Determination of P, Cl, K and Ca in Several Control Serums by Alpha-Particle Activation Analysis Applying the New Internal Standard Method Coupled with the Standard Addition Method

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In charged particle activation analysis, the comparative technique has been used in almost all cases. In this technique, both the sample and comparative standard must usually be bombarded by particles with the same flux in regard to the intensity and energy distribution. In practice, however, it is very difficult to keep the bombarding particles in the same flux at all points of the sample and comparative standard, because the flux is subjected to vary as a function of time. On the other hand, the comparative standard must also be made of a material containing an accurately known amount of interesting element in the same matrix as that of the sample, because the stopping power of the target, dE/dx, is closely related to the chemical compositions of the sample and comparative standard. In fact, however, it is also very hard to obtain such materials for a wide variety of samples.

In order to solve the above problems, in a previous paper[1] we have proposed a new internal standard method coupled with the standard addition method for all sorts of activation analysis. This method has the great advantages that the comparative standard has the same matrix as that of the sample, and that the absolute concentration of the interesting element can be determined easily even when the sample and comparative standard are bombarded separately by particles with different flux. Accordingly, when a suitable standard reference material is absent, it can be used effectively to prepare an optimum working standard in a series of experiments.

In the present experiments, preparation of a working standard for human serums was tried by applying the above new internal standard method, and then simultaneous determination of P, Cl, K and Ca in several control serums were also tried by ordinary alpha-particle activation analysis using the above working standard.

A freeze-dried human serum obtained from the National Institute for Environmental Studies of Japan (NIES Candidate Reference Material No.4) was arranged as an unknown sample, and it was used as a working standard after the concentrations of interesting elements were determined by applying the new internal standard method. As the other analytical samples, several commercially available control serums were used as such.

In order to determine P, Cl, K and Ca in the NIES CRM-No.4, two radionuclides produced from K and Ca, which are usually contained as major constituents in a wide variety of human serums, were used as internal

standards to check the roles with each other. Hence, two kinds of the comparative standards, which were spiked with either P, Cl and K or P, Cl and Ca, were prepared by using the standard addition method. The sample and the above comparative standard were then pressed into smooth-surface pellets, and the pellets were wrapped individually in pure Al foils for bombardment. By using a rotating-target assembly[2] settled on the axis of external alpha beam, 12 pellets were bombarded simultaneously with 1 μA beam of 17 MeV alpha[3] for 30 min. After each wrapping foil was exchanged with a new Al foil, radioactivities produced were measured by the use of a high resolution Ge(Li) detector coupled with a multichannel analyzer at least 20 min after the end of bombardment to allow sufficient decay of the short lived positron emitters, except for the case of ^{38}K measurement.

The gamma-ray spectrum of the radionuclides observed in the NIES CRM-No.4 30 min after the end of bombardment is shown in Fig. 1. As can be seen in the figure, the best choice of the bombarding conditions served to minimize the number of radionuclides produced. The absolute concentration of each interesting element, W_a, was determined on the basis of the following simple equation[1], $W_a = W_a^*/[(A_R^*/A_R) - 1]$, where W_a^* is an exactly known amount of the interesting element added by the standard addition method to the comparative standard, A_R and A_R^* are count ratios of gamma-rays emitted by the radionuclides produced in the sample and comparative standard.

The results obtained in four replicate runs are summarized in Table 1 together with the reference values given by NIES. The values obtained by using two internal standards were proved to be reproducible, and the relative standard deviations were given within ±5%. As a matter of course, each average value was in excellent agreement with the reference value.

Determination of four elements in the commercially available control serums was carried out by ordinary alpha-particle activation analysis using the above NIES CRM-No.4 as a working standard. In these experiments, the sample and comparative standard were bombarded under the same conditions as above. The results obtained in four replicate runs are also shown in Table 2 together with the assigned values given by each maker. Unfortunately, the total concentrations of P in these samples have not yet been determined by the other analytical methods. The values obtained for each element were also found to be reproducible, and the average values except for P were good agreement with each assigned value.

References

1) Yagi M. and Masumoto K., J. Radioanal. Nucl. Chem. 83 (1984) 319.
2) Masumoto K. and Yagi M., J. Radioanal. Chem. 78 (1983) 233.
3) Masumoto K. and Yagi M., Ann. Rept. CYRIC 1982, 69.

Table 1. Determination of P, Cl, K and Ca in the NIES CRM-No.4 human serum.

Internal standard	Concentration of element ($\mu g/cm^3$)			
	P	Cl	K	Ca
K (^{44}Sc)	112.2	3094	·····	81.5
	105.1	3190	·····	76.6
	101.2	3533	·····	82.7
	110.5	3287	·····	80.3
Ca (^{43}Sc)	107.6	3425	173.2	·····
	104.3	3049	165.0	·····
	105.9	3112	163.7	·····
	108.2	3191	174.1	·····
Average value	106.9±3.5	3235±170	169.0±5.4	80.3±2.6
Reference value	107	3200	173	78

Table 2. Determination of P, Cl, K and Ca in commercially available control serums.

Sample	Concentration of element ($\mu g/cm^3$)						
	P	Cl		K		Ca	
	Found	Found	Assigned	Found	Assigned	Found	Assigned
Validate	100.0 92.6 94.0 95.5	3719 3581 3798 3707	3720	222 228 212 221	215	98.1 92.2 96.0 97.1	96
	Av. 95.5±3.2	Av. 3701±90		Av. 221±7		Av. 95.9±2.6	
Validate A	117.6 107.0 121.4 117.0	2968 3541 3343 3282	3260	254 250 260 255	250	123.3 122.7 125.9 123.5	127
	Av. 115.8±6.2	Av. 3284±238		Av. 255±4		Av. 123.9±1.4	
Omega I	125.0 127.4 126.9 128.1	3136 3213 3284 3214	3330	144 142 143 151	137	100.7 98.1 103.4 100.1	100
	Av. 126.9±1.3	Av. 3212±60		Av. 145±4		Av. 100.6±2.2	
Omega II	194.6 194.7 192.9 197.7	3800 3977 3759 3700	3970	253 242 238 241	238	125.5 130.2 126.3 121.0	126
	Av. 195.0±2.0	Av. 3809±119		Av. 244±7		Av. 125.8±3.8	

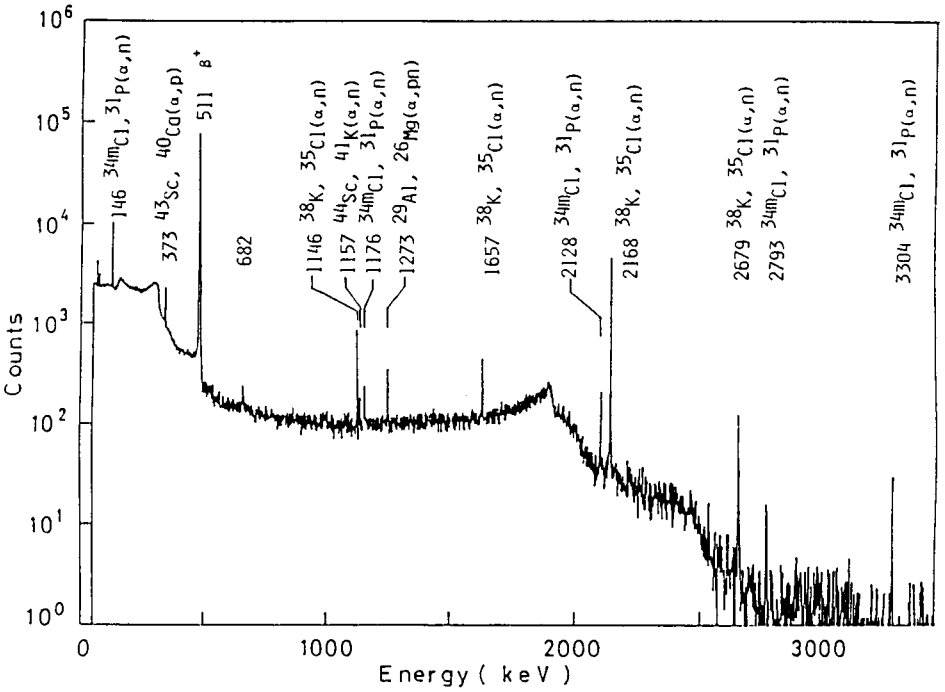

Fig. 1. Gamma-ray spectrum of the NIES CRM-No.4 human serum bombarded with 17 MeV alphas.