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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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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

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

Introduction

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

This method describes the measurement of the isotopes directly from the process gas UF_6.

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

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_6) with ^{235}U concentrations less than or equal to 5 %[1] by mass and ^{234}U and ^{236}U concentration between 0,001 % by mass and 1,5 % by mass. The method is in routine use to determine conformance to UF_6 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):

$$R = \frac{m \times a}{\Delta m \times b} \tag{1}$$

where

a is the distance between the centres of peaks;

b is the peakwidth;

m is the mass;

Δm 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 ^{235}U in the standards should not be more than 40 % above or below that of the sample. In the mass spectrometer, UF_5^+ 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 ^{234}U, ^{235}U and ^{236}U to that of ^{238}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.

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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}U, the range from 0,7 % by mass to 5 % by mass ^{235}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 ($^{238}UF_5^+$) to mass 331 ($^{236}UF_5^+$) is less than 1×10^{-5}.

d) The pumping system of the mass spectrometer analyser tube must maintain a pressure of less than 1×10^{-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 UF_6 standard. The standards shall also contain some ^{234}U and ^{236}U.

The relative ^{235}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 UF_6 inlet system

The inlet system, which is specially developed for 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 1×10^{-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 1×10^{-2} Pa to minimize memory effect.

5.2.3 Vacuum system of the mass spectrometer

A vacuum of less than 1×10^{-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 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}U,^{235}U,^{236}U and ^{238}U. As a current of approximately 10^{-10} A to 10^{-11} A must be measured, a very high resistance of 10^{10} Ω 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 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 UF_6 standards used were certified by the Institute for Reference Materials and Measurements (IRMM), in Geel, Belguim.

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.