
Simultaneous determination of uranium and plutonium in dissolver solutions from reprocessing plants — Combined method using K-absorption edge and X-ray fluorescence spectrometry

Dosage simultané de l'uranium et du plutonium dans les solutions de dissolution des usines de retraitement — Méthode combinée de spectrométrie d'absorptiométrie K et de fluorescence X



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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.

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 13464 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee 5, *Nuclear fuel technology*.

Annex A of this International Standard is for information only.

Simultaneous determination of uranium and plutonium in dissolver solutions from reprocessing plants — Combined method using K-absorption edge and X-ray fluorescence spectrometry

1 Scope

This International Standard specifies a nondestructive method for the simultaneous determination of volumetric uranium and plutonium concentrations in nitric acid feed solutions from reprocessing plants. The method is directly applicable, without interference, to the original feed solution, which typically contains about 150 g·l⁻¹ to 300 g·l⁻¹ uranium and 1 g·l⁻¹ to 2,5 g·l⁻¹ plutonium in the presence of fission products with a total β,γ -activity of up to 10 TBq·l⁻¹.

2 Principle

A sample vial containing about 3 ml to 5 ml of the feed solution is irradiated with a suitably filtered X-ray continuum from an X-ray tube. The X-ray continuum must include X-ray energies up to about 150 keV, and the maximum of its intensity distribution should occur at an energy above 100 keV.

The attenuation of a highly collimated X-ray beam, crossing a well-defined pathlength of solution in the vial, is measured with a HPGe detector below and above the K-absorption edge energy of uranium ($E_K = 115,6$ keV). The underlying K-edge densitometry (KED) method determines the uranium concentration from the abrupt change of the photon attenuation across the K-absorption edge.

At the same time a second HPGe detector, viewing a larger portion of the sample, measures the intensity ratio of fluoresced $K\alpha$ X-rays from uranium and plutonium. This X-ray fluorescence (XRF) measurement determines the U/Pu element ratio. The plutonium concentration is calculated from the measured values for the uranium concentration and the U/Pu ratio. The advantage of this XRF method arises from the fact that it is totally based on intensity ratio measurements. Further details on the measurement principle can be found in [1].

3 Apparatus

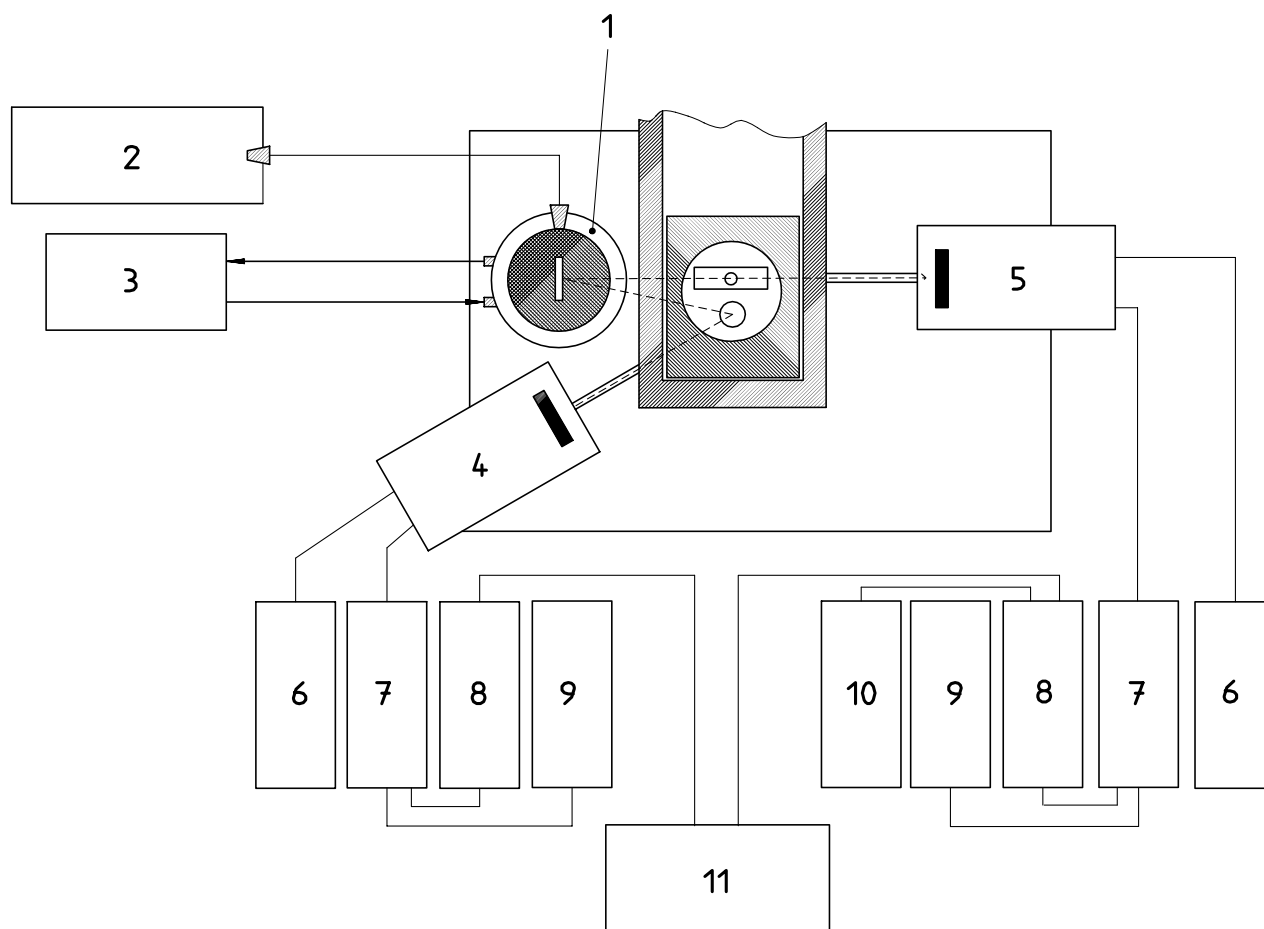
3.1 Components

3.1.1 Advanced standard spectroscopy equipment for high-resolution gamma spectroscopy including two HPGe detectors, electronics for fast pulse processing, and a computer-based multichannel analyzer for spectrum acquisition and evaluation.

3.1.2 X-ray equipment with X-ray tube, high voltage power supply, operating console and a circuit for water cooling.

3.1.3 Mechanics including beam collimators, shielding materials, adjustment device for the X-ray tube, and a system for transferring the sample into a defined position in the spectrometer.

A block diagram of the instrument components is shown in figure 1.

**Key:**

1	X-ray tube	7	Main amplifier
2	H.V. generator	8	ADC
3	Water cooling	9	Ratemeter
4	HPGe detector XRF	10	Stabilizer
5	HPGe detector K-edge	11	MCA and PC system
6	H.V.		

Figure 1 — Block diagram of the instrument components**3.2 Geometric arrangement**

The geometric arrangement (see figure 2) shall satisfy three basic requirements:

- The distance D between X-ray tube and sample shall be kept as short as technically feasible ($D \approx 6,5$ cm) in order to achieve the highest possible flux of X-rays from the tube at the sample position (integrated flux of X-rays with energies from 115 keV to 150 keV $\approx 5 \times 10^{10}$ photons·cm⁻²·s⁻¹).
- The angle θ between primary X-ray beam and collimator axis directed towards the XRF detector shall take values $\theta \geq 150^\circ$ in order to shift inelastically scattered radiation out of the energy region of analysis in the XRF spectrum.
- The solid angle subtended by the collimator between sample and K-edge detector shall be limited to values $\Omega \leq 10^{-4}$ sr in order to minimize elastic scattering towards the detector.

The transfer of the sample into the spectrometer can be accomplished either horizontally by means of a suitably designed sample conveyor system coupled to a shielded glovebox or hot cell facility (as indicated in figure 2), or vertically through a pneumatic sample transfer system.