

Certificate of Analysis

3:1 MOX POWDER UKMOX3

1. Description

The uranium-plutonium mixed oxide was prepared by blending solutions of uranium and plutonium in dilute nitric acid, to obtain a nominal U/Pu ratio of 3. After co-precipitation using ammonia, the mixture was calcined in 4% hydrogen in argon at 850°C for four hours to produce the mixed oxide.

ca. 1.3g portions of the material have been encapsulated in tared IAEA BC4 vials, numbers 34153 to 34171.

This mixed oxide forms part of a set of five oxide powders (UKMOX1-100), covering U/Pu ratios of 1, 3, 10, 50 and 100.

2. Methods of Characterisation

The uranium and plutonium contents of the material were determined by Davies and Gray titration and controlled potential coulometry, respectively. Analyses were undertaken on replicate solutions prepared from four 0.5g samples for uranium and three 0.5g samples for plutonium. Sample solutions were measured with reference to the uranium standard UKU1 and the plutonium standard UKPu1.

The uranium and plutonium isotopic composition of the material was determined by thermal ionisation mass spectrometry from four sample measurements. Mass fractionation corrections were made with reference to the 1:1:1 ^{233}U : ^{235}U : ^{238}U standard UKU2 and the 1:1:1 ^{239}Pu : ^{240}Pu : ^{242}Pu standard UKPu3. The $^{238}\text{Pu}/^{239}\text{Pu}$ ratio was determined by alpha spectrometry.

The $^{241}\text{Am}/\text{Pu}$ ratio was determined by anion exchange separation followed by coulometry (plutonium) and liquid scintillation counting (americium). The $^{237}\text{Np}/\text{Pu}$ ratio was determined by gamma spectrometry, using the 300keV and 297keV emission lines, whilst the metallic impurities in the material were determined by inductively coupled plasma optical emission spectrometry.

3. Certified Composition

The certified composition of the mixed oxide standard material UKMOX3 is given in the following table, valid for the date of bottling. Overall uncertainties are quoted at the 95% CI.

Quantity	Certified Result	Uncertainty (2S)
U/Pu ratio	3.3518	0.0051
Pu concentration	19.959 wt %	0.022 wt %
U concentration	66.898 wt %	0.069 wt %
²³⁷ Np/Pu	0.00057	0.00003
²⁴¹ Am/Pu	0.02522	0.00015
Total metal impurities (excluding Np, Am)	<0.08 wt %	
²³⁸ Pu/ ²³⁹ Pu	0.001546	0.000008
²⁴⁰ Pu/ ²³⁹ Pu	0.28706	0.00014
²⁴¹ Pu/ ²³⁹ Pu	0.017756	0.000030
²⁴² Pu/ ²³⁹ Pu	0.009531	0.000094
²⁴⁴ Pu/ ²³⁹ Pu	0.000023	0.000018
²³⁴ U/ ²³⁸ U	0.000250	0.000006
²³⁵ U/ ²³⁸ U	0.016089	0.000034
²³⁶ U/ ²³⁸ U	0.000707	0.000010
²³⁸ Pu abundance	0.11684 wt %	0.00061 wt %
²³⁹ Pu abundance	75.908 wt %	0.010 wt %
²⁴⁰ Pu abundance	21.882 wt %	0.008 wt %
²⁴¹ Pu abundance	1.3591 wt %	0.0023 wt %
²⁴² Pu abundance	0.7326 wt %	0.0072 wt %
²⁴⁴ Pu abundance	0.0018 wt %	0.0014 wt %
²³⁴ U abundance	0.0242 wt %	0.0006 wt %
²³⁵ U abundance	1.5623 wt %	0.0032 wt %
²³⁶ U abundance	0.0689 wt %	0.0010 wt %
²³⁸ U abundance	98.3446 wt %	0.0035 wt %
Plutonium atomic weight	239.318	
Uranium atomic weight	238.001	
Reference date	1 March 1997, results valid for sample weight on bottling.	

Authorisation:



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Reference: J W A Tushingham, C A J McInnes, J A Tibbles, I A S Carvel, R Cooke and A Johnson.
"The preparation and characterisation of a series of UO₂-PuO₂ (MOX Powder) reference materials." AEAT- 1200, SRDP-R229 (1997).