HIGHLY ACCURATE AND PRECISE MULTIELEMENT DETERMINATION OF ENVIRONMENTAL SAMPLES BY MEANS OF PHOTON ACTIVATION USING THE INTERNAL STANDARD METHOD

K. MASUMOTO, M. YAGI

Laboratory of Nuclear Science, Tohoku University, Mikamine, Sendai 982 (Japan)

(Received October 30, 1985)

In order to examine effective applications of the newly developed internal standard method, determinations of Cr, Co, Ni, Zn, As, Rb, Sr, Y, Zr, Nb, Cs, Ce and Pb in three environmental materials, such as pond, lake and estuarine sediments, have been tried by means of photon activation. In these examinations, some major constituent elements in the above sediments were properly used as excellent and effective internal standards. In consequence, it was confirmed that highly accurate and precise determinations of the above 13 elements were achieved easily and favourably.

Introduction

In a previous paper,1 we have proposed a new internal standard method which is applicable to all sorts of activation analysis. When a sample contains W_a g of a trace element A to be determined together with an element B which is usable as an internal standard, and when the comparative standard is prepared by adding an exactly known amount of element A, W_{a*}g to the duplicated sample, W_a can easily be determined by the following equation even if the above sample and comparative standard are irradiated separately by neutrons, photons or charged particles with different fluxes:

\[
W_a = W_{a*} / [ (A_{a*} / A_R) - 1 ]
\]

(1)

where A_R and A_{a*} are count ratios of gamma-rays emitted by two radioactive nuclides produced from element A and B in the sample and comparative standard, respectively. The method can be applied to multielement determination in various materials by providing the comparative standard containing accurately known amounts of each element to be determined.

Elsevier Sequoia S. A., Lausanne
Akadémiai Kiadó, Budapest
On the other hand, in the field of activation analysis, numerous investigators have often applied a multielement technique, allowing determination of a large number of elements per sample. In the cases of environmental pollution and geological studies, it has also been demonstrated that this technique is especially useful because multiple data are required frequently in such cases. If comprehensive and accurate results are to be obtained, the optimum multielement technique which is applicable to all sorts of samples must be determined carefully.

The above new internal standard method can be applied to a wide variety of environmental samples as the most suitable multielement technique. In the present study, therefore, highly accurate and precise determinations of Cr, Co, Ni, Zn, As, Rb, Sr, Y, Zr, Nb, Cs, Ce and Pb in three kinds of environmental standard reference materials, such as pond, lake and estuarine sediments, have been examined by means of photon activation.

**Experimental**

*Materials and chemicals*

The environmental standard reference materials, pond sediment supplied by the National Institute for Environmental Studies of Japan (NIES CRM-No. 2), lake sediment obtained from the International Atomic Energy Agency (IAEA SL–1) and estuarine sediment purchased from the National Bureau of Standards (NBS SRM–1646), were used as unknown samples for the present study. Portions of these samples were dried at 110 °C for four hours and kept in a desiccator with silica gel before weighing. The weight losses of these samples were 10.3% for the pond sediment, 5.2% for the lake sediment and 1.8% for the estuarine sediment. The above main cause was concluded to be due to loss of the hygroscopic moisture.

The stock solutions to prepare the comparative standards containing the elements to be determined were atomic absorption standard dilute solutions (Junsei Chemical Co.) and/or dilute solutions prepared by dissolving exactly known amounts of suitable stable salts of A.R. grade in dilute mineral acid. All the other chemicals used were of guaranteed reagent grade.

*Preparation of the comparative standard*

The comparative standard spiked with the above 13 elements was prepared according to the method reported by MITCHELL et al. After about 1 g of each sample was weighed and transferred into a Teflon crucible, aliquots of each dilute