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Analysis of ultra-trace concentration of americium in sediments by HR-ICP-MS following a simple extraction chromatography separation

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Content

In radioecology, the measure of radionuclides in environmental samples is a key issue. The analysis of americium present at ultra-trace concentrations in sediments (˜mBq.kg-1) is often carried out by α-spectrometry after chemical separation. Americium is extracted from sample using a leaching process in 3M HNO3 then purified through co-precipitation and/or chemical separation using chromatographic extraction resins [1, 2, 3]. In general, the above procedure suffers from several drawbacks. It needs to use several resins, a long time consuming chemical separation step and a relatively long analysis time by α-spectrometry. The objectives of this study are to simplify the chemical separation procedure and to limit the analysis time using a high resolution plasma source mass spectrometry equipped with an APEX-Q (Elemental Scientific Inc.) high efficiency sample introduction system combined with a membrane desolvation unit (ACM). The separation is completed using a single chromatographic extraction column employing the N, N, N', N'-tetra-n-octyldiglycolamide (DGA) resin to separate ultra-trace concentrations of americium from the interfering polyatomic and isobaric elements (Bi, Pb, Tl, Hg, U) with minimal amount of major elements (Ca, Na, Mg, K ....). The chemical separation is performed following three successive washing steps, i.e. 3M HNO3 (removal of Tl, Pb, Mg, Al, Fe), 0.1M HNO3 (removal of U) and 0.1M HCl (removal of Ca and elution of Am) [4]. In order to limit the 238U peak tailing effect from ultra-traces of uranium on 241Am [5], a second purification step (U/Am) is performed after sample evaporation using the Evapoclean® device (Analab). The feasibility of the chemical approach was first assessed with synthetic solutions prior to the use of real leaching solutions (10 g of sediment per 50 mL of 3M HNO3) with recovery yields of 85.1 ± 4.6% and 56.1 ± 2.0%, respectively. The methodology robustness was validated using certified reference material (AIEA 385). The concentration of americium equals to 4.69 ± 0.17 Bq.kg-1 agrees with the average certified 241Am value (4.31 Bq.kg-1), taking in account the confidence interval (4.26-4.48 Bq.kg-1). As a result, the proposed methodology allows to reduce the analysis time by a factor of about four with respect to that generally necessary for α-spectrometry assuming total analysis time of three weeks in order to achieve 1 mBq.kg-1 detection limit. The methodology was finally applied to assess the environmental level of contamination of 241Am in samples recovered from the Loire River estuary in order to fix a reference concentrations for sediments surrounding nuclear facilities in France. References [1] J. Eikenberg, M. Jaggi, H. Beer, M. Ruthi, I. Zumsteg, Separation techniques for low-level determination of actinides in soil samples, Applied Radiation and Isotopes, 67,(2009), 776-780. [2] J. Letho, X. Hou, Chemeistry and Analysis of Radionuclides, WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim, 2011. [3] M.H. Lee, E.C. Jung, W.H. Kim, K.Y. Jee, Sequential separation of the actinides in environmental and radioactive waste samples, Journal of Alloys and Compounds, 444–445, (2007), 544–549 [4] Z. H. Kazi, J. R. Cornett, X. Zhao, Liam Kieser, Americium and plutonium separation by extraction chromatography for determination by accelerator mass spectrometry, Analytica Chimica Acta, 829, (2014), 75-80. [5] G. Xiao, D. Saunders, R. L. Jones, K. L. Caldwell, Determination of 241Am in urine using sector field inductively coupled plasma mass spectrometry (SF-ICP-MS), J. Radional. Nucl. Chem, 301, (2014), 285-291.

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