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Published in:

Publication date:
2016

Document Version
Publisher's PDF, also known as Version of record

Link back to DTU Orbit

Citation (APA):

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especially for large size environmental samples. This paper gives an overview of analytical protocols developed for plutonium determination in DTU Nutech, and emphasizes challenges and strategies during the methodology development process. Moreover, recent improvement achieved by applying effective sample pre-treatment approaches and sophisticated automation techniques is also presented.

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ENVIRONMENTAL $^{129}$I LEVEL, DISTRIBUTION AND SOURCE IN QINGHAI REGION OF CHINA

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Keywords: long-lived radionuclide, AMS, nuclear environmental safety, soil, iodine

In this work, we collected surface soil samples (0–5 cm) in Qinghai region, and determined $^{127}$I and $^{129}$I in these samples using an effective chemical separation combined with a high sensitivity AMS measurement, in order to investigate $^{129}$I level and distribution in Qinghai region, explore its sources in this region. The data is also useful for establishment $^{129}$I environmental background in Qinghai region, and investigation on the impact of early human nuclear activities on the environment in the region.

The collected soil samples was dried, ground and sieve through a 200 mesh sieve. About 5 g ground soil samples was taken to a quartz boat, 1.0 kBq $^{125}$I tracer was spiked for measurement of chemical yield. The boat with sample as put to a quartz working tube in a tube furnace for separation of iodine using combustion. The temperature of the furnace was gradually increased to 800 °C and kept for 1.5 hours under oxygen gas flow. The off gas from the working tube passed through a bubbler filled with 0.5 M NaOH–0.02 M NaHSO$_3$, liberated iodine from the sample was trapped in the solution in the bubbler. The entire combustion took about 3 hours. 3 ml of trap solution was taken to a plastic tube and measured using a gamma detector for $^{125}$I, which was compared with the $^{125}$I standard (the same amount of $^{125}$I spike solution and diluted to 3 ml using the same trapping solution) for measurement of chemical yield of iodine during combustion. Chemical yield of 97–102% with average of 99% were obtained for soil samples. After measurement of $^{125}$I, the solution is combined to remained trap solution. 1.0 ml trapped solution was taken and diluted 10 times using deionized water for measurement of $^{127}$I using ICP-MS. To the remained solution, NaHSO$_3$ and 0.5 ml of $^{127}$I carrier solution with a concentration of 2.0 mg/ml (prepared from a $^{127}$I free iodine provided by Woodward company, USA, with a measured $^{129}$I/$^{127}$I ratio less than $5\times10^{-14}$ were added, and pH was adjusted to 1–2 using HNO$_3$. After mixed, 1 ml of 1.0 mol/L AgNO$_3$ was added for precipitate iodine as AgI, which was separated by centrifuge. After dried, AgI precipitated was ground and mixed with niobium powder in a mass ratio of 1:5, which was then pressed in copper target holder. $^{129}$I/$^{127}$I atomic ratio was measured using 3 MV accelerator mass spectrometry in Xi’an AMS Center. I$^{5+}$ ion was selected for $^{129}$I measurement. Procedure blanks were prepared using the same procedure as samples, the measured $^{129}$I/$^{127}$I in the blanks are $(1–2)\times10^{-13}$, which is 2–3 orders of magnitude lower than that in samples.

More than 100 surface soil samples was analyzed, the results show that the concentrations of $^{127}$I in Qinghai area are 0.75–16.8 µg/g (dry mass), average 4.72 µg/g, and agree with the literature values (0.5–40 µg/g); $^{129}$I concentration are (0.14–32.4)×10$^7$atmos/g, with an average of 6.61×10$^7$ atmos/g. An abnormally high value of 3.24×10$^8$ atmos/g was observed in the northeast of Qinghai, concentration of this might be associated with early nuclear activities in this regional; $^{129}$I/$^{127}$I atomic ratios in all samples range (0.9–102)×10$^{-10}$, with an average of 3.43×10$^{-9}$, which is higher than pre-nuclear level of 10$^{-12}$ by 1–4 orders of magnitude, indicating that the surface environment in Qinghai region was significantly influenced by human activities.

Acknowledgement

This work was financially supported by China Ministry of Science and Technology through two projects (2012IM030200 and 2015FY110800).