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Determination of plutonium content in plutonium dioxide (PuO₂) of nuclear grade quality — Gravimetric method

*Détermination de la teneur en plutonium dans du dioxyde de plutonium (PuO₂) de qualité
nucléaire — Méthode gravimétrique*

Reference number
ISO 8300:1987 (E)

Foreword

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International Standard ISO 8300 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*.

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Determination of plutonium content in plutonium dioxide (PuO₂) 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 (PuO₂) 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 $-40\text{ }^{\circ}\text{C}$). (The dew point shall be verified by using a measuring instrument and by checking the stability of the mass of an aliquot portion of PuO₂.) Ignition in air between 1 200 and 1 250 $^{\circ}\text{C}$ 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 $^{\circ}\text{C}$. 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 $^{\circ}\text{C}$ 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 $-40\text{ }^{\circ}\text{C}$) equipped with

- a) an analytical balance accurate to $\pm 0,1\text{ mg}$;
- 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 $^{\circ}\text{C}$.

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 $^{\circ}\text{C}$. Cool for 20 min in the desiccator (5.5) and then for 5 min in the balance [5.1 a)]; weigh to within $\pm 0,1\text{ mg}$. Repeat the ignition until the mass remains constant to within $\pm 0,1\text{ mg}$.