SYNTHETIC MULTI-ELEMENT STANDARDS (SMELS) USED FOR THE VALIDATION OF K_0-NAA

Background

As more and more labs dealing with nuclear analytical techniques are working under a quality assurance system, the need for demonstrating the analytical quality of the method becomes more and more important. For non-standard methods, such as k_0-standardisation Neutron Activation Analysis (k_0-NAA), this means a full validation report demonstrating amongst others the bias and the reproducibility of the method and a continuous quality control using appropriate control material. In environmental laboratories using chemical techniques for example several multi-element liquid solutions are available for this purpose. For k_0-NAA a set of synthetic multi-element standards (SMELS) was made in cooperation with INW, Gent (B) and IRMM, Geel (B). The SMELS material is based on a phenol formaldehyde resin (Bakelite) that was spiked with 33 different elements. Three types of materials were produced:

- Type I: Au, Cl, Cs, Cu, I, La, Mn and V; forming short-lived radionuclides after irradiation;
- Type II : As, Au, Br, Ce, Mo, Pr, Sb, Th, Yb and Zn; forming medium-lived radionuclides ;
- Type III : Au, Co, Cr, Cs, Fe, In, Sb, Sc, Se, Sr, Th, Tm, Yb, Zn and Zr; forming long-lived radionuclides.

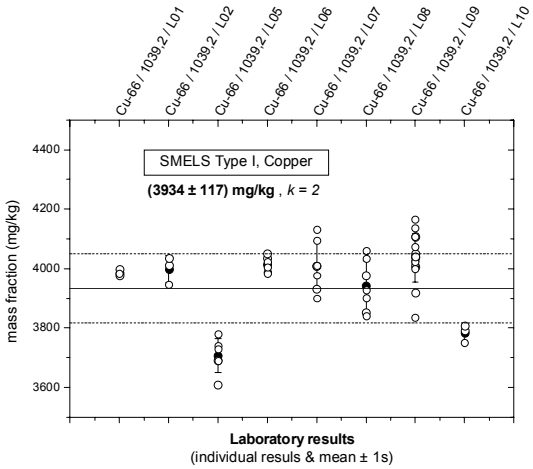
These elements have cross-sections and resonance energies with Q_0-values ranging from very low to very high and thus serve as quality control of the irradiation facility and the calibration of detectors.

The homogeneity of the material was demonstrated by IRMM for a minimum sample intake of 50 mg. In the reactor of Řež near Prague it was proven that the Bakelite matrix has an excellent radiation stability and this up to high fluence density of both thermal and fast neutrons (up to 1.1014 n.cm-2.s-1 and 3.1013 n.cm-2s-1 respectively).

Objectives

The last step before SMELS can be used by k_0-NAA laboratories is the elemental characterisation of the material. SCK•CEN took the initiative for this and organised in 2005 an inter-laboratory comparison comprising eight expert labs that already had participated extensively in international inter-comparisons at the highest level such as CCQM and thus their methods have been compared to other well validated techniques. The aim was to obtain consensus values (later called the "assigned" values) for the elemental concentration. Different NAA techniques were used: relative NAA, k_0-NAA, k_0-NAA with internal comparator or relative standardisation derived from the k_0-NAA approach.

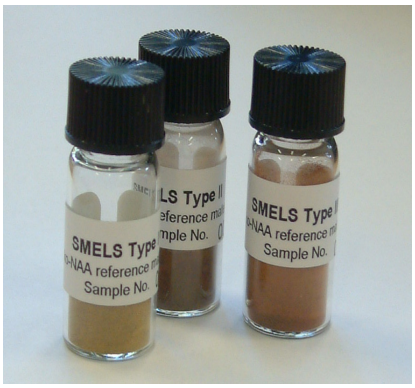
Principal results



Systematically we verified for all of the 33 elements, yielding about 40 radionuclides and about 60 gamma lines, that each result for specific nuclides and gamma lines corresponded well and if it was justifiable to pool all the results of each participant. This is e.g. illustrated in the figure for ^{65}Cu (n,γ) ^{66}Cu with a half-live 5.12 min and a main gamma energy peak at 1039.2 keV, which as irradiated for 5 min and counted for about 2 min on a 40 % HP Ge detector.

We have shown that the SMELS material is a unique material for the k_0-NAA users since it can be used for short, medium and long lived radio-isotopes and that it is fit for its purpose:

- it is an inert material
- it is a material with a well characterised inhomogeneity with a between-bottle reproducibility of less then 1 % for most elements;
- it is radiation stable even at very high fluxes;
- it has an elemental concentration with assigned values for up to 33 elements with an expanded uncertainty in the order of a few percent, except for Type II: Ce, Mo, Sb, Th and Yb, the maximum being 5,5 % for Yb.



On this basis we made a certificate for analysis for this material and labelled the bottles, as seen on the picture, ready for dispatching. Unfortunately there is only a limited number of bottles available for the scientific community, but the people involved in the production of SMELS decided that from September 2005 on, the material can be distributed freely to the k0-users. SCK•CEN will be the gatekeeper for this process.

Future work

The thermal to epithermal neutron flux ratio (*f*) and the deviation of the epithermal neutron spectrum from the 1/E shape (α) are essential parameters for the correct application of k_0-standardised neutron activation analyses. Several methods are applied for the determination of *f* and α. They are based on Cd-ratio or Cd-covered multi-monitor methods or on bare-irradiations methods. SMELS Type III contains Au and Zr, thus also allowing the direct determination of *f* and α with only this material. SMELS can also be used as a quality control material to monitor the irradiation facility and the detector, thus replacing the traditional flux monitors. Future work will present the accuracy of the *f* and α determination in channels Y4 and S84 of the BR1 reactor.

Main contact person

Peter Vermaercke, peter.vermaercke@sckcen.be

Main reference

P. Vermaercke, P. Robouch, M. Eguskiza, F. De Corte, G. Kennedy, B. Smodis, C. Yonezawa, H. Matsue, X. Lin, M. Blaauw, J. Kučera, *Nuclear Instruments and Methods in Physics Research Section A,* to be published in 2006