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An inter-calibration campaign using various selected Pu spike isotopic reference materials

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The measurement of amounts of plutonium is recognised as one of the most important tasks in fissile material control. Public opinion is especially sensitive to this element, which presents analysts with a difficult task of measuring plutonium at all levels from large (multi-gram) amounts down to traces in the environment. The Institute for Reference Materials and Measurements (IRMM) is one of the few institutes worldwide that produces and certifies plutonium reference materials. They are part of a systematic IRMM programme to supply spike isotopic reference materials of various isotopes, in particular uranium and plutonium, at different concentrations.

Plutonium spike solutions present unusual problems that impinge on the certification of the isotope amounts. Due to radioactive decay certified values have to be constantly corrected, a process that leads to an increase in uncertainty depending on the spike isotope composition. The solutions also have a possibility of being unstable over time, probably due to radiolysis of the solutions.

At IRMM plutonium spikes are available for the isotopes ²³⁹Pu, ²⁴⁰Pu, ²⁴²Pu and ²⁴⁴Pu. This enables us to make an inter-comparison of the values of these spikes. An Intercalibration campaign using state-of-the art measurement procedures has therefore been carried out at IRMM linking the plutonium spike reference materials available at the institute. The following spikes were used in the program: IRMM-049c (94.1% ²⁴²Pu), IRMM-049b (99.9% ²⁴²Pu), IRMM-046b (94.3% ²⁴²Pu and 98.0% ²³³U), IRMM-083 (98.97% ²⁴⁰Pu) and IRMM-042a (97.9% ²⁴⁴Pu), IRMM-081a (97.8% ²³⁹Pu) from CETAMA were used.

As comparison material a recently prepared ²³⁹Pu large-sized dried (LSD) spike, IRMM-1027M and external isotopic reference materials NBL CRM-131 and CETAMA MP2 were used for isotope dilution mass spectrometry (IDMS).

The uncertainties for the IDMS were estimated according to the Guide to the Expression of Uncertainty in Measurement (GUM). The results obtained by IDMS using the various spike isotopic reference materials will be presented and discussed. This inter-calibration study confirms the traceability of the values of the plutonium isotopic contents to the SI. Furthermore conclusions can be drawn for the need of recertification of particular reference materials.

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