Contribution ID: **977** Type: **Verbal**

Feasibility study on the use of neutron activation analysis for nuclear forensics: Determination of elemental impurities in uranium materials after pre-irradiation removal of uranium

Thursday, 19 May 2022 09:00 (20 minutes)

In the framework of Collaborative Materials Exercises (CMX) organized by the Nuclear Forensics International Technical Working Group (ITWG) we received 4 radioactive samples S1 –S4 of unknown origin in two different physical forms (2 powders, 2 metal pieces), which contained unknown content of uranium with unknown enrichment and were asked to answer the following questions:
1) Do samples have the same material properties?
2) Is the material regulated by national law or international agreement?
3) Is a sample consistent with material having a known set of technical specifications?
4) Is a sample consistent with material resulting from a known activity or process? e.g. a particular application or step in the fuel cycle?
5) Is a sample consistent with materials information contained in a database or library?
6) Is the sample consistent with material from a particular origin?
In addition to classical forensic examinations, e.g., fingerprints on packaging materials, a variety of analytical methods, namely micro-Raman spectroscopy, XRF analysis, SEM-EDX analysis, XRD analysis, alpha-spectrometry, gamma-spectrometry, SIMS, and ICP-MS were used in the early stage of the exercise to determine the basic physical properties and chemical composition. To extend knowledge about elemental composition of the samples and to gain information about a possible linkage among the samples, NAA was also used in the later stage of the exercise for determination of minor and trace elements. Since neutron irradiation of the samples in a nuclear reactor would yield overwhelming activities of ^{239}U, its daughter ^{239}Np, and fission products, which would mask gamma-ray peaks of other nuclides arisen from neutron activation of minor and trace elements, a procedure of neutron activation analysis (NAA) with pre-irradiation separation of U using UTEVA resin (Triskem International, France) has been developed. The procedure consists in passing the samples dissolved in 3 M HNO_3 through three columns of UTEVA resin packed in 2-mL cartridges (pre-conditioned with 3 M HNO_3) in succession, washing the columns with 30 mL of 3 M HNO_3, reduction of the effluent volume to 5 mL, pipetting effluent aliquots into polyethylene (PE) irradiation capsules, and their evaporation to dryness. A uranium decontamination factor of ~ 10^6 was achieved. Thus, using NAA with both short- and long-time irradiation (90 s and 3 h, respectively), contents of the elements Na, Cl, Mn, Ca, Sc, V, Cr, Mn, Fe, Co, Zn, As, Sb, Ba, La, Ce, Sm, Eu, W, and U could be determined, while only limits of detection (LOD) could be evaluated for other 10 –15 elements. Simultaneously with the aliquots obtained after U removal, two types of blanks (PE capsules and a process blank) were analysed. Most operations with the samples during their preparation for NAA were carried out in a clean laboratory (Class ISO 6 ¬–ISO 5) using plastic ware pre-cleaned by washing in 3 M HNO_3 and rinsed with demi-water. For quality control of NAA (check of the multielemental calibrators), NIST SRM 1547 Peach Leaves was also co-irradiated. The usefulness of the NAA procedure developed for the collaborative exercise is discussed with special emphasis on accuracy and uncertainty of results and element LODs. The results for lanthanoids are also compared with those obtained by ICP-MS.

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Session Classification: Nuclear Analytical Methods

Track Classification: Nuclear Analytical Methods