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Guidelines for plutonium dioxide (PuO_2) sampling in a nuclear reprocessing plant

Lignes directrices pour l'échantillonnage du dioxyde de plutonium (PuO_2) dans une usine de retraitement

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11482 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

Annexes A and B of this International Standard are for information only.

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Guidelines for plutonium dioxide (PuO_2) sampling in a nuclear reprocessing plant

1 Scope

This International Standard gives guidelines to ensure that the homogenization and the sampling system supply representative samples from a batch of plutonium oxide and to ensure that the said sample remains representative until aliquoting. Possible mass change of the sample during transport and storage is also considered.

These guidelines apply to PuO_2 powder with a specific surface area lower than 25 m^2/g.

2 General

The methods and installations used in reprocessing plants vary considerably and it is impossible to establish standardized procedures for the homogenization and the sampling systems.

Because of the physico-chemical properties of the plutonium oxide, numerous factors can yield samples which are not representative of the batch. Special attention should be given to the following:

a) The processing line between the homogenizer and the packing station, as well as the sampling line shall be completely emptied between the preparation of batches so as to avoid one batch contaminating the following one.

b) The chemical composition of plutonium oxide powders is prone to change with time: according to its physical state and the conditions under which it was manufactured, the oxide can increase or decrease in mass during the course of the canning and sampling operations, even in glove boxes supplied with dry air. The kinetics of the mass changes depend on the technological conditions of the installations used and in particular:

— on the dew point of the air or of the ventilation gas;

— on the air (gas) pressure within the glove box;

— on the operating conditions;

— on the previous history of the plutonium oxide.

In glove boxes supplied with dry air or dry gas like Ar or N_2 (– 65 °C dew point) with a depression of 30 mm water and with 10 air changes per hour, the kinetics of mass increase are slow. They depend on:

— the initial concentration of volatile impurities which is defined as being the mass loss after heating in a furnace for 1 h at 135 °C;

— the specific surface area.

For a specific surface area of 7 m^2/g and an initial concentration of volatile impurities of 0 % on leaving the oxalate calcining furnace, the oxide gains mass at a rate of about 0,015 % per 24 h.

c) Under the operating conditions which have just been described, the mass increase is negligible when the initial concentration of volatile impurities is less than 0,3 %. Mass losses are observed during the calcining of the sample; they become larger as the initial concentration of volatile impurities increases. For an initial volatile impurity concentration of 0,67 %, a mass loss of 0,37 % is observed in 24 h.

d) It is therefore evident that an installation in which one is to sample plutonium oxide must first of all be tested. For a plutonium oxide of known specific surface area and known concentrations of

volatile impurities, mass change curves as a function of time shall be plotted.

e) This assumes a well controlled installation with regard to dew point, depression and air changes. No transfer of equipment shall be made into the glove box within 1 h of these operations. The installation for the subdivision of the sample shall operate under the same technological conditions as the sampling installations. The term subdivision designates all the operations which must be done to weigh all the aliquots necessary for the complete analysis of the batch.

f) The homogenizer itself shall also be tested: the necessary operating time required must be defined in order to be certain of having a homogeneous batch. This operation can be carried out by filling the homogenizer with precisely weighed masses of plutonium oxide having very different isotopic compositions, sampling after successive homogenization and measuring the isotopic composition or the neutron emission of these samples.

3 Sampling

The requisite number of samples required for the following operations shall be taken:

— verification of the representativeness in relation to the batch;

— performance of the mass-balance analysis and specification analysis of the oxide.

3.1 Verification of the representativeness in relation to the batch

It is important that no influencing factors are transmitted from one batch to another via either the fabrication lines or the sample lines. This point is checked by setting up a validation exercise of the procedures for emptying the canning and sample lines. The isotopic composition of a sample taken from the first container which has been filled is compared to the isotopic composition measured on the batch representative sample. The homogeneity of the batch is checked by measuring the minor plutonium isotope on a set of samples taken successively during the PuO_2 canning.

3.2 Sampling for analysis

The sampling takes place during the canning of a batch and it should be representative of the batch. The term canning applies here to all the operations which take place after the oxide leaves the homogenizer and until the last can is filled. These operations last a variable length of time which depends on the size of the batches. For a 50 kg batch, this time is estimated to be 24 h. During the course of these operations the flow of the oxide shall be sampled at regular intervals. The technological conditions (dew point, depression, etc.) shall be the same for the flow of the oxide and for the sample.

3.3 Sample subdivision

The sample subdivision consists of very accurately weighing masses of plutonium oxide which are placed in transfer containers and which will then be used for the different analytical determinations. Before carrying on sample subdivision, the bulk powder which has been sampled is to be homogenized inside the subdivision glove box. An example of the procedure to be used for the subdivision operations is given in annex A.

4 Operating constraints

Taking into account the labile nature of the oxide powder, the following constraints have to be maintained in order to preserve the representativeness of the sample in relation to the batch:

a) For an installation of defined technological parameters, a mass increase graph shall be plotted for a plutonium oxide at a given specific area having an initial concentration of volatile impurities close to 0 % and it shall be plotted up to a constant mass: then measure the concentration of volatile impurities present in the powder at equilibrium. This equilibrium depends on the storage atmosphere of PuO_2 (temperature, moisture, depression).

b) The fabrication process shall be conducted in such a manner that, on leaving the homogenizer, the concentration of volatile impurities is below that which corresponds to equilibrium.

c) The specific surface area has a great influence on the mass change during handling operations.

d) In order to avoid ambient air entering, no transfer operations shall be made into or out of the glove boxes during the sampling operations.

5 Type of vials recommended for the shipment of small samples

For the shipment of small samples it is possible to use:

— the vials which are utilized by the International Atomic Energy Agency (IAEA), such as stoppered Erlenmeyer flasks contained in special plastic packaging, provided their use is approved by the plant safety officer;

— stainless steel containers which are commonly utilized by EURATOM.

6 Sample weight variation

For samples and aliquots for analysis:

— the container tare and the sample mass shall be measured to an accuracy of 10^4 g when the sample is taken, and the result of the measurement shall be transferred to the laboratory in charge of the analysis;

— on receipt of the sample and before using it, the laboratory shall weigh the sample and the container tare to an accuracy of 10^4 g;

— the laboratory shall take into account the possible mass change on the PuO_2 sample, due to transportation and storage.

Annex A
(informative)

Example of procedure to be used for the subdivision of sample

Nine aliquots of plutonium, each weighing more than 1 g, are prepared in the subdivision glove box and are used as follows:

— one aliquot is used as a test specimen for following the variation of mass: it will be weighed at the beginning and again at the end of sample subdivision operations. Its mass should not change more than 0,1 %. If it does, all masses have to be corrected;

— two aliquots for carrying out amperometric (ISO 8298) or coulometric (ISO 12183) plutonium determinations and the isotopic analysis of the plutonium oxide, as sampled;

— two aliquots for carrying out amperometric (ISO 8298) or coulometric (ISO 12183) plutonium determinations on the plutonium oxide after ignition at 950 °C for 4 h in a platinum boat;

— two aliquots for carrying out gravimetric plutonium determinations (ISO 8300) on the plutonium oxide, as sampled;

— two aliquots for carrying out gravimetric plutonium determinations (ISO 8300) on the plutonium oxide after ignition at 4 h in a platinum boat.

The different aliquots correctly identified are then transferred to the analytical facilities where the whole aliquots are used for each of the relevant assay, with mass measurements of the container tare and aliquots.

If the subdivision is performed far from the conditioning facility, the batch to be divided into fractions shall be sent to the subdivision glove box in tight containers.