Transactions of the Korean Nuclear Society Spring Meeting
Gwangju, Korea, May 30-31, 2013

Optimization and Validation of Instrumental Neutron Activation Analysis for the Determination of Elemental Contents in a Human Serum

J. H. Moon*, G. M. Sun, S. H. Kim, Y. S. Chung
Korea Atomic Energy Research Institute, Daeduk-Daero 989-111, Dukjin-dong, Yuseong-gu, Daejeon, Korea
**Corresponding author: jhmoon1@kaeri.re.kr*

1. Introduction

Human serum has a complicated chemical composition and contains large amounts of proteins and dissolved salts. More specifically, the concentrations of the elements Na, K, Ca, Mg, Cl, S, and P are higher than those of all other elements except for C, H, O, and N. Elemental contents in serum may vary with the diet habit and health status. Many studies have been conducted to identify the relationship between the level of the trace elemental concentration in serum and human diseases [1]. For the determination of the trace elements in serum, various sensitive analytical methods such as an inductively coupled plasma mass spectroscopy, total reflection X-ray fluorescence and inductively coupled plasma atomic emission spectroscopy have been applied [2].

This study was executed to establish an optimum condition of instrumental neutron activation analysis (INAA) for the determination of elemental contents in a human serum sample and to validate analytical results by INAA. For this purpose, INAA for actual human serum and NIST SRM 1598a-Animal Serum was carried out and the analytical data was evaluated in terms of accuracy, precision and detection limits.

2. Experiments

2.1 Sampling and Sample Preparation

For actual serum samples, blood was collected at delivery and centrifuged for 10 minutes to extract serum samples. Serum was stored immediately after collection at -80°C. 1.5 mL of serum samples was taken using an auto-pipette and put into a pre-weighed plastic tube that was cleaned with triple distilled water and 5% HNO_3 [3]. The serum samples in the plastic tubes were freeze-dried at -50 °C for 48 hours, and the weights of the moisture-removed samples were measured and recorded. NIST SRM1598a-Animial Serum sample was prepared for INAA by freeze-drying at -50°C for 24 hours and the moisture contents was 91%.

2.2 Instrumental Neutron Activation Analysis

Approximately 15 mg of serum samples for short-lived nuclides and 100 mg for long-lived nuclides were put into polyethylene vials and irradiated by neutrons for 60 s and 4 hrs, respectively. The irradiation hole used was NAA#1 at the HANARO research reactor. For the measurement of the gamma-rays emitted from irradiated samples, an HPGe detector (EG&G Ortec, 40% relative efficiency, FWHM 1.85 keV at 1332 keV of ^{60}Co) coupled to DSPEC^{PLUS} was used. The elemental contents in the samples was calculated by a software program, so called POWER NAA, which was developed for a fast and comfortable calculation of the INAA.

3. Results and Discussion

3.1 Establishment of Optimum Analytical Condition

From the analytical procedure, it was found that high contents of Na and Cl cause a serious problem for detecting medium and short-lived nuclides. In the condition of short irradiation, Cl-38 nuclide could be detected after 3 hrs decay and K-42 be detected after 8 hrs decay. In the case of long irradiation, Br-82 and Na-24 nuclides were detected after 10days decay and Ca-47(Sc-47) was detected after 12 days decay. After 20 days decay, Cr-51, Co-60, Sb-124, Se-75, Rb-86, Fe-59 and Zn-65 could be detected. The optimum analytical condition is shown in Table1.

Table 1. Optimum condition of INAA for a serum sample

Nuclide by Half-life	Sample Weight (mg)	Irradiation Time	Cooling time	Counting Time	Nuclides detected (Gamma-Ray Energy, keV)
Short (Medium)	10-20	1 min.	2 - 3 hrs.	400 sec.	^{38}Cl(1643)
			5 - 10 hrs.	6000 sec.	^{42}K(1524)
Long	more or less 100	4 hrs.	9 - 11 days	1000 sec.	^{82}Br(554), ^{24}Na(1368)
			12 - 13 days	20000 sec.	^{47}Ca(1297), ^{47}Sc(159) for Ca, ^{75}Se(264)
			longer than 20 days	80000 sec.	^{47}Ca(1297), ^{51}Cr(320), ^{134}Cs(796), ^{124}Sb(1690), ^{75}Se(264), ^{60}Co(1173, 1332), ^{86}Rb(1076), ^{59}Fe(1099, 1291), ^{65}Zn(1115)

3.2 Validation of INAA Method

12 elements from 13 nuclides can be determined by INAA method. Analytical results of 10 elemets determeined from NIST SRM 1598a-Animal Serum were compared with certified or reference values. The result is shown in Fig. 1. The relative error of 7 elements except for Ca, Cs and K are within 20%. Spectral interference of 1291 keV of Fe-59 cause an inaccurate result of Ca determination and Cs in SRM 1598a has a reference value. In case of K, big relative error comes from poor gamma-ray counting statistics.

The uncertainty of gamma-ray measurement for 12 elements in SRM 1598a by INAA is shown in Fig. 2. K and Sb exceed 20% and Cs is about 8%. The other 9 elements are within 5%.

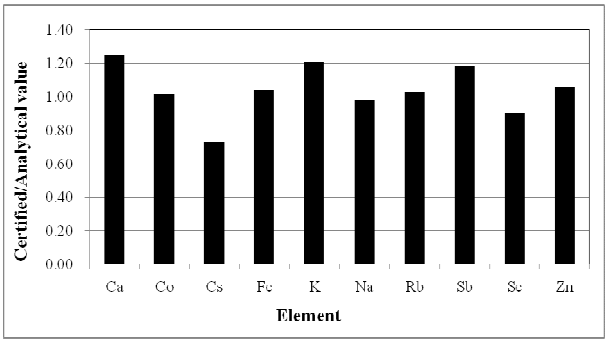


Fig.1. Relative error of 10 elements in NIST SRM 1598a-Animal Serum determined by INAA

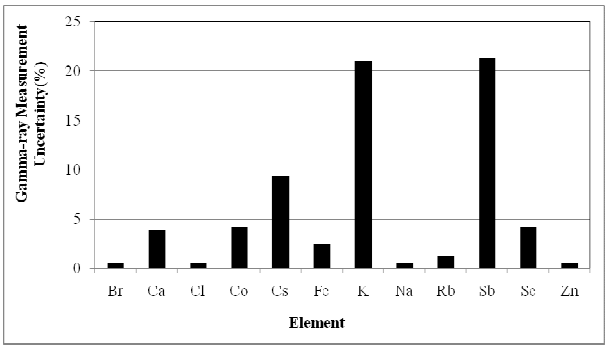
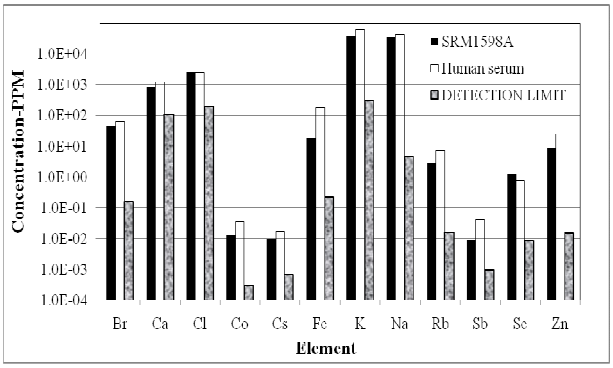


Fig.2. Gamma-ray measurement uncertainty of 12 elements in NIST SRM 1598a-Animal Serum determined by INAA

Elemental contents of Na and K were the highest in the serum samples which are 3% to 6% in dry weight basis. Cl and Ca are around 2000 ppm and 1000 ppm, respectively. Br, Fe and Zn were in the range of 8 ppm to 200 ppm. The other's elemental contents are trace level. The detection limit of 12 elements is lower than the determined value of those.



4. Conclusions

INAA for actual human serum and NIST SRM 1598a-Animal Serum was performed to establish an optimum condition for the determination of elemental contents in a human serum sample and to validate INAA method. 12 elements were determined and the analytical results were evaluated in terms of accuracy, gamma-ray measurement uncertainty and detection limits. In this study, it is shown that 7 elements such as Co, Fe, Na, Rb, Sb, Se and Zn in a serum sample can be determined reliably by INAA.

REFERENCES

[1] World Health Organization, Trace elements in human nutrition and health. Geneva, WHO, Belgium, 2006.

[2] Shengqing Li, Bin Hu, Zucheng Jiang, Rui Chen, "Direct determination of trace refractory elements in human serum by ETV–ICP–MS with in-situ matrix removal" Anal Bioanal Chem, Vol 379, p. 1076–1082, 2004.

[3] Jihye Kim, Young Ju Kim, Rena Lee, JongHwa Moon, Inho Jo," Serum levels of zinc, calcium, and iron are associated with the risk of preeclampsia in pregnant women" Nutrition Research, Vol. 32, p. 764-769, 2012.