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**Nuclear energy — Isotopic analysis of  
uranium hexafluoride — Double-standard  
gas-source mass spectrometric method**

*Énergie nucléaire — Analyse isotopique de l'hexafluorure d'uranium —  
Méthode du double étalon pour la spectrométrie de masse avec source  
à gaz*

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Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.org](mailto:copyright@iso.org)  
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## 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.

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The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15647 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

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## Introduction

Isotopic measurements of uranium isotopes are of great importance to the uranium enrichment facilities, not only for an accurate determination of the  $^{235}\text{U}$  enrichment, but also for a determination of the minor isotopes  $^{234}\text{U}$  and  $^{236}\text{U}$ .

This method describes the measurement of the isotopes directly from the process gas  $\text{UF}_6$ .

The method can be used for the measurement of samples, but also for on-line measurements.

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# Nuclear energy — Isotopic analysis of uranium hexafluoride — Double-standard gas-source mass spectrometric method

## 1 Scope

This International Standard specifies a method of isotopic analysis of uranium hexafluoride (UF<sub>6</sub>) with <sup>235</sup>U concentrations less than or equal to 5 %<sup>1)</sup> by mass and <sup>234</sup>U and <sup>236</sup>U concentration between 0,001 % by mass and 1,5 % by mass. The method is in routine use to determine conformance to UF<sub>6</sub> specifications.

Uranium hexafluoride is a basic material to prepare nuclear reactor fuel. To be suitable for this purpose, the material must meet criteria for isotopic composition.

This International Standard is applicable if the mass spectrometer uses Faraday cups.

## 2 Terms and definitions

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

### 2.1 mass resolving power mass resolution

*R*

capability of a mass spectrometer to differentiate between masses, as defined by Equation (1):

$$R = \frac{m \times a}{\Delta m \times b} \quad (1)$$

where

*a* is the distance between the centres of peaks;

*b* is the peakwidth;

*m* is the mass;

$\Delta m$  is the mass difference.

## 3 Principle

The unknown sample and two standards are introduced in sequence into the mass spectrometer. One measuring cycle comprises measurements of standard 1, sample, standard 2 and sample in that order. The concentration of <sup>235</sup>U in the standards should not be more than 40 % above or below that of the sample. In the mass spectrometer, UF<sub>5</sub><sup>+</sup> ions of the various isotopes are focussed through a mass-resolving slit. The result of a measurement is the mole ratio of the ions containing <sup>234</sup>U, <sup>235</sup>U and <sup>236</sup>U to that of <sup>238</sup>U. The final result is the mean obtained from 100 measuring cycles. The computer programme calculates the mean values with rejection of possible outliers during the calculation procedure.

1) The limit up to 5 % is not given to the method itself but because of the license.

Because the standards have a known composition, the measured mole ratios permit a calculation of the isotopic composition of the sample. Memory corrections are applied, based on the measurement of the ratio of three standards before a measurement cycle. With a choice of two standards of approximately 2 % by mass and 4 % by mass  $^{235}\text{U}$ , the range from 0,7 % by mass to 5 % by mass  $^{235}\text{U}$  can be measured with a good accuracy and without influences of memory effect.

## 4 Apparatus

4.1 **Sector mass spectrometer**, with the following features and capabilities.

- a) The resolving power of the mass spectrometer is not less than 500 atomic mass units.
- b) The four collectors have slits for ions with masses of 329, 330, 331 and 333.
- c) The abundance sensitivity of the mass spectrometer is specified such that the contribution of mass 333 ( $^{238}\text{UF}_5^+$ ) to mass 331 ( $^{236}\text{UF}_5^+$ ) is less than  $1 \times 10^{-5}$ .
- d) The pumping system of the mass spectrometer analyser tube must maintain a pressure of less than  $1 \times 10^{-7}$  Pa with sample flowing in the ion source.

## 5 Procedure

### 5.1 Calibration with isotopic standards

Peak heights and peak half-heights are verified and eventually adjusted before calibration. Calibration is carried out by the measurement of certified standards with a lower and with a higher concentration against a certified  $\text{UF}_6$  standard. The standards shall also contain some  $^{234}\text{U}$  and  $^{236}\text{U}$ .

The relative  $^{235}\text{U}$  concentration shall not differ by more than 40 % from the lower and the higher certified standards.

The measurement takes place in sequence following:

- a) reference sample against the low standard;
- b) reference sample against the high standard.

### 5.2 Instrument description

#### 5.2.1 $\text{UF}_6$ inlet system

The inlet system, which is specially developed for  $\text{UF}_6$ , has high requirements with respect to the vacuum and to the cleanliness of the system. Reaction products and other contaminants will affect the results due to a high memory effect. After a measurement, the gas must be transferred quickly from the inlet system without leaving a residue. The vacuum of the inlet system should be adapted to the requirements for the vacuum in the spectrometer and should be less than  $1 \times 10^{-3}$  Pa (see 5.2.3).

#### 5.2.2 Vacuum system for sample preparation

Gas samples must be prepared with the system before a measurement takes place. This system also requires a good vacuum of less than  $1 \times 10^{-2}$  Pa to minimize memory effect.

### 5.2.3 Vacuum system of the mass spectrometer

A vacuum of less than  $1 \times 10^{-7}$  Pa is required here for the ionization and focusing of the ion beams. To minimize the memory effect, a cryo-system is required in which the non-ionized ions are removed from the source. (Cryo-cooled sources usually claim low, if not zero, memory effects.)

### 5.2.4 Ion source

After the ionization of the  $\text{UF}_6$  molecules, the ions are accelerated out of the source by a high voltage (2 000 v to 8 000 v) and arrive via the focussing system in the separation chamber.

### 5.2.5 Detection system

A detection system consisting of four Faraday-cup collectors is used for the simultaneous measurement of  $^{234}\text{U}$ ,  $^{235}\text{U}$ ,  $^{236}\text{U}$  and  $^{238}\text{U}$ . As a current of approximately  $10^{-10}$  A to  $10^{-11}$  A must be measured, a very high resistance of  $10^{10} \Omega$  is therefore required.

## 5.3 Sample preparation

- a) Start with a test programme. Use a certified reference sample. The memory correction factor is estimated by this programme and saved for further determinations.
- b) Connect the sample bottles to be measured containing not less than 5 g of  $\text{UF}_6$  to the free connection points.
- c) Clean the sample bottle by cooling with liquid nitrogen for 10 min, pump off the headspace of the sample bottle and wait for 10 min.
- d) If no presence of light components are present (the pressure indicator shows no pressure rise), the sample is ready to be measured. If light components are present, repeat procedure 5.3 c).
- e) Eventually open the hand-valves necessary to introduce the standards.
- f) Start the computer-controlled test programme.
- g) If the test results are approved, the samples will be measured using the computer-controlled programme for the required application.
- h) After a sample run has been terminated, the ratios and weight percents are calculated and printed out. Finish by pumping off and closing the hand-valves.

## 5.4 Reference material

The  $\text{UF}_6$  standards used were certified by the Institute for Reference Materials and Measurements (IRMM), in Geel, Belgium.

$\text{UF}_6$  standard reference materials can be obtained from institutes as IRMM or the National Institute of Standards and Technology (USA) or can be made from standard reference oxide materials by fluorination of the oxide material.

## 6 Calculation

The fundamental relationship for mass spectrometric analysis is given by Equation (2):

$$\frac{m}{e} = \frac{H^2 \times r^2}{2V} \quad (2)$$

where

- $m$  is the mass;
- $H$  is the magnetic field strength;
- $r$  is the radius of the orbit;
- $V$  is the high voltage;
- $e$  is the number of charges on the ion.

The intensity of the ions of the isotopes are measured and the ratios,  $F$ , are calculated from the peak heights. The peak height of  $^{238}\text{U}$  is set equal to 100.

The ratios are calculated in accordance with Equations (3) to (5):

$$F_{235} = \frac{^{235}\text{U}}{^{238}\text{U}} \quad (3)$$

$$F_{234} = \frac{^{234}\text{U}}{^{238}\text{U}} \quad (4)$$

$$F_{236} = \frac{^{236}\text{U}}{^{238}\text{U}} \quad (5)$$

The "double standard method" has a high accuracy if the ratio is calculated by the weighted average and if the relative  $^{235}\text{U}$  concentration of the standards does not differ by more than 40 % from the sample. By choosing a standard as high as possible with respect to the sample, the memory effect is minimized.

The calculated ratio by weighted average,  $X$ , is determined in accordance with Equation (6):

$$X = \frac{(B \times X_{\text{ref}A}) - (A \times X_{\text{ref}B})}{(X_{\text{ref}A} - A) + (B - X_{\text{ref}B})} \quad (6)$$

where

- $A$  is the ratio of the low standard;
- $B$  is the ratio of the high standard;
- $X_{\text{ref}A}$  is the ratio of  $X$  measured against  $A$ ;
- $X_{\text{ref}B}$  is the ratio of  $X$  measured against  $B$ .

From the calculated ratios, the mass fractions,  $N$ , in percent, is determined in accordance with Equation (7):

$$N_{235} = \frac{235,04 \times F_{235} \times 100}{235,04 \times F_{235} + 234,04 \times F_{234} + 236,05 \times F_{236} + 238,05} \quad (7)$$

$N_{234}$  and  $N_{236}$  are calculated from analogous equations.

## 7 Accuracy

The accuracy of one analysis, including error of the standard used, for  $^{235}\text{U}$ ,  $^{234}\text{U}$ ,  $^{236}\text{U}$  is shown in the table below. The error values established are based on numerous measurements (more than 1 000) of samples and standards performed on different mass spectrometers over a period of more than 10 years.

The confidence level is 95 %.

**Table 1 —Accuracy**

Isotope	Concentration mass percent	Accuracy percent
$^{234}\text{U}$	0,001 0 to 0,100 0	< 5
$^{235}\text{U}$	0,700 0 to 5,000 0	< 0,1
$^{236}\text{U}$	0,001 0 to 0,100 0	< 5

## 8 Test report

The test report shall include the following information:

- identification of the sample;
- method used by reference to this International Standard;
- results and the form in which they are expressed;
- any unusual features noted during the test;
- any operations not included in this International Standard, or regarded as optional.