.6340
²⁴⁰ Pu	9.11570
²⁴¹ Pu	0.16188
²⁴² Pu	0.07263

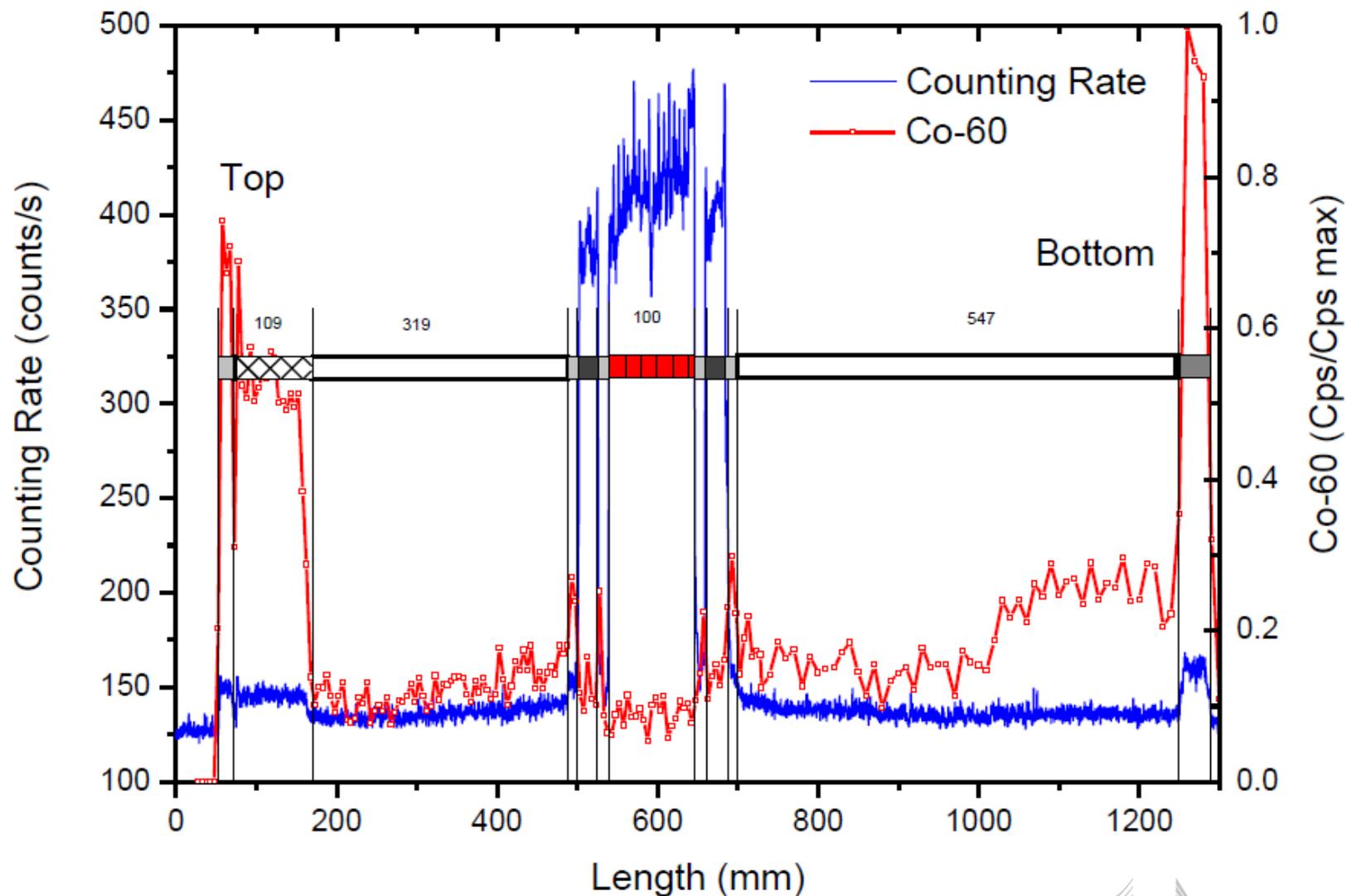
Table 3.- Particle size distribution of plutonium compounds following calcination.

Range µm	FX5 (%wt)	FX6 (%wt)
>225	0.02	0.03
125-225	28.24	38.51
90-125	47.21	38.07
63-90	21.82	14.24
32-63	2.33	6.03
<32	0.38	3.11

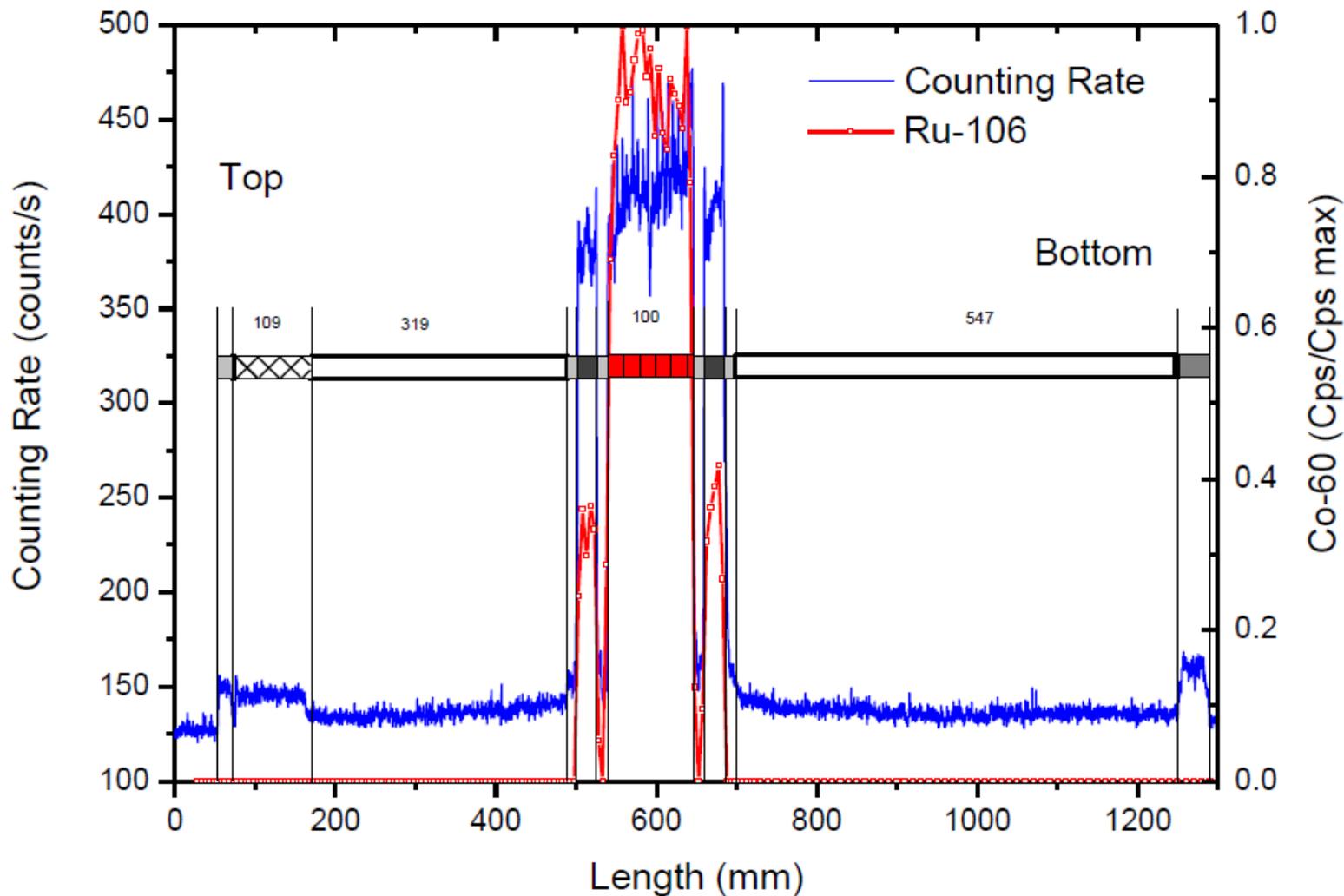
Profilometry ITU-6



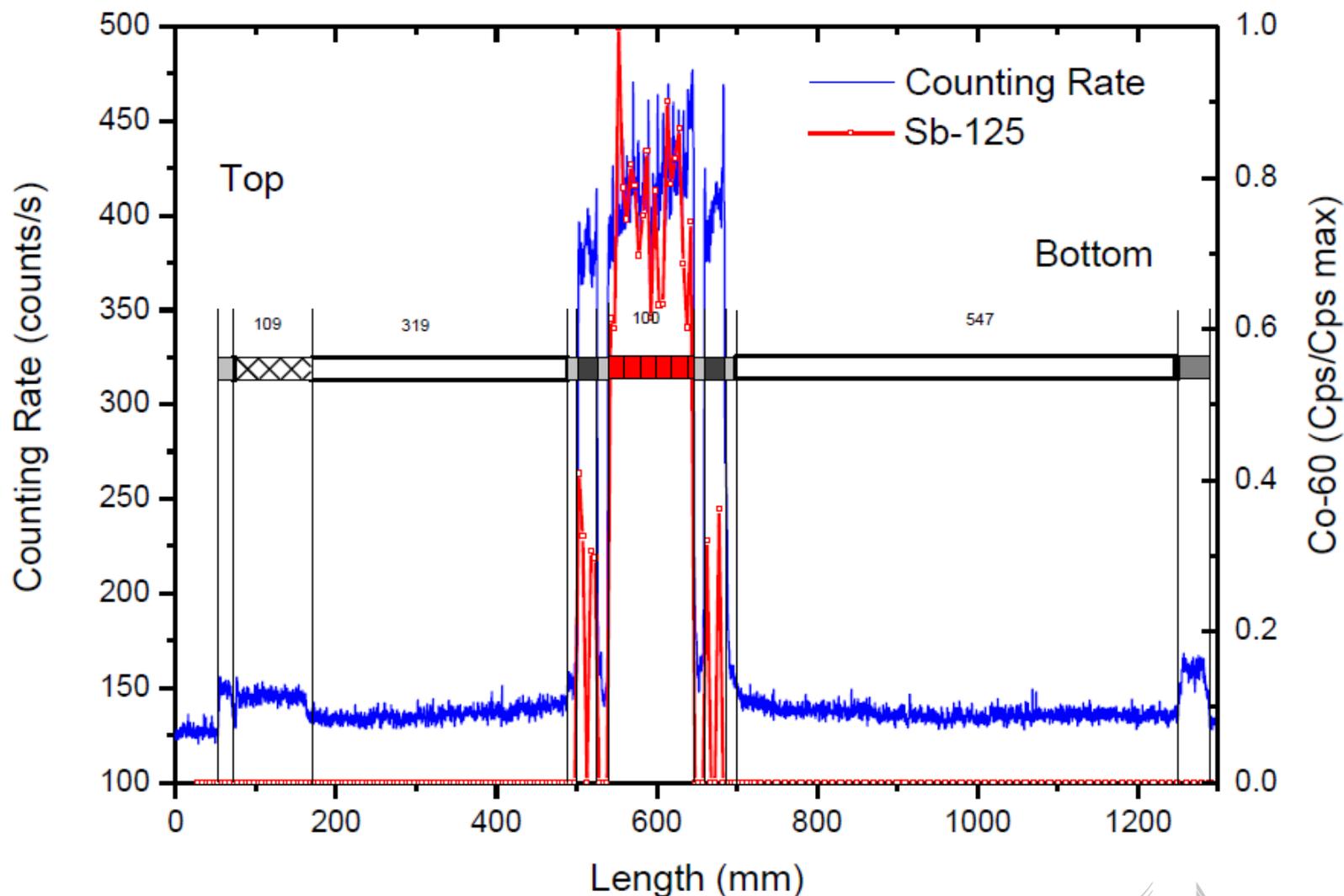
Gamma Spectrometry of ITU-5 (^{60}Co)



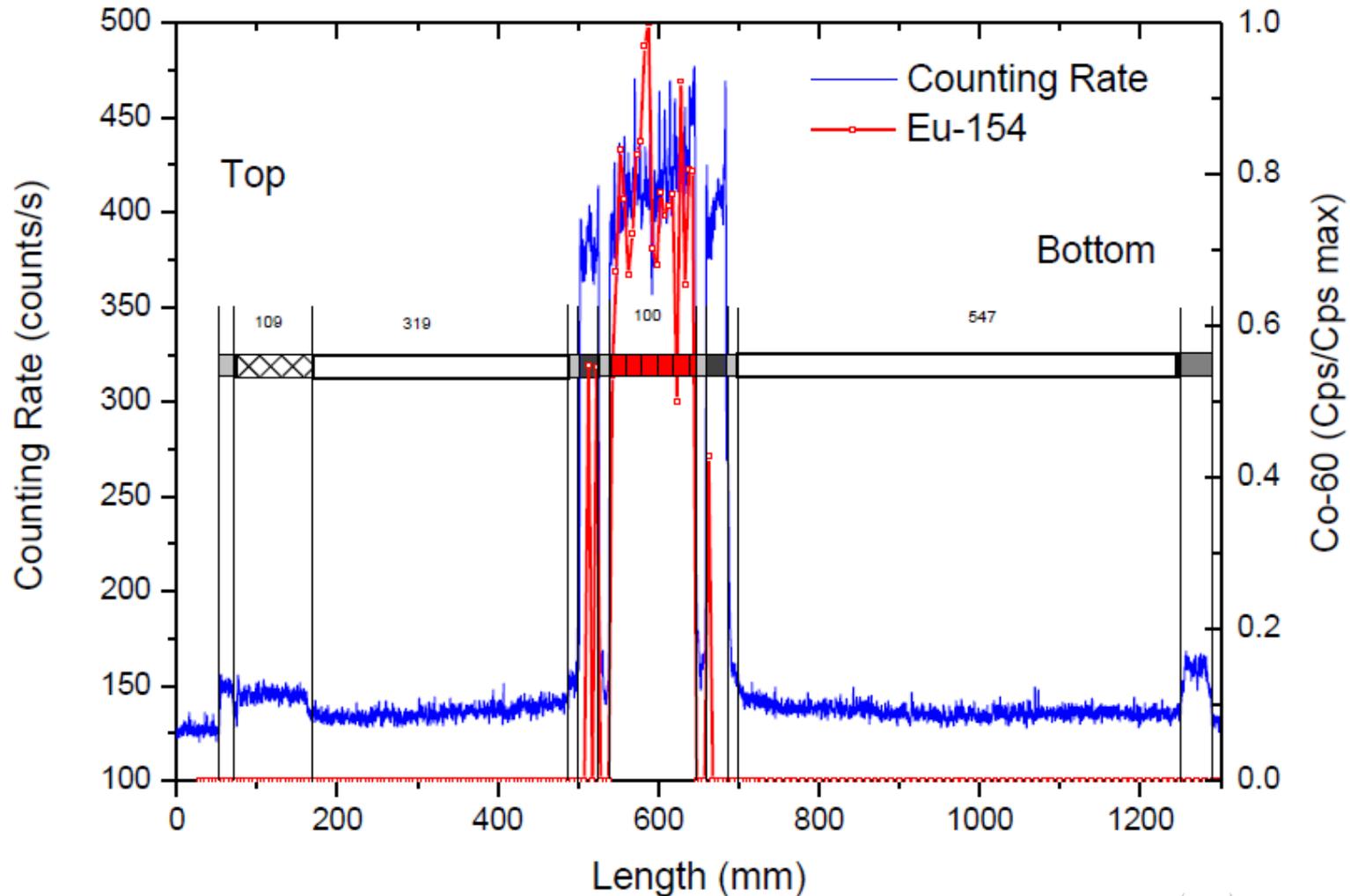
Gamma Spectrometry of ITU-5 (^{106}Ru)



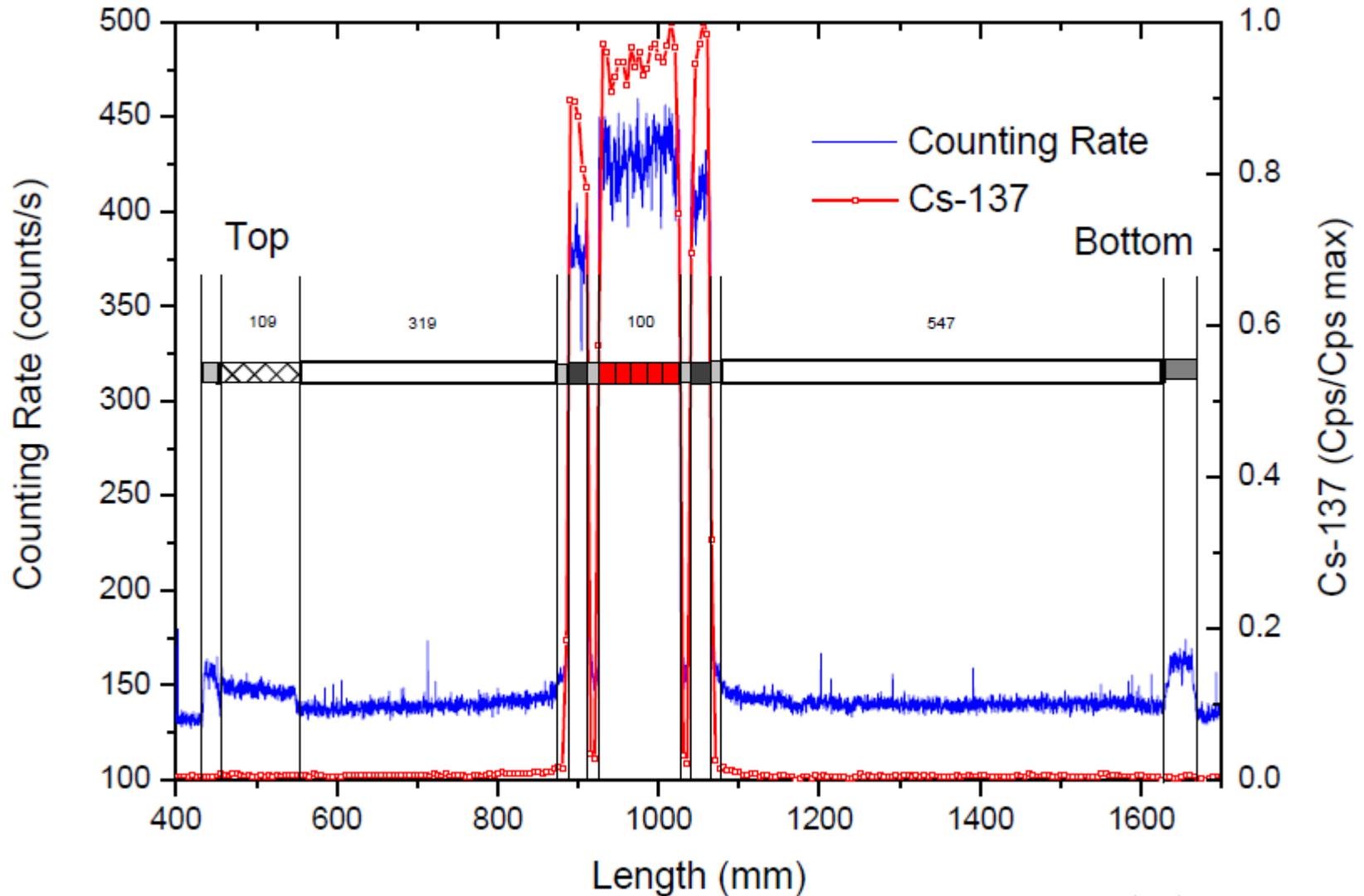
Gamma Spectrometry of ITU-5 (^{125}Sb)



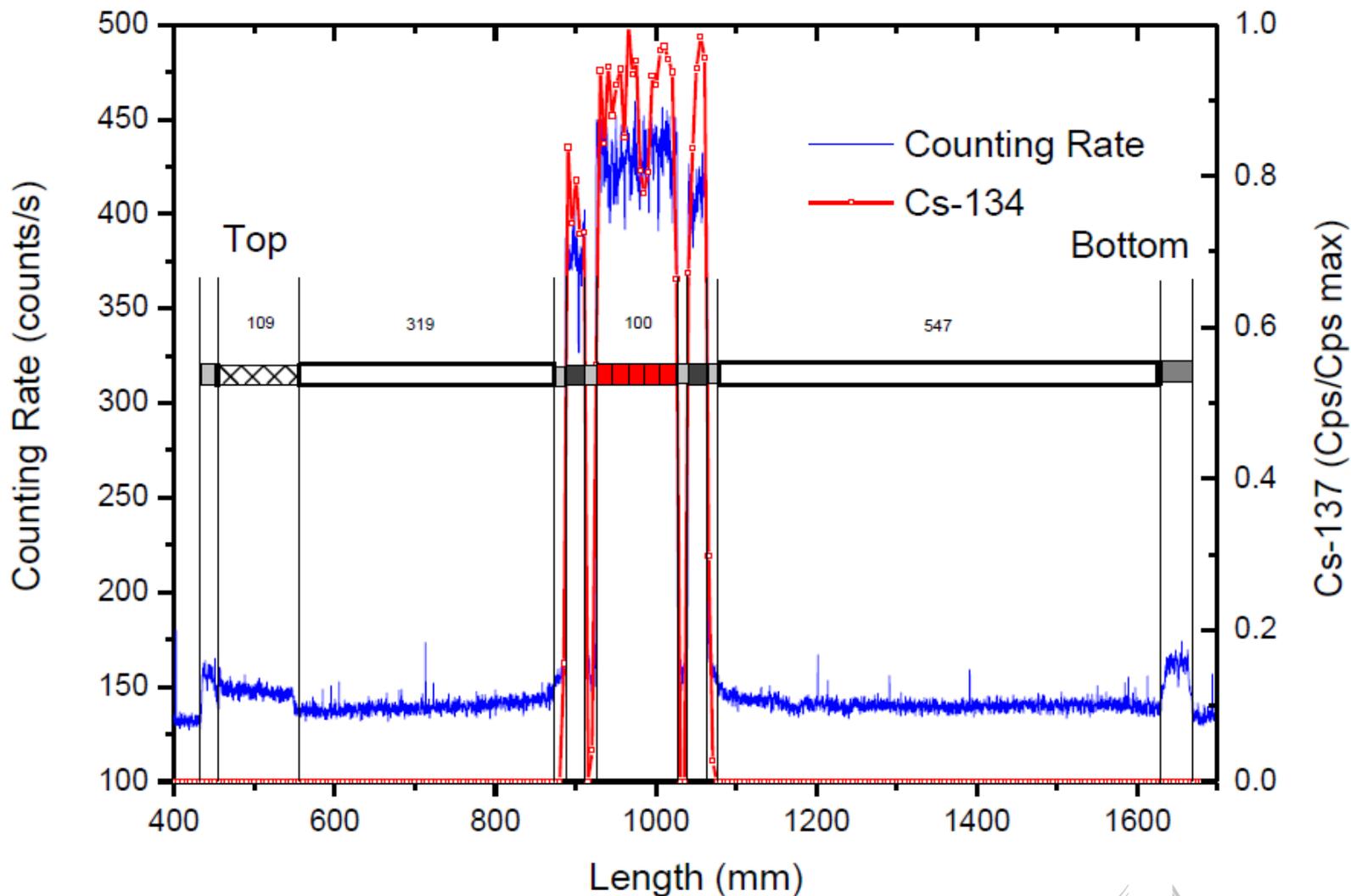
Gamma Spectrometry of ITU-5 (^{154}Eu)



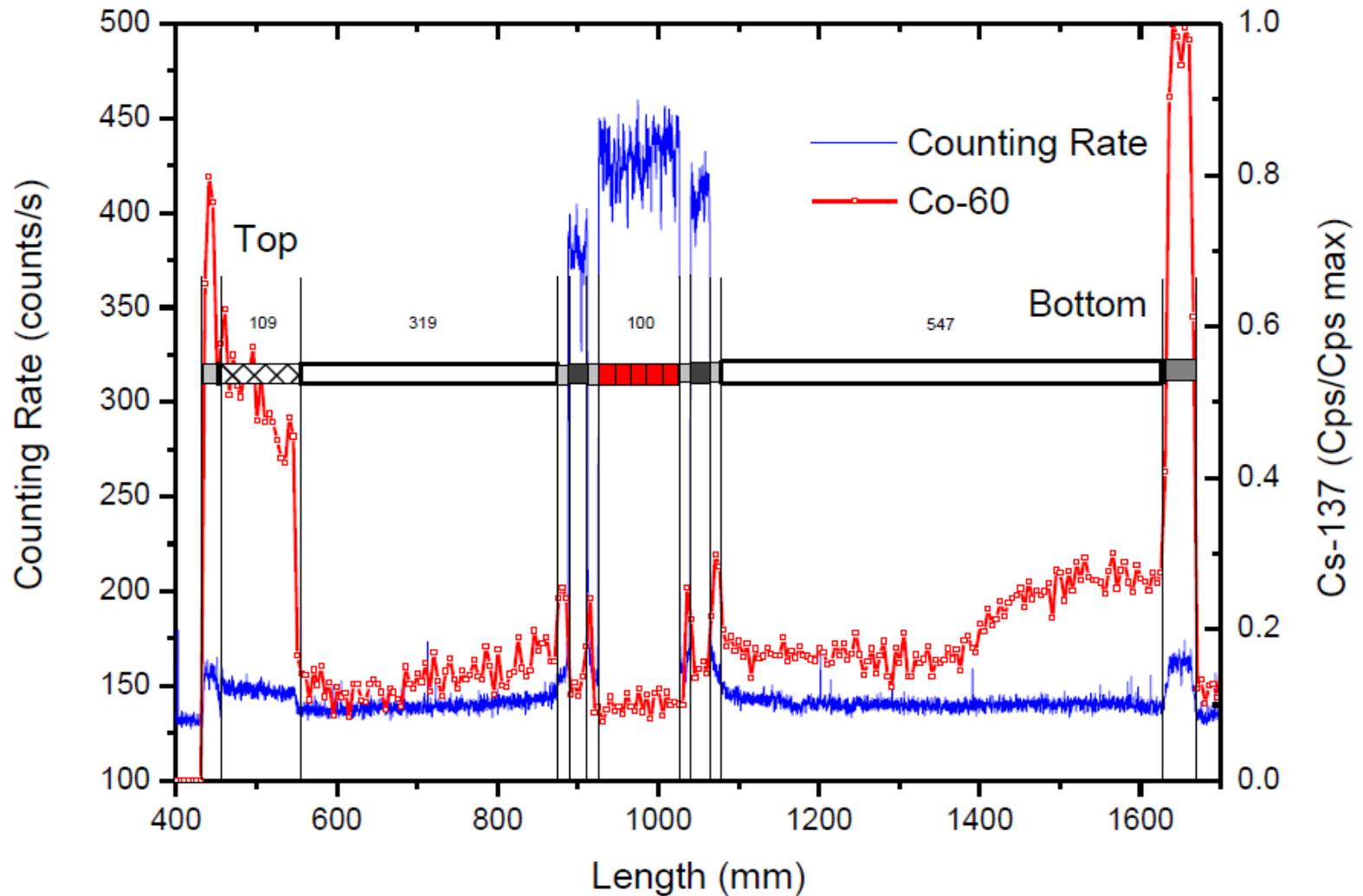
Gamma Spectrometry of ITU-6 (^{137}Cs)



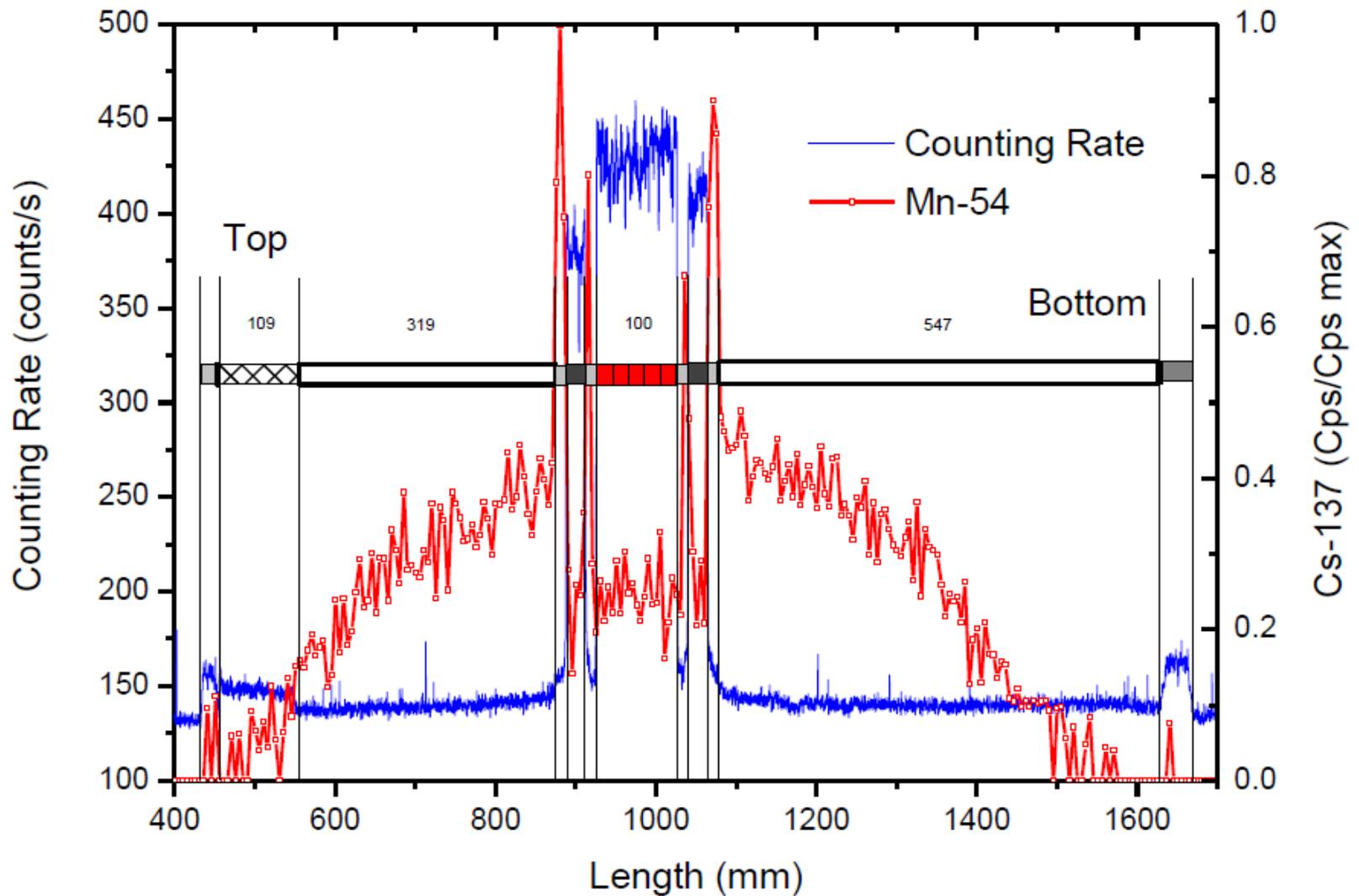
Gamma Spectrometry of ITU-6 (^{134}Cs)



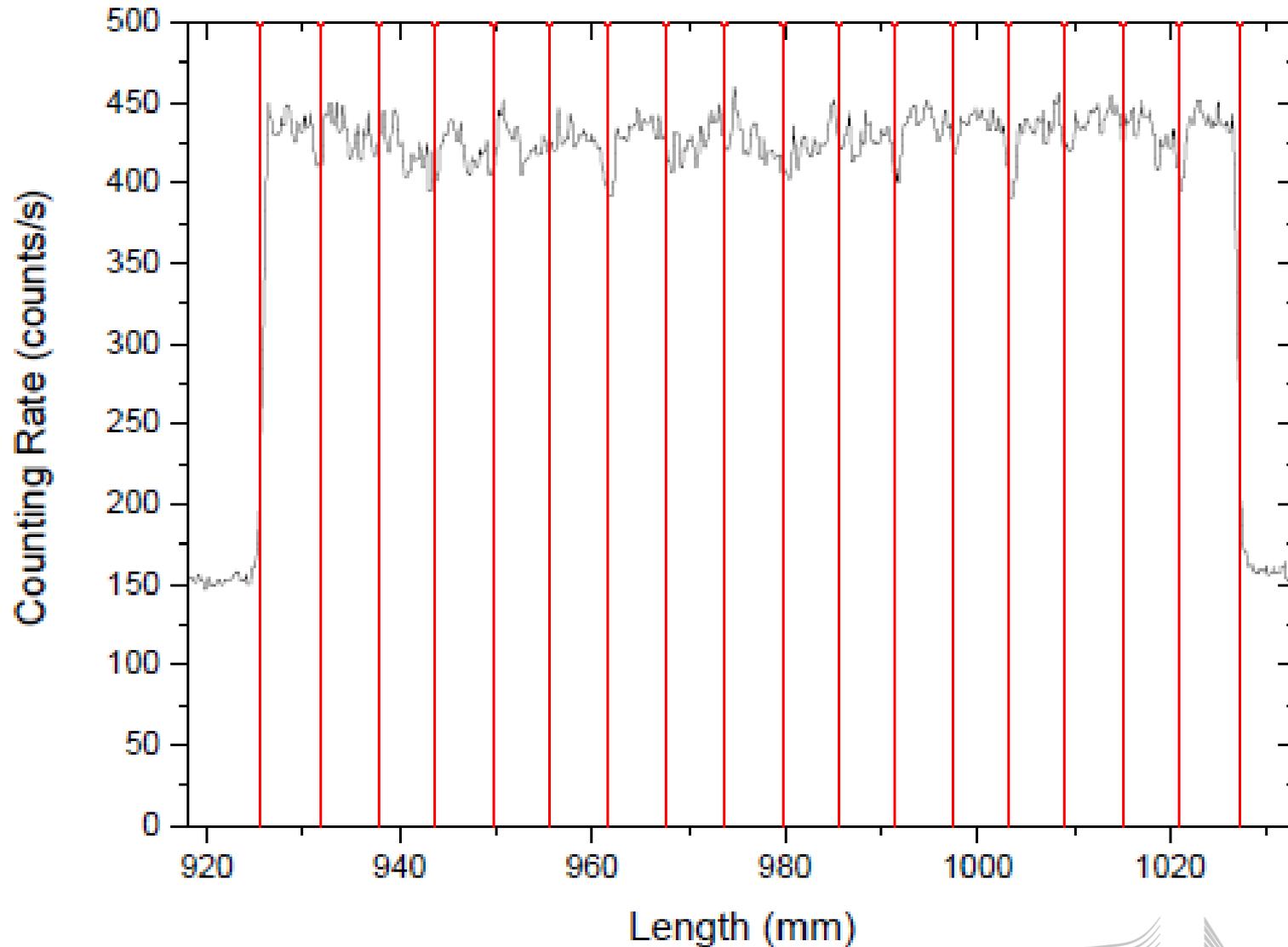
Gamma Spectrometry of ITU-6 (^{60}Co)



Gamma Spectrometry of ITU-6 (^{54}Mn)



Determination of Pellet Positions ITU-6



Gas Puncturing Results

Pin ID	ITU-5	ITU-6	CEA-7	CEA-8
Date of puncturing	June 2015		February 2014	
Pressure at puncturing stage (MPa)	0.148	0.195	0.106	0.118
Free volume in the pin(cm ³)	19.18	18.93	18.9±3.1	18.9±3.2

Initial free volume: 18.6 ml

Pin	ITU-5	ITU-6	CEA-7	CEA-8
%H ₂	-	-	0.002	0.002
% He	83.23	87.18	98.942	99.336
% Xe	14.34	10.3	0.466	0.100
% Kr	2.04	2.02	0.033	0.008
% O ₂	0.03	0.04	0.102	0.065
% N ₂	0.11	0.12	0.425	0.330
% Ar	0.05	0.14	0.024	0.154
% CO ₂	0.19	0.21	0.006	0.005

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HoD G.I
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HoU G.II.6
Klaus Lutzenkirchen

Facilities and Equipment at JRC Karlsruhe

24 hot cells with capacities up to **1 Mio Curies** and some **400 glove boxes** in **30 alpha-laboratories**.

Minor Actinide Lab for preparing **irradiation targets** containing amounts of **actinides** for **transmutation** experiments

Clean Lab category 10-100-Large Geometry SIMS for safeguards and nuclear forensic applications

Destructive and non-destructive analyses

Basic materials science investigations

- Materials studies down to 1 K and up to 8000 K and 60 GPa
- Photo-electron spectroscopy, X-ray diffraction, (electrical resistance and optical properties) on highly radioactive samples, Mössbauer spectroscopy at normal and elevated pressures
- High-resolution electron microscopes,
- Shielded microprobes,

