Photon activation analysis of National Institute of Standards Technology standard reference material using the electron linear accelerator at Pohang Accelerator Laboratory

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Photon activation analysis was performed using a standard sediment SRM 1646a and the Bremsstrahlung from a LINAC at Pohang Accelerator Laboratory (PAL). The selected electron energy was 65 MeV and the mean beam current was 35 mA. A method is described for the simultaneous determination of scandium, manganese and zirconium by photon activation analysis using both elements and sediment SRM 1646a as internal standards. The results agree well with the values given by National Institute of Standards and Technology (NIST). However, a large amount of Zr in SRM 1646a was discovered which was not in the NIST certificate. The concentration of Zr was also determined and compare with the results of RPI. Finally, the PAA sensitivities for Sc, Mn and Zr based on the present experimental conditions were presented. The proposed method can be used for routine analysis of Sc, Mn and Zr in sediment samples.

I. Introduction

The LINAC laboratory of Pohang Accelerator Laboratory (PAL) is equipped with a electron accelerator whose electron energy can reach 100 MeV this accelerator is very suitable to be used as a photon source for Photon Activation Analysis (PAA), also a set of high quality gamma measurement system with a HPGe is available for sample analysis. These facilities make it possible to conduct PAA research at PAL. In this work, the main purpose is to examine the analysis capability of PAA based on our facilities. A standard material SRM 1646a Sediment obtained from National Institute of Standards and Technology (NIST) was analyzed, Internal Standard Method (ISM) was used in our research, because it does not need to maintain the same photon flux for all unknown samples and the reference standard. Using Iron as internal standard, the concentrations of Sc, Mn and Zr were obtained, the results were in good agreement with the data given by NIST. Meanwhile, during the analysis, it was found there existed Zirconium in SRM 1646a, but NIST does not give any information about the concentration of this element, in our work, the content of Zr has also been determined.

II. MATERIALS AND METHODS

Internal Standard Method (ISM), assuming that there is an element whose contents are known in both the sample and the reference, this element can be used as an internal standard. Eq. (1) is the formula which is used in ISM [1,3].

$$\frac{m_S}{m_R} = \frac{I_S}{I_R} \cdot \frac{e^{-\lambda \cdot T_{D,R}}}{e^{-\lambda \cdot T_{D,S}}} \cdot \frac{1-e^{-\lambda \cdot T_{C,R}}}{1-e^{-\lambda \cdot T_{C,S}}} \qquad (1)$$

The NIST 1646a powder was placed in a rectangular plastic tube with dimensions of 1 cm × 1 cm × 5 mm (length, width, and height, respectively), and two samples of the same size were fabricated, stacked, and wrapped with Al foil. These were designated as analytical and reference samples, Samples 1 and 2, and had masses of 0.2499 g and 0.2298 g, respectively.

The experimental arrangement of the bremsstrahlung production and the position of sample irradiation can be seen from in Figure 1. The LINAC machine was used as irradiation source, the irradiation was performed using bremsstrahlung from a 1mm thick Tungsten target. The electron energy was 65 MeV and the beam current was 35 mA, the irradiation time was 1 h.

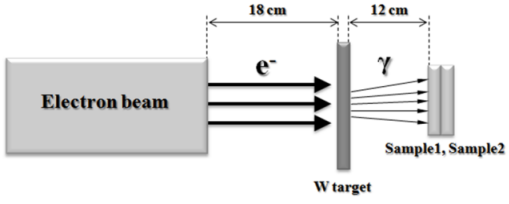

Figure 1. Experimental arrangement for sample irradiation.

After an irradiation and an appropriate waiting time, after which Sample 1 and Sample 2 were transferred into plastic vials, and then the induced gamma activities of the irradiated samples were measured by using γ-spectrometer without any chemical purification. The gamma spectrometer used for the measurements was a p-type coaxial Canberra high-purity germanium (HPGe) detector with an energy resolution of 1.8 keV and relative efficiency of 40 % at the 1.332 keV γ-peak of ^{60}Co.

III. RESULTS AND DISCUSSION

I.A. The nuclear reactions used for PAA

In PAA, the selection of activation products is very important. It is required that the products have suitable gamma energies and the half-lives [1]. The most important is that the selected gamma energies be interference free, which means these gamma energies can not be overlapped by other energies, and the selected product should not be produced by other elements. Table 1 gives the reactions used in this work.

Table 1. Nuclear reactions used in PAA

Element	Reaction	Energy(keV)	Half-life
Sc	$^{nat}Sc(\gamma,n)^{44}Sc$	1157.03	3.93 h
Mn	$^{nat}Mn(\gamma,xn)^{52}Mn$	744.23	5.591 d
Zr	$^{nat}Zr(\gamma,xn)^{89}Zr$	908.91	3.27 d

I.B. The concentrations of Sc, Mn and Zr in SRM 1646a

In the measured spectra, the gamma energies of 908.91, 1157.03 and 744.23 keV were observed, they were originated from $^{nat}Zr(\gamma,xn)^{89}Zr$, which shows that there exists Zr in SRM 1646a, but NIST does not give the concentration of Zr. The concentration of Zr was also determined and compare with the results of RPI [2] . ISM was used to determine the concentrations of Sc, Mn and Zr was used as internal standard element, the results are listed in Table 2 along with the data given by NIST & RPI. From Table 2, it can be seen that the results from Sample 1 and Sample 2 agree with each other and they are all in good agreement with the values given by NIST & RPI, moreover, we obtained the concentration of Zr which was not included with NIST information.

Table 2. The concentrations of Sc, Mn and Zr

Element	Sample 1 (reference standard)	Sample 2 (unknown)
Sc	5 mg/kg (NIST)	6.15 mg/kg
Mn	234.5 mg/kg (NIST)	275.20 mg/kg
Zr	415.4 mg/kg (RPI)	412.60 mg/kg

II. CONCLUSIONS

The results of this study confirm that PAA measurements for the mass spectrometry of trace elements can be performed using the electron LINAC at PAL. A standard material SRM 1646a was analyzed using Internal Standard Method. The obtained concentrations of Sc and Mn agree well with the data given by NIST, and the concentration of Zr was also determined, Zr value does not appear in the NIST 1646a SRM certification, but according to the photon activation analysis conducted by the Rensselaer Polytechnic Institute (RPI). All of the results, further counter correction or uncertainty evaluation is expected to facilitate mass spectrometry allowing for more precision.

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