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Determination of plutonium content in plutonium dioxide (PuO_2) of nuclear grade quality — Gravimetric method

Détermination de la teneur en plutonium dans du dioxyde de plutonium (PuO_2) de qualité nucléaire — Méthode gravimétrique

Reference number
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Foreword

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Determination of plutonium content in plutonium dioxide (PuO_2) of nuclear grade quality — Gravimetric method

1 Scope and field of application

This International Standard specifies a precise and accurate gravimetric method for determining the plutonium content in plutonium dioxide (PuO_2) of nuclear grade quality, containing less than 0,5 % of non-volatile impurities. The content of these impurities shall be measured and a correction made to allow for them.

The method is used to cross-check accountancy analyses of plutonium dioxide.

2 Principle

Sampling and weighing of the sample in dry atmosphere (dew point less than or equal to − 40 °C). (The dew point shall be verified by using a measuring instrument and by checking the stability of the mass of an aliquot portion of PuO_2.) Ignition in air between 1 200 and 1 250 °C to constant mass in order to obtain a stoichiometric plutonium dioxide, which is stable and non-hygroscopic. Weighing of the calx. Impurity analysis and correction for non-volatile impurities. Calculation of plutonium concentration.

3 Interferences

3.1 Atmospheric moisture

Plutonium dioxide is hygroscopic unless it has been ignited at a temperature above 850 °C. Strict precautions shall therefore be normally taken to avoid moisture being absorbed when transferring the sample from the process line to the sub-sampling station; the sample shall, therefore, be taken and transferred in a tight, metallic vial. For the same reason, the sub-sampling station shall be subject to the same dry atmosphere as the process line.

3.2 Non-volatile impurities

All impurities which are not volatile at 1 200 °C cause a positive bias in the analysis. Their actual content shall be measured with appropriate techniques, including, for example, atomic emission or absorption spectroscopy.

If the total impurity content of the ignited oxide amounts to less than 0,5 %, a 20 % relative accuracy is satisfactory in the impurity analyses.

4 Reagents

None required.

5 Apparatus

5.1 Sub-sampling station, comprising a glove box under dry atmosphere (dew point less than or equal to − 40 °C) equipped with

a) an analytical balance accurate to ± 0,1 mg;

b) a hygrometer to measure the actual dew point in the box.

5.2 Ignition box, supplied with ambient air and equipped with a temperature-regulated muffle furnace capable of operating at 1 200 to 1 250 °C.

5.3 Stainless steel sampling vials.

5.4 Platinum crucibles.

5.5 Desiccator.

6 Procedure

6.1 Handling of the sample at the sampling station

6.1.1 Transfer at least 10 g of the material to be analysed into a vial (5.3).

6.1.2 Hermetically seal the vial.

6.1.3 Transfer the vial rapidly to the sub-sampling station (5.1).

6.2 Taring of crucibles

6.2.1 Ignite a clean crucible (5.4) for 1 h at 1 200 to 1 250 °C. Cool for 20 min in the desiccator (5.5) and then for 5 min in the balance [5.1 a)]; weigh to within ± 0,1 mg. Repeat the ignition until the mass remains constant to within ± 0,1 mg.

6.2.2 Record the constant mass, m_1, to an accuracy of ± 0,1 mg.

6.3 Sub-sampling

6.3.1 As soon as possible after receiving the vial containing the sample, transfer about 1,5 g of the sample into the tared crucible.

6.3.2 Measure and record the gross mass of the crucible, m_2, to an accuracy of ± 0,1 mg.

6.3.3 If several sub-samples are taken, keep the first in the sub-sampling station and weigh it again after all the sub-samples have been taken.

6.3.4 If the change in mass of the first sub-sample is less than 0,5 mg, transfer the sub-samples to the ignition box (5.2). If that is not the case, discard the sub-samples, adjust the hygrometry of the box and repeat the sampling and the procedure.

6.4 Ignition

6.4.1 Ignite the 1,5 g sample for 1 h at 1 200 to 1 250 °C.

6.4.2 Cool for 20 min in the desiccator and weigh it to within ± 0,1 mg.

6.4.3 Repeat 6.4.1 and 6.4.2 until the mass remains constant to within ± 0,1 mg.

6.4.4 Record the new gross mass, m_3, to an accuracy of ± 0,1 mg.

6.5 Additional measurements

6.5.1 Perform an isotopic analysis of plutonium to calculate its mean relative atomic mass, $A_r(\mathrm{Pu})$.

6.5.2 Perform an analysis of the impurities that are not volatile at 1 200 °C.

7 Expression of results

7.1 Calculation of the gravimetric conversion factor

Calculate the gravimetric conversion factor using the formula

$$C_{\mathrm{Pu}} = \frac{A_r(\mathrm{Pu})}{A_r(\mathrm{Pu}) + 2\,A_r(\mathrm{O})}$$

where

$A_r(\mathrm{O})$ (= 15,999 4) is the relative atomic mass of oxygen;

$A_r(\mathrm{Pu})$ is the mean relative atomic mass of plutonium calculated using the expression

$$A_r(\mathrm{Pu}) = \frac{1}{\dfrac{m_{238}}{238{,}050} + \dfrac{m_{239}}{239{,}052} + \dfrac{m_{240}}{240{,}054} + \dfrac{m_{241}}{241{,}057} + \dfrac{m_{242}}{242{,}059} + \dfrac{m_{244}}{244{,}064}}$$

where m_{238}, m_{239}, etc. are the mass fractions of the plutonium isotopes $^{238}\mathrm{Pu}$, $^{239}\mathrm{Pu}$, etc. in the samples.

7.2 Calculation of impurity correction factor

Express the results of the impurity analyses in micrograms of each impurity element per gram of the original sample (I_n).

Calculate the total mass of impurities, I_0, in the ignited sample using the formula

$$I_0 = 10^{-6} \times (m_2 - m_1) \times \sum_n (I_n C_n)$$

where

$m_2 - m_1$ is the mass of the sample before ignition :

m_2 being the gross mass before ignition (sample plus crucible),

m_1 being the mass of the crucible;

I_n is the quantity of impurity element n, in micrograms per gram of the original sample;

C_n is the gravimetric conversion factor for element n (see the annex).

7.3 Calculation of plutonium concentration

Calculate the plutonium concentration, Pu, as a percentage, in the sample using the formula :

$$\mathrm{Pu} = C_{\mathrm{Pu}} \times \frac{m_3 - m_1 - I_0}{m_2 - m_1} \times 100$$

where m_3 is the gross mass after ignition (sample plus crucible).

7.4 Repeatability

7.4.1 The coefficient of variation for a single gravimetric determination is about 0,04 %.

7.4.2 The standard deviation of the impurity correction factor is expected to be less than or equal to 1 000 ppm.

7.4.3 Consequently, the coefficient of variation of the total random error of a single determination is expected to vary between 0,04 % and 0,1 %, depending upon the purity of the ignited oxide.

7.5 Systematic errors

7.5.1 The systematic errors due to weighing have a coefficient of variation no greater than 0,014 %.

7.5.2 Non-stoichiometry of the plutonium oxide is a potential systematic error or bias; the coefficient of variation of this factor is expected to be less than 0,1 %.

7.5.3 Non-volatile impurities are responsible for three further possible sources of bias :

a) calibration errors in the impurity analysis;

b) uncertainties in the impurity conversion factors;

c) the impurities that are not corrected for, because they are neither measured nor detected, are a source of positive bias.

These sources may cause a systematic error of up to 20 % of the total impurity concentration.

8 Test report

The test report shall include the following information :

a) identification of the sample;

b) the reference of the method used;

c) the results and method of expression used;

d) any unusual features noted during the test;

e) any operations not included in this International Standard.

Annex

Gravimetric conversion factors for non-volatile impurities

(This annex forms an integral part of the Standard.)

Impurity	Probable form of impurity	Conversion factor, C_n
Ag	Ag	1,00
Al	Al_2O_3	1,89
Am	AmO_2	1,13
Ba	BaO	1,12
B	B_2O_3	3,22
Be	BeO	2,78
Bi	Bi_2O_3	1,11
Ca	CaO	1,40
Cd	Cd	1,00
Co	CoO	1,27
Cr	Cr_2O_3	1,46
Cu	Cu	1,00
Fe	Fe_3O_4	1,38
K	K_2O	1,21
Mg	MgO	1,66
Mn	Mn_3O_4	1,39
Rare earth elements	M_2O_3	1,16
Na	Na_2O	1,35
Ni	Ni_2O_3	1,40
Np	NpO_2	1,13
P	P_2O_5	2,29
Pb	PbO	1,07
Sb	Sb_2O_3	1,26
Si	SiO_2	2,14
Sn	SnO	1,13
Ta	Ta_2O_5	1,22
Th	ThO_2	1,14
Ti	TiO_2	1,67
U	U_3O_8	1,18
V	V_2O_5	1,78
W	WO_3	1,26
Zn	ZnO	1,24
Zr	ZrO_2	1,35

NOTE — These values are based on the best information available, with account being taken of the ignition and cooling conditions, and the effects of the plutonium oxide matrix.