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Analytical procedure for the determination of non-gamma-emitting radionuclides in radioactive waste

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A variety of aqueous radioactive wastes are generated during the operation of reactors and other nuclear fuel cycle facilities. The evaporation of low level effluents is a common operation that gives rise to such wastes in a form of evaporator's concentrates. Concentrated aqueous wastes from a number of sources have variety of chemical/radiochemical composition. Such solutions contain high concentration (10 ÷ 500) g·L-1 of dissolved salts. The radionuclides present in these wastes, also known as wet solid wastes, included fission products, activation products and actinides. Due to their composition and activity, they are adjusted to the form suitable for long-term storage in appropriate repositories.
The extraction chromatography is a good alternative for separation of radionuclides from these radioactive wastes.
The purpose of this study was to develop suitable and advanced analytical separation procedure of 237Np, 239,240Pu, 238Pu and 241Am for their determination in evaporator's concentrate which results from waste management operations at commercial Nuclear Power Plant.
The extraction chromatography with commercial available resin AnaLig®Pu02 was a method used for the separation of 237Np, 239,240Pu, 238Pu and 241Am from simulated evaporator's concentrate samples and actual evaporator's concentrate from Mochovce NPP, Slovakia. Currently, there are two units in operation at Mochovce NPP. The other two units are under construction. The reactors are of the Russian WWER-440 design. The predicted overall production of concentrates from four reactors at Mochovce NPP is 600 m3 per year. The evaporator's concentrate which is represented by fluids of dark brown colour belongs to the most difficult sample of analysis. Samples of WWER-type units contain a high content of boric acid with concentration of borates up to 100 g•L-1. Borates tend to precipitate from solution when there are changes of pH. Solubility of boric acid and borates generally increase with increasing temperature. At pH > 10, boric acid is in the meta borate form [B(OH)4]- with solubility of (14 ÷ 34) wt % from 0 to 50℃. The specific activity lies in the range between (3.7 ÷ 7.4) x 107 Bq·L-1. For this reason, treatment of the sample is very important. The pH of samples of model and actual solution of evaporator's concentrate was adjusted to ~ 13 by adding of NaOH because of higher solubility of borate solution.
There was a method developed on the model samples of 5 mL volume and hence applied on actual solution of evaporator's concentrate of the same volume as well as conditions of separation were optimised. Samples were spiked with 237Np, 241Am and 239Pu to determine the radiochemical yields of separation procedure. After evaporation, sample was dissolved in 4 M HNO3. AnaLig®Pu02 resin can only extract tetravalent actinides, oxidation states of monitored radionuclides were adjusted by adding of 0.5 g NaNO2 and sample was transferred to a column. The success of the separation process depends on choice of suitable elution agent. Americium was eluted with 8 M HNO3, plutonium with 0.5 M NH4I in 7 M HCl and neptunium with 9 M HCl with addition of TiCl3.
ORTEC 576A α-spectrometer equipped with ULTRATM ion implanted silicon detectors (600 mm2 active area) was used for counting the alpha radioactivity. The spectra were processed by using the Alpha-vision TM 32-bit emulation software from the EG&G ORTEC company.
This method reduces analysis time, cost, labour, organic solvent consumption and disposal and provides high radiochemical yields.

Key words: radioactive wastes, extraction chromatography, americium, plutonium, neptunium, tracer techniques, alpha spectroscopy

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