ELEMENTAL CHARACTERIZATION ANALYSIS OF DECOMMISSIONING MATERIALS FROM FiR1 TRIGA MARK II TYPE RESEARCH REACTOR

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INTRODUCTION

In June 2015, VTT's research reactor FiR 1 (TRIGA Mark II, General Atomics) was shut down and planning of its decommissioning was started. Prior to decommissioning, all different reactor materials are characterized in order to define radionuclide inventories and activities in different materials and subsequent waste streams. [1] As a part of FiR1's characterization, original chemical composition of lead, aluminium and graphite from the reactor was analysed with High Resolution sector Field Inductively Coupled Plasma-Mass Spectrometer (HR ICP-MS, Element 2, ThermoScientific).

SAMPLE PRETREATMENT AND DISSOLUTION

Suitable pretreatment and dissolution method for each material was tested and selected. Lead and aluminium samples were pretreated with 1 M HNO_3 and ethanol, in order to remove any possible contamination on the material surface. Graphite samples were not pretreated, because of its porosity. Suitable amount of pretreated lead or aluminium was weighed and dissolved completely with microwave (Milestone MLS 1200 Mega) assisted acid digestion method. Graphite samples were dissolved with traditional acid digestion method. *Table 1* shows the selected dissolution method for lead, aluminium and graphite samples.

Table 1. Selected dissolution methods for each sample matrix.

Matrix	Mass of Sample [mg]	Dissolution solution	Dissolution method
Lead	200	3 mL conc. HNO_3 + 3 mL MilliQ H_2O	Microwave assisted acid digestion
Aluminium	300	5 mL *aqua regia*	Microwave assisted acid digestion
Graphite	200	12 mL conc. H_2SO_4 + 4 mL conc. HNO_3 + 1 mL conc. $HClO_4$	Acid digestion

ELEMENTAL ANALYSIS

Goal was to find the important elements in each material, which can be activated in reactor's neutron flux and result in radioactive inventory and activity in the decommission wastes. Analysed elements were selected based on the theoretical info of the elemental composition of each material, neutron flux material has been exposed to and the main contributing radionuclides according to the inventory calculations [1]. *Table 2* shows the analysed elements in each matrix. Elemental analyses were performed from diluted sample solutions with HR ICP-MS equipment. Calibration and control samples were diluted from ICP-MS Multi-Element Solutions by SPEXCertiPrep®, Inorganic Ventures™ and AccuStandard®. All samples contained internal standard; 10 µg/L of In or Rh. The samples were injected through SeaSpray nebulizer (0.4 ml/min) and double pass spray chamber equipped with Peltier cooling unit. Aluminium sample cone and Nickel skimmer cone were used during the measurement.

Table 2. Elements analysed in each sample type with HR ICP-MS.

Sample type	Analysed elements
Lead	Ag, As, Bi, Ca, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Sb, Se, Sn, Zn
Aluminium	Ag, Al, As, B, Ba, Bi, Ca, Cd, Co, Cr, Cs, Cu, Fe, Ga, Ge, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Re, S, Sc, Se, Si, Sn, Sr, Ta, Th, Ti, Tl, U, V, W, Zn, Zr
Graphite	Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cs, Cu, Eu Fe, Ga, Ge, In, K, Li, Mg, Mn, Mo, Na, Ni, Nb, P, Pb, Re, Rb, S, Sc, Si, Sr, Ta, Th, Ti, Tl, U, Y, V, W, Zn, Zr

RESULTS

Figures 1 a, b and *c* shows the main elemental impurities in each analysed material.

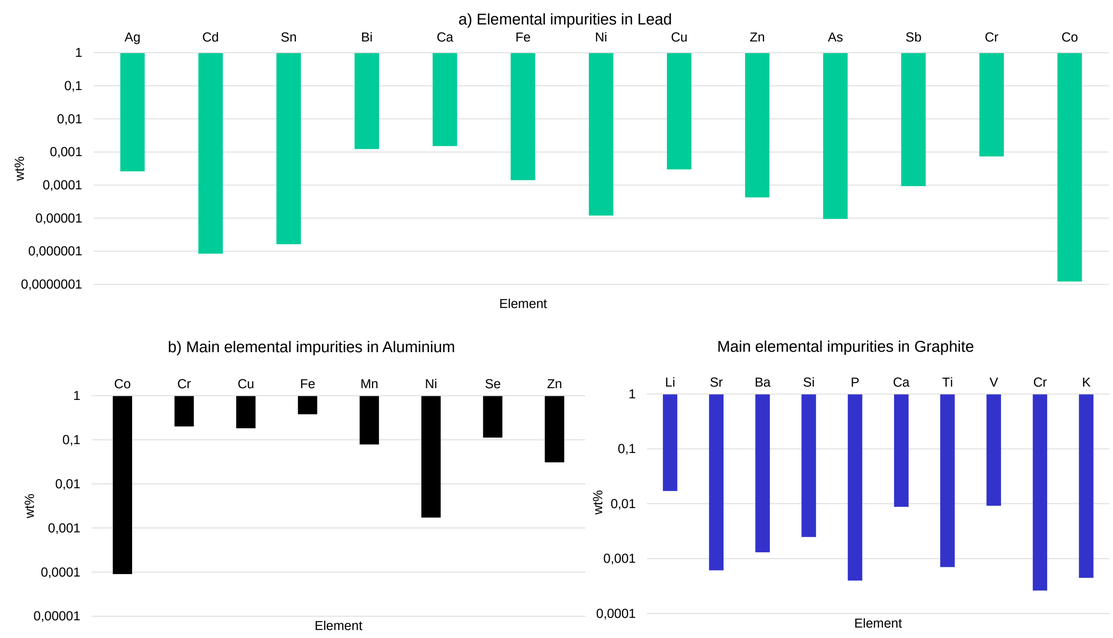

Figure 1. a) Elemental impurities in Lead. b) Main elemental impurities in Aluminium. c) Main elemental impurities in Graphite. In addition to these elements, also N and Eu are important elements, which activate and increase radionuclide inventory of the material. N cannot be analysed with HR ICP-MS technique, Eu will be determined in the near future.

Based on the measured elemental composition, gamma nuclide activities are calculated. Lead and aluminium calculation results were compared with the actual measured gamma activities and they were in a relatively good accordance with the actual measured gamma nuclide activities (*Table 3*). [1] Graphite results will be calculated and compared, in order to evaluate the analytical method.

Table 3. Lead and aluminium gamma nuclide calculation results compared with the actual measured gamma activities.

Element	Ag-110M	Sc-46	Cr-51	Mn-54	Fe-59	Co-60	Zn-65
Material	Lead	Aluminium	Aluminium	Aluminium	Aluminium	Aluminium	Aluminium
Measured gamma [Bq/g]	0.81	5.5	700	1.8	14	5.2	81
Calculated gamma [Bq/g]	0.93	7.4	640	4.1	21	18	134

Conclusions

- Elemental composition, especially main impurities of lead, aluminium and graphite samples were analysed with HR ICP-MS equipment.
- Based on measured elemental composition, gamma nuclide activities were calculated; Lead and aluminium gamma nuclide activities were in a relatively good accordance with the actual measured gamma nuclide activities.

Reference

[1] A. Räty et al., Preliminary waste characterization measurements in FIR1 TRIGA research reactor decommissioning project, Nuclear Technology, Accepted 21 Feb 2018, Published online: 06 Apr 2018, https://www.tandfonline.com/doi/abs/10.1080/00295450.2018.1445402

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