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Determination of uranium content in samples coming from the nuclear fuel cycle by L-absorption edge spectrometry

Détermination de l'uranium dans les solutions du cycle du combustible nucléaire par absorption de rayons X à la discontinuité L

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy, nuclear technologies, and radio protection*, Subcommittee SC 5, *Analytical methodology in the nuclear fuel cycle*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Determination of uranium content in samples coming from the nuclear fuel cycle by L-absorption edge spectrometry

1 Scope

This document specifies a method for the determination of uranium concentrations in nitric acid or TBP-DILUANT (for example TBP-kerosene) solutions coming from the nuclear fuel cycle.

The method is applicable

— for process control of solutions, free of suspension, which contain between 10 g/l to 300 g/l uranium, and

— for high accuracy purposes (Safeguards) to nitric acid solutions, free of suspension, which contain between 100 g/l and 220 g/l uranium.

Having

— the content of neptunium and plutonium impurities in the solution less than 1 % of the uranium content.

— the content of neutron poisons (gadolinium, erbium) less than 1 % of the uranium content to ensure the absence of significant interferences at the level of required precision, for high accuracy purposes.

The method is applicable to solid samples as well, provided that they can be fully dissolved in nitric acid.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological 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/

4 Principle

A highly collimated X-ray beam passes through a uranium solution with well-defined path length. The transmission spectrum is recorded with a solid-state detector. A sharp jump of the photon transmission, which is related to the concentration of uranium, occurs at the L-edge energy of uranium (E_{LIII} = 17,17 keV). Uranium concentration is determined from the size of the jump using calibration and spectrum processing algorithms.

The proposed spectrum processing algorithms require the acquisition of reference spectrum to cancel out the influence of the matrix.

For high accuracy measurement, the isotopic composition of uranium and the temperature shall be known and corrections may apply.

The uncertainty of the number of counts in the channels before and after the L-edge is one of the main contributors to the measurement uncertainty.

The acquisition time needed to reach the necessary number of counts depends of the total count rate. The total count rate is a function of the intensity of the X-ray generator, of the characteristic of instrument, and of the concentration of uranium.

It is therefore specific to the laboratory, which shall evaluate beforehand the fit-for-purpose accuracy that will decide the target total count in the spectrum, the count rate and the acquisition time.

For high accuracy measurements, a minimum total number of counts of 2 000 000 in the fitting window is required to ensure satisfactory statistics around the L-edge. The count rates and measurement times given as indication in the document reflect this requirement. The reference, calibration and sample spectra shall be recorded with the same count rate and the same high voltage (HV) cut-off.

For process control purposes, the necessary total number of counts will depend of the needed accuracy. The measurement should be performed at the same HV cut-off and current.

5 Reagents and materials

Only analytical grade reagents shall be used.

All aqueous solutions shall be prepared with distilled water or deionized water.

5.1 Nitric acid solutions, $c(HNO_3) \approx 3$ mol/l, prepared from $w(HNO_3)$ 65 %, $\rho = 1{,}42$ g/cm^3.

5.2 Uranium reference solutions.

These solutions are used for calibration and quality control purposes. They should ideally be prepared from a solid or liquid material with known uranium concentration, and known uranium isotopic composition for high accuracy purposes. The standard uncertainty of the uranium concentration should be equal or better than 0,06 %. The use of certified reference material traceable to SI units is recommended.

6 Apparatus

The L-absorption edge spectrometer consists of an X-ray tube with its HV control unit, an X-ray detector, a multichannel analyser (MCA), a temperature sensor and software for data acquisition and processing. A principle diagram of the instrument components is shown in Figure 1.