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Determination of solubility in nitric acid of plutonium in unirradiated mixed oxide fuel pellets (U,Pu)O_2

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Détermination de la solubilité dans l'acide nitrique du plutonium des pastilles (U,Pu)O_2 *de combustibles d'oxydes mixtes non irradiés*

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Foreword

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Determination of solubility in nitric acid of plutonium in unirradiated mixed oxide fuel pellets $(U,Pu)O_2$

1 Scope

This International Standard specifies an analytical method for determining the solubility in nitric acid of plutonium in whole pellets of unirradiated mixed oxide fuel (light water reactor fuels). The results provide information about the expected dissolution behaviour of irradiated pellets under industrial reprocessing conditions.

2 Principle

A specified number of mixed oxide pellets of known plutonium content and mass are dissolved in a boiling nitric acid solution. The initial concentration of the nitric acid, the final concentration of U, Pu and the boiling time are carefully controlled. The undissolved residue is then dissolved quantitatively by boiling in a mixture of nitric acid and hydrofluoric acid. The plutonium content of this residue is determined by an appropriate analytical method. The solubility is expressed by the ratio of the amount of plutonium dissolved in nitric acid to the amount of plutonium in the sample.

3 Interferences

The dissolution apparatus (5.2) and the reagents shall not be contaminated with fluoride, as fluoride can cause an increase of the solubility of the pellet in nitric acid. The analytical method for the determination of plutonium shall be chosen in order to avoid interferences that could cause an understatement of the plutonium content. Such a method has to be qualified on representative solutions. The method specified in ISO 9463[1] is suitable.

4 Reagents

Use only reagents of analytical grade and distilled or demineralized water or water of equivalent purity. Prepare the reagents in compliance with the local laboratory safety instructions.

4.1 Concentrated nitric acid, $\rho = 1{,}40$ g/ml or more.

4.2 Nitric acid, solution $[c(HNO_3) = 5{,}5$ mol/l$]$.

4.3 Nitric acid, solution $[c(HNO_3) = 0{,}5$ mol/l$]$.

4.4 Concentrated hydrofluoric acid, $\rho = 1{,}13$ g/ml.

4.5 Mixture of nitric acid, solution $[c(HNO_3) = 14{,}4$ mol/l$]$ and **hydrofluoric acid**, solution $[c(HF) = 0{,}05$ mol/l$]$.

4.6 Sodium hydroxide, solution $[c(NaOH) = 2$ mol/l$]$ (optional).

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Analytical balance, with a 0,1 mg accuracy.

5.2 Dissolution apparatus, consisting of dissolver flask (pyrex or polytetrafluoroethylene), heater, total reflux condenser, gas inlet tube (optional) and gas washer bottle (optional).

1) ISO 9463:1990, *Nitric acid feed solutions from reprocessing plants — Spectrophotometric determination of plutonium after oxidation to plutonium(VI).*

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5.3 Second dissolution apparatus (polytetrafluoroethylene), for the dissolution with nitric acid/hydrofluoric acid mixture.

5.4 Filter apparatus, consisting of a vacuum funnel and a nitric acid-resistant filter of a pore size less than 1 µm (e.g. Millipore 1 µm).

6 Sampling

The sample size shall be large enough to be representative of the lot. Only entire, undamaged pellets shall be included in the sample. The minimum sample size shall be chosen as a function of the pellet composition and the laboratory's detection limit and ability to detect a plutonium residue ratio of 10 g Pu per ton of metal.

The minimum sample size shall be 45 g when the mass fraction of plutonium

$$W_{Pu} = \frac{m_{Pu}}{m_U + m_{Pu}}$$

is smaller than 5 %, where m_{Pu} and m_U are the masses of plutonium and uranium in the sample.

The minimum sample size shall be 25 g when W_{Pu} is between 5 % and 10 %.

See 8.2 for the effect of sample size on repeatability.

7 Procedure

7.1 Preparation of the sample

Weigh the pellets and transfer quantitatively into the dissolver flask (5.2). In order to reach a heavy metal concentration of 1 mol/l, the total volume V, in litres, of nitric acid solution (4.2) to be used is given by:

$$V = \frac{m}{cM}$$

where

m is the sum of the mass of uranium and plutonium in the sample, in grams;

M is the average atomic weights of the metals, in grams per mole;

c is the concentration of the uranium and plutonium in the solution, in moles per litre (c = 1 mol/l).

7.2 Dissolution procedure

Introduce the calculated volume of nitric acid of solution (4.2) into the flask containing the sample pellets. Boil the contents of the flask under reflux for 6 h. Boiling should begin within 30 min. Cooling to below 50 °C should be ended within 30 min. To avoid a delay in cooling, compressed air may be passed through the solution. The arising nitrous vapour may be trapped in a gas washer bottle filled with sodium hydroxide solution (4.6).

7.3 Treatment of the residue

Quantitatively transfer the solution and the undissolved residue into the filter apparatus and filter the solution using the vacuum funnel. Thoroughly rinse the residue on the filter with nitric acid solution (4.3).

Repeat the test if the filtered residue contains fragments of pellets (see 7.5). A tare filter should be used if it is desirable to weigh the residue.

Place the filter and residue in a dissolver flask (5.3) and add a portion (25 ml minimum) of the nitric acid/hydrofluoric acid (4.5). Heat the acid to its boiling point and pursue ebullition under flux until the residue is completely dissolved. However, if a residue remains after 2 h, add 5 ml of hydrofluoric acid solution [c(HF) = 0,05 mol/l] and continue the dissolution. If a residue appears after a further 2 h, filter the solution and use an appropriate procedure (e.g. alpha-autoradiography) to verify that the residue does not contain plutonium. If it does, repeat the test.

7.4 Plutonium determination

Make up the solution resulting from the treatment of the undissolved residue to a known volume. Take samples and analyze their plutonium content by a suitable method. The method shall have an uncertainty of 10 % or better, even in the presence of higher level of impurities contained in the dissolved material. The method described in ISO 9463 is adequate. Other methods should first be qualified with representative samples.

7.5 Repeat solubility test

If visible pellet fragments are observed in the residue of the first dissolution test, perform a new test on a fresh sample. For such a repeat, the test duration (up to 18 h), concentration of nitric acid (up to 10 mol/l and (U + Pu) concentration (down to 0,9 mol/l) shall be defined prior to carrying out the test. The conditions of each test and its results should be reported.

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8 Expression of results

8.1 Method of calculation

The plutonium solubility, L, expressed as a mass fraction in percent, can be calculated from the following equation:

$$L = \left(1 - \frac{m_2}{m_1 W_1}\right) \times 100$$

where

m_1 is the mass of the sample, in grams;

m_2 is the mass of the plutonium in the residue, in grams;

W_1 is the mass fraction of the plutonium in the sample.

8.2 Repeatability

The repeatability of the results depends upon the mass fraction of the plutonium W_{Pu} and the sample size. For a material with a mass fraction of plutonium greater than 5 % and a sample size of 25 g, the standard deviation of the repeatability is smaller than 0,1 %.

For a mass fraction of plutonium less than 5 %, a minimum sample size of 45 g is needed to obtain a standard deviation of the repeatability smaller than 0,1 %. In order to avoid waste of material, the sample size may be reduced to between 18 g and 22 g with a mass fraction of plutonium less than 5 %. Then the standard deviation of the repeatability can be as large as 0,2 %.

9 Test report

The test report shall include the following information:

— date and place of the test carried out;

— identification of the sample;

— the reference of the analytical method used;

— the method of calculation of the results including the boiling time;

— the results and their units;

— any unusual features observed during the test;

— any operation regarded as optional.

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