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Separation techniques for determination of actinides in various samples

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Separation methods for determination of actinides applied at the Jožef Stefan Institute are described. Soil and sediment samples were decomposed by conventional wet dissolution with mixtures of HNO3, HClO4 and HF acids, microwave dissolution using HNO3 and HF, and alkaline fusion with Na2CO3 and Na2O2. while preconcentration of actinides from liquid samples was carried out by precipitation using iron (III) hydroxide and ammonium hydrogen phosphate. The dry residue or precipitate with actinides and added tracers were then dissolved in 2M HNO2. After adjustment of the plutonium oxidation state to Pu (IV) in 2M HNO3 with 1.25M FeCl2, 1M NH2(OH)HCl and 1M NaNO3, the solution was adjusted to 8M using conc. HNO3 and the actinides selectively separated with a combination of ion exchange and extraction chromatography. This 8M HNO3 solution was passed through an anion exchange column prepared from Dowex 1X8, 100–200 mesh Cl- form resin. The 8M HNO3 effluent was used for analysis of americium and uranium radionuclides. The column was washed with 50 mL of 9M HCl to elute thorium. Pu was then eluted with 50 mL of 9M HCl/0.1M NH4I solution and neptunium was later stripped with 50 mL of 4M HCl solution. The 8M HNO3 effluen, containing the uranium and americium fraction was evaporated to dryness. The residue was dissolved in 2M HNO3 and the solution was transferred to sequential columns of Eichrom UTEVA and TRU preconditioned with 2M HNO3. The columns were washed with 20 mL of 2M HNO3. After that, columns were split. The UTEVA column was washed with 20 mL of 5M HCl/0.05M oxalic acid to remove impurities. Uranium was stripped from the UTEVA column with 15 mL of 1M HCl. The TRU column with americium was cleaned up with 5 ml of a mixture of 2M HNO3/0.1M NaNO2 and americium was stripped from the TRU column with 3 mL of 9M HCl and 20 mL of 4M HCl. The solutions with isolated radionuclides were evaporated to dryness and source preparation for alpha-particle spectrometry carried out by the microcoprecipitation method with neodymium fluoride. The methods were applied to reference materials and various real samples such as soil, sediments and water, all analysed in intercomparison exercises for determination of actinides.

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