DEVELOPMENT OF A SO-CALLED HOUR METHOD FOR ANALYSIS OF PLUTONIUM IN SOIL SAMPLES

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(Received October 1, 1993)

Following the definition from the International Atomic Energy Agency, a so-called hour method is that which needs six hours time interval from the sample solution preparation to the data evaluation. Two alternative procedures to be applied to soil samples were studied. One based on LaF₃ precipitation and ion-exchange and another on extraction chromatography with TOPO/Silicagel. Both methods gave good results in terms of accuracy, chemical yield and time requirement, but the extraction chromatographic procedure is recommended because it involves fewer steps.

During the first meeting of the Co-ordinated Research Program (CRP) on Rapid Instrumental and Separation Methods for Monitoring Radionuclides in Food and Environmental Samples, which focuses on the development of rapid methods for the determination of radionuclides in the intermediate and late post-accident phase in contaminated food and environmental samples, some needs and requirements related with these so-called rapid methods were identified.¹

In particular, related to the alpha emitters, the need was identified of a so-called "hour" method to be applied to water and soil samples. An hour method was defined as a method with a processing time of 6 hours or less and a desired accuracy between ± 20% to a factor of 2. Supposing 2 hours for counting and evaluation, one has 4 hours to perform the work from sample dissolution to the counting source preparation. Since the counting time of around 100 minutes is not so large, soil sample amounts of at least 10 grams were recommended.¹

As a consequence of this 4-hour period for the whole chemical procedure, it became clear that time-consuming steps such as evaporation should be avoided and, when possible, one step should be used with two simultaneous purposes, e.g. micro-precipitation with LaF₃ for Pu/U separation and source preparation.

We decided to try two different procedures for the separation of plutonium from the matrix and from the natural-occurring alpha emitters. One was based on the co-precipitation with LaF₃ and ion-exchange, and the other one was based on the extraction chromatography with Tri-n-octylphosphine oxide (TOPO). The present work shows the results obtained with both procedures.

Elsevier Sequoia S. A., Lausanne
Akadémiai Kiadó, Budapest
Experimental

Tracers

$^{239}$Pu, $^{239}$Pu, $^{229}$Th, $^{210}$Pb and $^{211}$U solutions were obtained by gravimetric dilution from Amersham standard solutions. $^{239}$Th solution was prepared as described by Abrão.²

TOPO/Silicagel

The impregnation of silanised silicagel 70-230 mesh was performed as described by Lauria and Godoy.³

Columns

Columns used for the ion-exchange separations are shown in Figure 1. Columns used for the chromatographic separations are shown in Figure 2.

![Figure 1. Glass column for the ion-exchange separation.](image1)

![Figure 2. Glass column for the extraction chromatography.](image2)

Alpha spectrometer

The alpha spectrometer was ORTEC 576 surface barrier detectors coupled to one ORTEC ADCAM 918 and a personal computer.