Contribution ID: **48** Type: **Poster**

A simple determination of 41Ca by ICPMS in concrete samples as a tool for the decommissioning of nuclear facilities

Monday, 12 May 2014 17:15 (1h 30m)

In October 2011 the European Joint research Project MetroRWM –Metrology for Radioactive Waste Management of the European Metrology Research and Program (EMRP) began, with a total duration of three years. One of the objectives of this project is to develop new measurement methodologies for the assessment of radioactive waste, focusing its attention on waste packages containing solid low and intermediate level radioactive waste consigned to near-surface or geological repository.
In the decommissioning of shielding concrete from nuclear facilities, the radionuclides of interest include 41Ca, 55Fe, 60Co, 63Ni, 133Ba and transuranics. Calcium-41 is of particular interest, due to its ubiquity in reactor bioshields, its bone-seeking chemistry and its radiotoxicity if ingested. Among these radionuclides gamma emitters can be easily determined using gamma-spectrometry. However for the determination of beta and alpha emitters, such as 41Ca, the radionuclides need to be separated individually from matrix and other radionuclides before being measured. Calcium-41 is a long-lived radionuclide produced by neutron activation in the concrete shield around the reactor, 40Ca (n,γ) 41Ca and displays high mobility in the environment and as well bone-seeking bioavailability. Calcium-41 decays to the ground state of 41K by pure electron capture, emitting X-rays and Auger electrons of very low energy (0.3-3.6 keV). Measurement by X-ray spectrometry is challenging due to the relatively low detection efficiencies encountered in this technique, the low abundance of X-rays from 41Ca (11.4 % for 3.31 keV X-ray) and the possibility of severe interferences produced by other beta-gamma radionuclides with similar energy. It can be measured by liquid scintillation but the procedure is time-consuming because all other radionuclides have to be removed due to the poor energy resolution of beta spectra and the low energy of its X-rays and Auger electrons.
Mass spectrometry techniques such as accelerator spectrometry (AMS) and resonance ionization mass spectrometry (RIMS) have also been used for the 41Ca determination but both of these techniques are expensive and not easily available for the routine analysis. The aim of this paper is to describe a rapid and reliable method for the determination of 41Ca in concrete samples by ICPMS, overcoming the complex and time consuming chemical separation of 41Ca from the matrix.
Samples are firstly ground and fused with LiBO2 and Li2B4O7 (in the presence of ~0.5% LiBr) and then dissolved in dilute acid. Then silicates are removed by means of PEG-precipitation. The acid solution can be analyzed directly, using the reaction cell mode, in order to avoid the polyatomic and isobaric interferences. Preliminary recovery tests using solutions of known concentrations of K and Ca were performed. The average recovery is 101.3% with a relative standard deviation of 3.2 % showing the method has good reproducibility

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Session Classification: Poster Session - Nuclear Analytical Methods

Track Classification: Nuclear Analytical Methods