PROTON ACTIVATION ANALYSIS OF BIOLOGICAL MATERIALS*

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ABSTRACT

Biological materials of garlic, Wulong-tea and human hair were investigated by 14.2 MeV proton activation analysis using internal standard method. Elemental concentrations of Ca, Ti, V, Cr, Fe, Ni, Cu, Zn, Ge, Se, Sr, Cd, La and Pb in biological samples were determined under the 5×10^{-8}—2×10^{-5}g/g of sensitivities

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1 INTRODUCTION

Proton activation analysis of light elements, such as B, C, N and O in Si, SiO_2 or Cu had been reported in our previous papers[1,2]. Isshiki *et al.*[3] reported activation analysis of 18 elements in cobalt using 10.4 MeV proton. Yagi *et al.*[4,5] proposed an internal standard method to determine trace elements in biological materials by means of 12 MeV proton activation.

In this work, 14 elements in biological materials of garlic, Wulong tea and human hair have been accurately determined by 14.2 MeV proton activation analysis using yttrium as an internal standard and

Table 1

(p,n) reactions for 15 elements

Target nuclide	Abundance (%)	Produced nuclide	Half-life	γ-ray energy (keV)
^{44}Ca	2.06	^{44}Sc	2.4d	271
^{48}Ti	73.94	^{48}V	16.1d	984, 1312
^{51}V	99.76	^{51}Cr	27.8d	320
^{52}Cr	83.76	^{52}Mn	5.7d	935, 1434
^{56}Fe	91.16	^{56}Co	77.3d	847, 1238
^{61}Ni	1.19	^{61}Cu	3.3h	660
^{63}Cu	69.09	^{63}Zn	0.63h	669
^{65}Cu	30.91	^{65}Zn	245d	1116
^{67}Zn	4.11	^{67}Ga	78 h	92, 184, 300
^{72}Ge	27.43	^{72}As	26 h	834, 630
^{82}Se	9.19	^{82}Br	35.7 h	550, 780
^{87}Sr	7.02	^{87}Y	80 h	388, 480
^{89}Y	100	^{89}Zr	79 h	909
^{111}Cd	12.75	^{111}In	2.81 d	173, 247
^{139}Ce	99.91	^{139}Ce	140 d	166
^{204}Pb	1.48	^{204}Pb	11.2 h	79

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graphitic powder as the matrix. The nuclear reactions related to this study are listed in Table 1.

The principle of the internal standard method can be described as follows. When the same amount of an elemental standard is added into both the samples to be examined and the standard samples for comparison, elemental concentrations in the sample, W_x, could be determined by Eq (1), even if the samples and comparative standard are bombarded by different fluxes of protons,

$$W_x = W_s/(A_{rs}/A_{rx}) \quad (1)$$

where W_s is the amount of the standard element in comparative standard, A_{rs} and A_{rx} are the count-ratios of the gamma-rays of the nuclides produced from the element under examination and the internal standard in the comparative standard and the sample, respectively.

2 EXPERIMENTAL

a. *Materials and reagents* For preparing the Y internal standard, 63.5 mg Y_2O_3 of AR grade was dissolved in 8 mol/l HNO_3, and diluted to a suitable concentration. Graphitic powder and HNO_3 were of SP and GR grades, respectively.

b. *Treatment of samples* One hundred μg of Y and 2 ml of conc. HNO_3 were added into the biological samples (~ 0.4 g). The solution was added with 1 g of graphitic powder. The mixture was stirred with a glass stick, dried at 150°C and kept at 400 °C in an electric furnace for 1 h.

c. *Preparation of the comparative standard* It was prepared by 1 g of graphitic powder adding with 100 μg each of Y and the elements of interest. The mixture was dried and homogenized by shaking in a plastic vessel for mixing.

d. *Preparation of targets* The powders of the sample and comparative standard prepared as above were pressed into discs of 14 mm in diameter and 2 mm in thickness. Every disc was covered with pure aluminium foil of 54 μm in thickness.

e. *Irradiation of the samples* The experiments were performed by using external proton beam from the cyclotron at Shanghai Institute of Nuclear Research. The discs of the samples and comparative standard were mounted on irradiation containers and transferred into an irradiation chamber by a pneumatic system. Each target was bombarded by 14.2 MeV proton with a current of 2—3 μA for 1 h.

f. *Radioactivity measurement* Each irradiated target was transferred to a plastic vessel one day after the bombardment, and the γ-rays emitted from the produced radioactive nuclides were measured with a Ge(Li) detector.

3 RESULTS AND DISCUSSION

a. *Results of determination* Nine elements in the biological samples of garlic,

Wulong-tea and human hair were detected. Table 2 shows the results. Typical γ-ray spectra are shown in Fig.1 for one of the Wulong-tea sample on the 1st and 8th day after the bombardment.

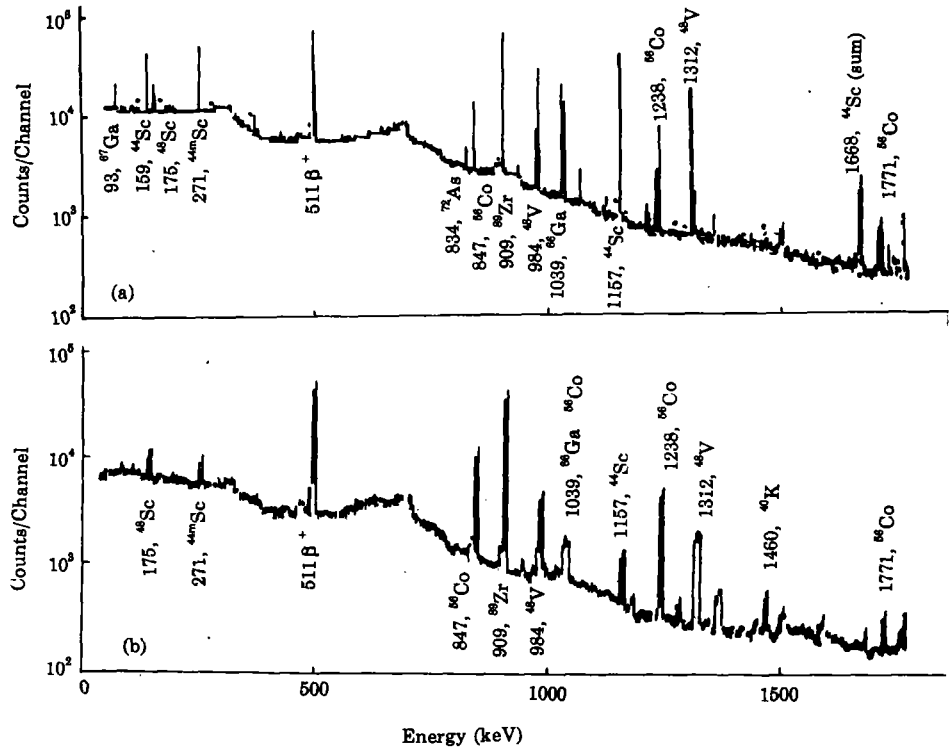

Fig.1 Gamma—ray spectra of Wulong—tea on 1st (a) and 8th (b) day after the bombardment

b. *Blank test* Graphitic powder of SP grade was used as matrix of samples and

Table 2

Concentration of elements in three samples μg/g

	Garlic	Wulong tea	Human hair
Ca	102±8.9	4011±93	1133±79
Ti	8.9±0.9	113.5±3	—
Fe	23.1±1.1	325.7±4	23.3±6.5
Cu	—	—	19.9±2.6
Zn	34.9±2	23.9±2	255.8±33.4
Ge	—	1.3±0.2	—
Sr	—	—	5.5±1.1
Cd	—	—	3.7±1.2

Table 3

Concentration of several elements in the horse kidney (H—8) μg/g

	No.1	No.2	No.3	Average value	Certified value
Ca	943	887	1087	972±103	924±77
Fe	276	254	285	272±13.3	265±15.1
Cu	30	39	36	35±4.6	31±1.7
Zn	204	190	196	201±7	193±6
Cd	170	164	175	170±5.5	189±4.5

comparative standard. The blank values of graphitic powder for 14 elements under investigation were very low, only 0.3 ppm of Zn could be detected in our experiment. Tetraethylsilicate was used as matrix, too, but the blank values were higher than that of graphitic powder. The blank tests suggested the use of graphitic powder as matrix of the samples and comparative standards.

c. *Reliability of method* An IAEA standard material, horse kidney (H-8), was analyzed by the internal standard method. The results and the certified value for 5 elements are listed in Table 3. The relative standard deviations are less than 15%.

d. *Radioactive ratio* One milligram each of an element to be examined and of the yttrium standard were added into 1.0 g of graphitic powder, targeted and irradiated for 10 min. Radioactivity ratios of activated nuclides were given in Table 4. The radioactivities of all the nuclides, except ^{63}Zn and ^{61}Cu, are less than that of ^{89}Zr.

Table 4

Detection limits of 14 elements μg/g

Element	Produced nuclide	γ-ray energy (keV)	Count ratio*	Detection limit
Ca	^{47}Sc	271	0.0044	20
Ti	^{48}V	984	0.1316	0.4
V	^{51}Cr	320	0.0567	0.37
Cr	^{54}Mn	940	0.0821	0.23
Fe	^{56}Co	843	0.0347	0.3
Ni	^{61}Cu	660	1.2485	2.6
Cu	^{65}Zn	669	22.4935	0.13
	^{65}Zn	1116	0.0028	15
Zn	^{67}Ga	92	0.1027	0.4
Ge	^{72}As	834	0.8229	0.05
Se	^{82}Br	554	0.3876	4.1
Sr	^{87}Y	390	0.3958	0.27
Y	^{89}Zr	909	1	—
Cd	^{111}In	247	0.4443	0.27
La	^{139}Ce	166	0.0252	1.5
Pb	^{203}Bi	79	0.0960	2.5

* Radioactivity ratio of nuclides produced by selected and internal standard element

e. *Detection limits* Table 4 shows the detection limits of the 14 elements by 1 h bombardment of 14.2 MeV protons with 2 μA. The detection limits (N) were calculated by $N=3.0\times B^{1/2}$, where B is the number of counts of the background.

It also can be seen from Table 4 that the sensitivities range from 5×10^{-8} to 2×10^{-5} g/g, which are good enough for a multielement analysis of biological samples.

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