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Nuclear fuel technology — Determination of plutonium content in plutonium dioxide of nuclear grade quality — Gravimetric method

Technologie du combustible nucléaire — Détermination de la teneur en plutonium dans du dioxyde de plutonium de qualité nucléaire — Méthode gravimétrique

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Foreword

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The committee responsible for this document is ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 5, *Nuclear fuel cycle*.

This second edition cancels and replaces the first edition (ISO 8300:1987), of which it constitutes a minor revision.

Introduction

The method specified in this International Standard is based on an oxidation of the plutonium followed by weighing. If the content of impurities is measured, a correction is made to allow for them.

Respecting certain conditions, the overall standard deviation on a single determination (gravimetric determination and impurities correction) can be below 0,1 %.

Nuclear fuel technology — Determination of plutonium content in plutonium dioxide of nuclear grade quality — Gravimetric method

1 Scope

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 a mass fraction of less than 0,65 % of non-volatile impurities.

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

2 Principle

The method specified in this International Standard consists of the following:

a) sampling and weighing of the sample in dry atmosphere;

b) heating in air between 1 200 °C and 1 250 °C to constant mass in order to obtain a stoichiometric plutonium dioxide, which is stable and non-hygroscopic;

c) weighing of the plutonium dioxide;

d) impurity analysis and correction for non-volatile impurities;

e) calculation of plutonium concentration;

f) calculation of the plutonium content using a gravimetric conversion factor which depends slightly on the isotopic composition of the plutonium.

If the latter is not known, it shall be measured, usually by mass spectrometry.

3 Interferences

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 non-volatile impurities content is of a mass fraction of up to 0,1 %, the overall uncertainty of the measurement will depend on the precision of the impurities determination.

4 Apparatus

4.1 Sub-sampling station, comprising a glove box under dry atmosphere (dew point less than or equal to −40 °C) equipped with an analytical balance accurate to ± 0,1 mg.

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

4.3 Stainless steel sampling vials.

4.4 Platinum crucibles.

4.5 Desiccators.

5 Procedure

5.1 Handling of the sample at the sampling station

5.1.1 Transfer at least 10 g of the material to be analysed into a vial (4.3).

5.1.2 Hermetically seal the vial.

5.1.3 Transfer the vial rapidly to the sub-sampling station (4.1).

5.2 Tarring of crucibles

5.2.1 Heat a clean crucible (4.4) for 1 h at 1 200 °C to 1 250 °C. Cool for 20 min in the desiccators (4.5) and then for 5 min in the balance (4.1 a), weigh to within ±0,1 mg. Repeat the heating until the mass remains constant to within ±0,1 mg.

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

5.3 Sub-sampling

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

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

5.3.3 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.

5.3.4 If the change in mass of the first sub-sample is less than 0,1 mg, transfer the sub-samples to the heating box (4.2). If this is not the case, discard the sub-samples, adjust the hygrometry of the box, and repeat the sampling and the procedure.

5.4 Heating

5.4.1 Heat the 1,5 g sample for 1 h at 1 200 °C to 1 250 °C.

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

5.4.3 Repeat 5.4.1 and 5.4.2 until the mass remains constant to within ±0,1 mg.

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

5.5 Additional measurements

5.5.1 Perform an isotopic analysis of plutonium to calculate its mean relative atomic mass, A_r(Pu).

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

6 Expression of result

6.1 Calculation of the gravimetric conversion factor

Calculate the gravimetric conversion factor using Formula (1).

$$C_{\mathrm{Pu}} = \frac{A_{\mathrm{r}}(\mathrm{Pu})}{A_{\mathrm{r}}(\mathrm{Pu}) + 2A_{\mathrm{r}}(\mathrm{O})} \tag{1}$$

where

$A_{\mathrm{r}}(\mathrm{O}) = 15{,}9994$ is the relative atomic mass of oxygen;

$A_{\mathrm{r}}(\mathrm{Pu})$ is the mean relative atomic mass of plutonium calculated using Formula (2).

$$A_r(\mathrm{Pu}) = \frac{1}{\frac{m_{238}}{238{,}050} + \frac{m_{239}}{239{,}052} + \frac{m_{240}}{240{,}054} + \frac{m_{241}}{241{,}057} + \frac{m_{242}}{242{,}059} + \frac{m_{244}}{244{,}064}} \tag{2}$$

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

6.2 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 grams, in the heated sample using Formula (3).

$$I_0 = 10^{-6} \times (m_2 - m_1) \times \sum_n (I_n C_n) \tag{3}$$

where

$m_2 - m_1$ is the mass of the sample before heating;

m_2 is the gross mass before heating, in grams (sample plus crucible);

m_1 is the mass of the crucible, in grams;

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

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

NOTE Depending on the context in which the results are to be used, mass (m_2 – m_1) can require standard corrections for air buoyancy effects.

6.3 Calculation of plutonium concentration

Calculate the plutonium concentration, *Pu*, as a percentage, in the sample using Formula (4).

$$\mathrm{Pu} = C_{\mathrm{Pu}} \times \frac{m_3 - m_1 - I_0}{m_2 - m_1} \times 100 \tag{4}$$

where

m_3 is the gross mass after heating (sample plus crucible), in grams.

6.4 Repeatability

The standard deviation for a single gravimetric determination is about 0,05 %.

In order for the standard deviation of the impurity correction factor to stand below 0,1 %, the impurities shall be measured to the following:

— with a standard deviation of 50 % (detection limit) up to 1 000 $\mu g \cdot g^{-1}$ of impurities;

— with a standard deviation of 25 % (semiquantitative analysis) up to 2 500 $\mu g \cdot g^{-1}$ of impurities;

— with a standard deviation of 10 % (quantitative analysis) up to 6 500 $\mu g \cdot g^{-1}$ of impurities.

In these conditions, the overall standard deviation on a single determination (gravimetric determination and impurities correction) is below 0,1 %.

6.5 Systematic errors

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

6.5.2 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 %.

6.5.3 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 can cause a systematic error of up to 20 % of the total impurity concentration.

7 Test report

The test report shall include the following information:

a) identification of the sample;

b) reference of the method used;

c) results of the measurement and the associated overall uncertainties, impurities percentage, and method of expression used;

d) unusual features noted during the test;

e) operations not included in this International Standard (i.e. ISO 8300).

Annex A
(informative)

Gravimetric conversion factor for the non-volatile impurities

Table A.1 — Gravimetric conversion factor for the non-volatile impurities

Impurity	Probable shape of the impurity	Conversion factor C_n
Ag	Ag	1,00
Al	Al_2O_3	1,89
Am	AmO_2	1,13
B	B_2O_3	3,22
Ba	BaO	1,12
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
Na	Na_2O	1,35
Ni	Ni_2O_3	1,40
P	P_2O_5	2,29
Pb	PbO	1,07
Rare earth	M_2O_3	1,16
Sb	Sb_2O_3	1,20
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
V	V_2O_5	1,78
W	WO_3	1,26

Table A.1 *(continued)*

Impurity	Probable shape of the impurity	Conversion factor C_n
Zn	ZnO	1,24
Zr	ZrO_2	1,35

NOTE This information is deduced from the most reliable available information, taking into account the calcinations and the cooling conditions and the matrices effects due to plutonium oxide. The chemical shape of the impurities is not well known and, consequently, the maximum amount of impurities is fixed at 0,65 % of the Pu mass.

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