Comparison of sample preparation methods for reliable plutonium and neptunium urinalysis using automatic extraction chromatography - DTU Orbit (31/12/2018)

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This paper describes improvement and comparison of analytical methods for simultaneous determination of trace-level plutonium and neptunium in urine samples by inductively coupled plasma mass spectrometry (ICP-MS). Four sample pre-concentration techniques, including calcium phosphate, iron hydroxide and manganese dioxide co-precipitation and evaporation were compared and the applicability of different techniques was discussed in order to evaluate and establish the optimal method for in vivo radioassay program. The analytical results indicate that the various sample pre-concentration approaches afford dissimilar method performances and care should be taken for specific experimental parameters for improving chemical yields. The best analytical performances in terms of turnaround time (6 h) and chemical yields for plutonium (88.7 +/- 11.6%) and neptunium (94.2 +/- 2.0%) were achieved by manganese dioxide co-precipitation. The need of drying ashing (>= 7 h) for calcium phosphate co-precipitation and long-term aging (5 d) for iron hydroxide co-precipitation, respectively, rendered time-consuming analytical protocols. Despite the fact that evaporation is also somewhat time-consuming (1.5 d), it endows urinalysis methods with better reliability and repeatability compared with co-precipitation techniques. In view of the applicability of different pre-concentration techniques proposed previously in the literature, the main challenge behind relevant method development is pointed to be the release of plutonium and neptunium associated with organic compounds in real urine assays. In this work, different protocols for decomposing organic matter in urine were investigated, of which potassium persulfate (K2S2O8) treatment provided the highest chemical yield of neptunium in the iron hydroxide co-precipitation step, yet, the occurrence of sulfur compounds in the processed sample deteriorated the analytical performance of the ensuing extraction chromatographic separation with chemical yields of

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