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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