

# Information Sheet!

12 januari 1987

## PuO<sub>2</sub> Pilot Reference Samples for Isotopic Composition Measurements by Gamma Spectrometry

CBNM - NRM 271

Specifications for set 0/9

### 1. PuO<sub>2</sub> Pellet Specifications

Pellet Specifications	Sample Identification			
	93 0/2	84 0/2	70 0/2	61 0/2
Areal density [g.cm <sup>-2</sup> ]	3.80	3.86	3.99	3.82
Diameter [mm]	14.90	14.84	14.58	14.84
Flatness [mm]	-----< ± 0.02-----			
Density [g.cm <sup>-3</sup> ]	9.88	10.14	10.93	10.55
Thickness [mm]	3.85	3.81	3.66	3.63
Mass [g]	6.630	6.685	6.680	6.624
Stoichiometry (PuO <sub>2-x</sub> ), X	-----< 0.05-----			

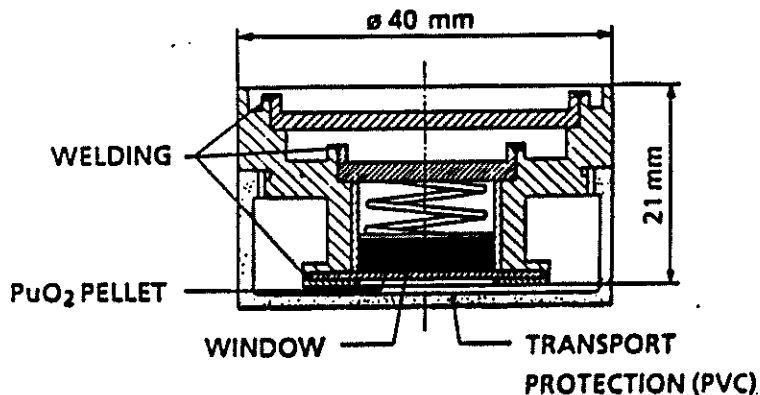
### 2. Canning

Container certified by BAM, F.R. Germany (approval D/0039/S), as type A, special form radioactive material following IAEA specifications.

Material : Stainless steel.

Window thickness : 0.7796 ± 0.0005 mm

### 3. Layout of PuO<sub>2</sub> Pilot Reference Samples



see over

|| preliminary presentation

4. Isotopic Composition of Sample Material

$\frac{Pu(iso)}{Pu(tot) + Am}$  [at %], preliminary values\* valid for 20 June 1986

Material type (% $^{239}Pu$ )	Isotope					
	$^{238}Pu$	$^{239}Pu$	$^{240}Pu$	$^{241}Pu$	$^{242}Pu$	$^{241}Am$
93	0.012	93.338	6.286	0.2212	0.039	0.1038
84	0.0706	84.214	14.130	1.0184	0.3526	0.2152
70	0.8420	72.581	18.039	5.3576	2.0307	1.1492
61	1.1889	61.771	24.992	6.5437	4.0880	1.4161

(\*) values are referring to synthetic mixtures.

5. Uncertainties of isotope abundances, preliminary figures (in percent of the isotope abundance values) for a confidence level of about 95 %.

Isotope	Material type (% $^{239}Pu$ )			
	93	84	70	61
$^{238}Pu$	$\pm 10$	$\pm 1.2$	$\pm 0.15$	$\pm 0.15$
$^{239}Pu$	$\pm 0.1$		$\pm 0.05$	
$^{240}Pu$	$\pm 0.2$		$\pm 0.10$	
$^{241}Pu$	$\pm 0.4$		$\pm 0.15$	
$^{242}Pu$	$\pm 5$	$\pm 0.20$	$\pm 0.15$	
$^{241}Am$	$\pm 2$		$\pm 1.0$	

6. Chemical Purity, maximum values of total impurities without  $^{241}Am$

Impurities [ug/g]	Material type (% $^{239}Pu$ )			
	93	84	70	61
for all elements, $Z \leq 30$	170	500	500	400
for low Z elements (B, Be, Li, F, Mg, Na) <sup>1</sup>	10	20	10	30
for all elements $Z > 30$	30	800 (20) <sup>2</sup>	600 (250) <sup>2</sup>	1200 (380) <sup>2</sup>

- (1) elements with high  $\alpha/n$  cross section.
- (2) included contributions of ingrown  $^{234}U$  from the decay of  $^{238}Pu$  (June 1986).

# Certified Nuclear Reference Material

## Certificate of Analysis

CBNM NUCLEAR REFERENCE MATERIAL 271  
Pu isotope Abundances and <sup>241</sup>Am Concentration  
Certified Reference Material (PuO<sub>2</sub>) for Gamma-Ray Spectrometry

$\frac{\text{Pu(iso)}}{\text{Pu(total)}} \cdot \frac{{}^{241}\text{Am}}{\text{Pu(total)}}$  abundances and uncertainties (95 % confidence level)  
valid at 20 June 1986

- atom per cent (at%)

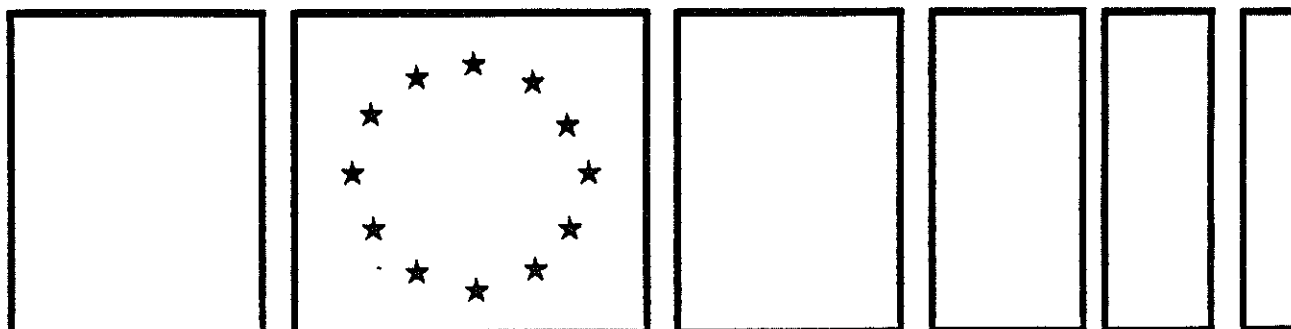
Material	Isotope	<sup>238</sup> Pu	<sup>239</sup> Pu	<sup>240</sup> Pu	<sup>241</sup> Pu	<sup>242</sup> Pu	<sup>241</sup> Am*
CBNM Pu93		0.0117 2	93.4392 40	6.2886 39	0.2215 4	0.0390 3	0.1039 21
CBNM Pu84		0.0706 6	84.3985 84	14.1578 85	1.0197 18	0.3534 10	0.2157 22
CBNM Pu70		0.8506 18	73.4248 98	18.2445 87	5.4257 34	2.0544 23	1.1624 116
CBNM Pu61		1.2045 25	62.6562 283	25.3526 241	6.6376 87	4.1491 64	1.4362 144

- mass per cent (m%)

Material	Isotope	<sup>238</sup> Pu	<sup>239</sup> Pu	<sup>240</sup> Pu	<sup>241</sup> Pu	<sup>242</sup> Pu	<sup>241</sup> Am*
CBNM Pu93		0.0117 2	93.4123 40	6.3131 39	0.2235 4	0.0395 3	0.1047 21
CBNM Pu84		0.0703 6	84.3377 84	14.2069 85	1.0275 18	0.3576 10	0.2173 22
CBNM Pu70		0.8458 18	73.3191 98	18.2945 87	5.4634 34	2.0772 23	1.1705 117
CBNM Pu61		1.1969 25	62.5255 283	25.4058 241	6.6793 87	4.1925 64	1.4452 144

This certificate applies to the reference samples : CBNM Pu 93 O/8  
CBNM Pu 84 O/8  
CBNM Pu 70 O/8  
CBNM Pu 61 O/8

Commission of the European Communities  
Joint Research Centre  
Central Bureau for Nuclear Measurements



## PURPOSE

This certified Nuclear Reference Material (NRM) is intended to be used for the non-destructive determination of the abundance of  $^{238}\text{Pu}$ ,  $^{239}\text{Pu}$ ,  $^{240}\text{Pu}$ ,  $^{241}\text{Pu}$  and  $^{241}\text{Am}$  by gamma-ray spectrometry using characteristic radiation of each isotope. For this purpose four parameters are specified : mass and geometry of the  $\text{PuO}_2$  layer, can window thickness and the chemical purity of the  $\text{PuO}_2$ , in addition to the Pu isotope abundances and the  $^{241}\text{Am}$  concentration .

## DESCRIPTION

CBNM NRM 271 (Central Bureau of Nuclear Measurements certified Nuclear Reference Material 271) consists of a set of four sealed cans, each of which is defined as a reference sample.

Each reference sample contains a sintered pellet of about 6.6 g  $\text{PuO}_2$  with one of the four isotopic compositions. The can is made of a stainless steel and has a maximum outer diameter of 40 mm and a height of 21 mm.

The bottom of the can has a specified thickness and serves as window for measuring the emitted gamma-ray radiation. A plastic cover protects the measurement window.

## IDENTIFICATION

Each reference sample has the following marks engraved on the outer can surface :

- CBNM : body issuing the certificate
- Pu, a two-digit number, followed by an O : the symbol of the element plutonium, the nominal  $^{239}\text{Pu}/\text{Pu}$  abundance and the indication that the chemical form of the reference material is oxide.
- A slash followed by a one or two-digit number specific for each NRM, i.e. for each set of reference samples.

This certificate relates to the set of reference samples with the numbers indicated on the first page.

## CHARACTERIZATION

In order to ensure the isotopic homogeneity of the sample material the total amount of the starting material was brought into solution and purified.

The plutonium isotope abundances were determined by thermal ionisation mass-spectrometry at CBNM. All measurements were calibrated by synthetic plutonium isotope mixtures prepared from purified isotopes.

The  $^{238}\text{Pu}$  abundance of the reference material Pu93 was also determined by  $\alpha$  spectrometry.

The  $^{241}\text{Am}$  concentration was determined by :

- calculation of the  $^{241}\text{Pu}$  decay since the date of chemical separation
- alpha spectrometry and low-energy gamma-ray spectrometry on liquid samples using  $^{241}\text{Am}$  reference material

High resolution gamma-ray spectrometry measurements of the reference samples confirmed that the plutonium isotope abundance homogeneity of the four sample materials must be equal or better than the overall accuracies obtained from mass spectrometry measurements.

## STATEMENT OF UNCERTAINTIES

The overall uncertainties of the abundances were estimated by combining the different uncertainty components from the mass spectrometric characterisation and homogeneity control measurements.

The uncertainty of the  $^{238}\text{Pu}$  abundance of the reference material Pu 93 was obtained by combining the different uncertainty components from the  $\alpha$  spectrometric measurements with the estimated uncertainty of incomplete  $^{241}\text{Am}$  purification.

The error propagation of all uncertainties was calculated according to the principles applicable to independent error contributions.

The overall uncertainties of the  $^{241}\text{Am}$  concentrations were estimated from the uncertainties resulting from the different determination methods. The resulting figures were enlarged to 1 % (2 % for the 93 %  $^{239}\text{Pu}$  sample).

#### NOTICE TO THE USER

The Bundesanstalt fuer Materialpruefung (BAM), Berlin has declared on 13 June 1983 under permission number D/0039/S that the canning as used for the reference material is conform to the requirements "Special Form Radioactive Material" as described in the IAEA "Regulations for Safe Transport of Radioactive Material", 1973, Revised Edition.

#### SUPPLEMENTARY SPECIFICATIONS

##### Can window

The uncertainties are given as maximum deviations.

- Thickness (all NRM's)	$0.780 \pm 0.002$ mm
- Thickness set 8 :	$0.7800 \pm 0.0010$ mm
- Deviation from flatness :	$\pm 0.1$ mm

##### Chemical purity of the $\text{PuO}_2$

The plutonium oxide is considered to be stoichiometric  $\text{PuO}_2$  containing impurities. Maximum total impurities are :-

Impurities (mg/kg <sup>-1</sup> )	material :	<u>Pu 93 O</u>	<u>Pu 84 O</u>	<u>Pu 70 O</u>	<u>Pu 61 O</u>
for all elements, $Z \leq 30$		170	500	500	400
for low Z elements (B, Be, Li, F, Mg, Na) <sup>1</sup>		10	20	10	30
for all elements, $Z > 30$		30	800(20) <sup>2</sup>	600(250) <sup>2</sup>	1200(380) <sup>2</sup>

(1) elements with high  $\alpha/n$  cross section

(2) included contributions of ingrown  $^{234}\text{U}$  from decay of  $^{238}\text{Pu}$  (June 1986)

##### $\text{PuO}_2$ filling (pellet)

The uncertainties for these NRM's are given as maximum deviations :

- For all NRM's				
- Mass :			$6.65 \pm 0.06$ g	
- Height :			$3.75 \pm 0.14$ mm	
- Diameter :			$14.77 \pm 0.21$ mm	
- Surface density :			$3.87 \pm 0.13$ g·cm <sup>-2</sup>	

- For set 8

Sample :	<u>93 O/8</u>	<u>84 O/8</u>	<u>70 O/8</u>	<u>61 O/8</u>
- Mass :	6.623	6.682	6.642	6.630 g
- Height :	3.80	3.83	3.63	3.64 mm
- Diameter	14.92	14.83	14.59	14.85 mm
- Surface density :	3.79	3.87	3.97	3.83 g·cm <sup>-2</sup>

## TIME OF CHEMICAL PURIFICATION OF THE PLUTONIUM BASE MATERIAL

- |                                      |      |
|--------------------------------------|------|
| - for material CBNM PU 84; 70 AND 61 | 1982 |
| - for material CBNM PU 93            | 1978 |

## CONTRIBUTIONS

The following laboratories have contributed to the fabrication of this reference material :

- CEN Grenoble, where the PuO<sub>2</sub> base material was purified;
- JRC Institute TUI Karlsruhe, where the PuO<sub>2</sub> pellets were produced and canned ;
- JRC Institute CBNM Geel, where the canning was designed and fabricated.

Furthermore the following laboratories have contributed to the analysis of this reference material.

- CEN Grenoble and ;
- CEN/SCK Mol ;
- JRC Institute CBNM Geel, Analytical Science Group, Mass Spectrometry group, Radio Nuclide group.

## LEGAL DISCLAIMER

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