

INTERNATIONAL  
STANDARD

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**Determination of solubility in nitric acid of  
plutonium in unirradiated mixed oxide fuel  
pellets (U,Pu)O<sub>2</sub>**

*Détermination de la solubilité dans l'acide nitrique du plutonium des  
pastilles (U,Pu)O<sub>2</sub> de combustibles d'oxydes mixtes non irradiés*



Reference number  
ISO 12184:1994(E)

## Foreword

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# Determination of solubility in nitric acid of plutonium in unirradiated mixed oxide fuel pellets (U,Pu)O<sub>2</sub>

## 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<sup>1)</sup> 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 [ $c(\text{HNO}_3) = 5,5$  mol/l].

**4.3 Nitric acid**, solution [ $c(\text{HNO}_3) = 0,5$  mol/l].

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

**4.5 Mixture of nitric acid**, solution [ $c(\text{HNO}_3) = 14,4$  mol/l] and **hydrofluoric acid**, solution [ $c(\text{HF}) = 0,05$  mol/l].

**4.6 Sodium hydroxide**, solution [ $c(\text{NaOH}) = 2$  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)*.