International Standard

ISO 6863

Nuclear fuel technology — Preparation of spikes for isotope dilution mass spectrometry (IDMS)

Technologie du combustible nucléaire — Préparation de traceurs pour les analyses par spectrométrie de masse avec dilution isotopique (IDMS)

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This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 5, *Nuclear installations, processes and technologies*.

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Nuclear fuel technology — Preparation of spikes for isotope dilution mass spectrometry (IDMS)

1 Scope

This document specifies a method which applies to the preparation and validation of the standard materials generally called “large size spikes” with an uncertainty suitable for international nuclear safeguards used for measuring the content of plutonium and/or uranium by isotope dilution mass spectrometry.

This measurement methodology can be applied to input solutions of irradiated Magnox and light water reactor fuels (boiling water reactor or pressurized water reactor); in final products at spent-fuel reprocessing plants; in feed and products of mixed oxide of plutonium and uranium (MOX); and in uranium fuel fabrication.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 8299, *Nuclear fuel technology — Determination of the isotopic and elemental uranium and plutonium concentrations of nuclear materials in nitric acid solutions by thermal-ionization mass spectrometry*

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3 Terms and definitions

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For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
solution spike
nitric acid solutions with accurately quantified uranium and/or plutonium content and isotopic composition

Note 1 to entry: One of the reference materials for IDMS.

3.2
dried spike
prepared by aliquoting *solution spikes* (3.1) into glass vessels and then drying

Note 1 to entry: One of the reference materials for IDMS.

3.3
large-sized dried spike
LSD spike
dried spike containing both uranium and plutonium in milligram size, were originally developed to analyse high concentrated input solution

Note 1 to entry: One of the reference materials for IDMS.

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Note 2 to entry: The LSD spikes can simplify the sample preparation process with only one spiking step to the sample, which also contains uranium and plutonium, and reduce measurement uncertainty by less dilution factor than smaller sized spikes.

4 Principle

Element content measurement using thermal ionization mass spectrometry (TIMS) is made on a sample and mixture of the sample and a spike, consisting of an enriched isotope of the element to be analysed and determine element content by calculating the difference of isotopic composition before and after spike mixture. This method of measuring an element's content is called isotope dilution mass spectrometry (IDMS).

The isotopic compositions of the sample and spike is required to be significantly different. Therefore, it is desirable that spikes are composed of isotopes that are not present, or only minimally present, in the unspiked sample. It is necessary that the isotopic composition and the content of spikes be known or measured accurately and has small uncertainties because it reflects to the uncertainty of the final results, element content.

Chemically-pure compounds of separated plutonium or uranium isotopes are dissolved to prepare stock solutions of spikes, in general terms, in 3 mol/l to 7 mol/l nitric acid to obtain an optimized content based on its design to obtain reliable results. Aliquots of the plutonium and uranium spike stock solutions can be mixed to prepare mixed spikes. Aliquoted spikes are used for IDMS in solution state or after dried.

5 Design of spikes

As the uncertainty to be considered in determining the reliability of nuclear fuel material analysis techniques for safeguards, International Target value (ITV)[1] is decided for IDMS. It is desirable that spikes are used for IDMS after being optimized for the sample to be analysed. Different target values are set depending on the environment to be measured and spikes used. Spikes shall be designed so that measurement by IDMS can achieve the appropriate ITVs.

There are two types of spikes: solution spikes and dry spikes. Basically, either spike is fine. In general, choose which spike to prepare according to user needs.

5.1 Optimization of spikes

In IDMS, when the isotopic composition in the unknown sample and in the spike are significantly different, the measurement accuracy become higher. On the other hand, the isotopic composition of available certified reference materials for preparing spikes are limited. Therefore, it is important to design the plutonium and/or uranium composition of spikes by evaluating, in advance, the isotopic composition and the amounts of elements of spikes that can be obtained sufficient accuracy. The following sentences describe an example of calculations for optimizing spikes.

The following Formula (1) can be obtained by partially differentiating the theoretical equation of IDMS with sample to spike mass ratio, p[2].

$$\left[\frac{\sigma(p)}{p}\right]^2 = \frac{1}{p^2}\cdot\frac{(1+p)^2(1+\gamma KR_T)^2}{(R_S-R_T)^2(1+\gamma KR_S)^2}\cdot\varepsilon^2{R_S}^2 + \frac{(1+p)^2(1+\gamma KR_S)^2}{(R_S-R_T)^2(1+\gamma KR_T)^2}\cdot\varepsilon^2{R_T}^2$$
$$+\frac{1}{p^2}\cdot\frac{[pR_T(1+\gamma KR_S)+R_S(1+\gamma KR_T)]^2[p(1+\gamma KR_S)+(1+\gamma KR_T)]^2}{(R_S-R_T)^2(1+\gamma KR_T)^2(1+\gamma KR_S)^2}\cdot\varepsilon^2$$
$$+\frac{\gamma^2(R_T-R_S)^2}{(1+\gamma KR_T)^2(1+\gamma KR_S)^2}\cdot{\sigma^2}_K \qquad (1)$$

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