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Determination of isotopic content and concentration of uranium and plutonium in nitric acid solution — Mass spectrometric method

Détermination de la teneur isotopique et chimique en uranium et plutonium d'une solution d'acide nitrique — Méthode par spectrométrie de masse

Reference number
ISO 8299:1993(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 8299 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Sub-Committee SC 5, *Nuclear fuel technology*.

Annex A forms an integral part of this International Standard.

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Determination of isotopic content and concentration of uranium and plutonium in nitric acid solution — Mass spectrometric method

1 Scope

This International Standard specifies a method to determine the isotopic content and the concentration of uranium and plutonium in input solutions of irradiated fuels in light water reactors (boiling water or pressurized water) and in final products at spent fuel reprocessing plants. The method is applicable to other fuels, but the chemical separation and spike solution shall, if necessary, be adapted to suit each type of fuel.

2 Principle of the method

The method is based on isotope ratio measurements by thermal ionization mass spectrometry.

The isotopic composition of uranium and plutonium is determined through isotopic ratio measurements.

^{238}Pu is determined by means of alpha spectrometry if interferences from ^{238}U cannot be eliminated.

The sample is diluted in order to economize on the amount of spike needed and to minimize the level of biological protection which is necessary.

A highly accurate gravimetric dilution is necessary when elemental concentrations are measured.

Because plutonium tends to hydrolyse at low acidities, a solution of nitric acid (HNO_3) with a molarity greater than 1 mol/l should be used for the dilution of the sample.

When elemental concentrations are measured, accurately weighed quantities of spike isotopes are added in quantities comparable to the quantities of ^{238}U and ^{239}Pu isotopes in the diluted sample. Although ^{233}U and ^{242}Pu isotopes are normally used as spikes, other separated isotopes (e.g. ^{244}Pu, ^{239}Pu, ^{235}U and ^{236}U) can be used when available and appropriate.

The isotopic composition of the spikes and the isotopic abundance of the ^{238}U and ^{239}Pu isotopes in the sample shall be accurately known. If the ^{242}Pu and ^{236}U isotopes are used as spikes, it is also necessary to measure the isotopic abundance of these isotopes in the sample.

A chemical separation is necessary to eliminate interfering elements (e.g. ^{241}Am, fission products) following a reduction-oxidation cycle to ensure isotopic exchange between spikes and sample.

The isotopic composition of fresh product material is normally measured without purification.

Plutonium aged more than 1 week should be purified to remove ^{241}Am.

The method includes the following steps:

a) dilution by weight;

b) spike addition by weight;

c) isotope exchange chemistry;

d) ion exchange purification/separation;

e) preparation of filaments;

f) mass spectrometric measurements and calculation of isotopic and elemental concentrations.

Care shall be taken to avoid cross contamination. For this purpose, it is recommended that disposable laboratory ware (e.g. vessels and columns) be used. Throughout the method it is necessary to follow good analytical practices regarding cleanliness, accuracy of measurement, avoiding evaporation errors, etc.