A Comparison of Various Procedures in Photon Activation Analysis with the Same Irradiation Setup

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[Abstract] A sample of known elemental concentrations was activated in the bremsstrahlung photon beam which was created by a pulsed electron LINAC. Several procedures of photon activation analysis, including those applied with/without reference material and with/without photon flux monitor, were conducted to make a comparison of their precision and accuracy in practice. Experimental results have indicated that: (1) relative procedures usually produce better outcome despite that the absolute measurement is straightforward and eliminate the assistance of reference materials; (2) among relative procedures, the method with internal flux monitor yields higher quality of the analytical results. In the article, the pros and cons of each procedure are discussed as well.

[Keywords]: Photon Activation Analysis; Quasi-absolute Method; Monte Carlo Simulation; LINAC.

1. Introduction

Photon activation analysis (PAA) has been a versatile nuclear technique in elemental analysis for more than half a century. The first published article of PAA dated back to 1951 when Gaudin and Pannell of MIT tried to determine the amount of beryllium in low grade beryl ores by photodisintegration of Beryllium [1]. They claimed the advantage of this method is “rapid, simple, and nondestructive”. In 1954, Basile suggested to analyze some of the light elements using photonuclear reaction induced by bremsstrahlung radiation from a betatron [2]. His work led to the wide use of the bremsstrahlung radiation as the high energy photon source of PAA. In 2008, Segebade gave a brief but complete overview of PAA research in a chapter of Encyclopedia of Analytical Chemistry [3]. In the recent few years, PAA has expanded its applications to radiotherapy, meteorology, geochemistry, archeology, industrial material, and environmental studies, etc. [4-10]

Traditionally, PAA has been conducted with the relative procedures which need the assistance of reference material. In irradiation, the sample and the reference are activated with the same high energy photon beam. By comparison of the decay spectra created by the same isotope in sample and in reference, scientists can deduce/calculate the unknown amount of the isotope in the sample according to the known concentration of the isotope in the reference. Usually, the photon fluxes in both the sample and the reference are unknown. To track the discrepancy/ratio of photon fluxes, researchers apply flux monitors (e.g. Ni foils) in activation and the relative procedures are divided into three categories accordingly: mass-based (analysis without monitor), external monitor method, and internal monitor method. Relative procedures, especially the internal monitor methods, are also well developed and widely used in neutron activation analysis (NAA) [11-14]. Recently, the developments of computer technology allowed scientists to make Monte Carlo Simulation of photon flux in the sample based on experimental setup, which raised a new absolute method in PAA — quasi-absolute method: with simulated photon flux and database of experimental cross sections, it is possible to calculate the concentration of target isotopes in the sample directly without any reference material [15, 18]. How is the outcome of this absolute method compared with traditional relative procedures? What is the accuracy difference among the different flux monitoring methods in relative procedures? Although there is scattered study on flux monitoring in PAA [16], no one has compared the absolute measurement with relative ones in details before. A study of comparison of the results of various procedures in PAA, whether it is relative or absolute, within the same sample of the same irradiation setup, will be the key to answer those questions.

2. Calculations in PAA

2.1. Quasi-absolute Method

According to Ref. [15, 17-19], the net peak area (or net counts) P of the characteristic gamma line of a particular isotope in the spectrum is

$$P = \eta \theta \zeta \int_{t_c} A(t) dt = \frac{\eta \theta \zeta m c_m h L}{A_r \lambda} (1 - e^{-\lambda t_i}) (1 - e^{-\lambda t_c}) e^{-\lambda t_d} \int_{E_{thres}}^{E_{max}} \varphi(E) \sigma(E) dE \quad (1)$$

where η is the spectrometer efficiency, θ is the branching ratio of the reaction channel, ζ is the absolute intensity of the corresponding gamma line, A is the activity of radioactive nuclide, m is the mass of the whole sample, c_m is the

concentration of the isotope of interest, h is the natural abundance of the target isotope, L is the Avogadro number, λ is the decay constant of product radioactive isotope, A_r is the atomic mass of the target isotope, $\varphi(E)$ is the energy differential photon flux in the sample, $\sigma(E)$ is the cross section of corresponding photonuclear reaction, t_i is the irradiation time, t_c is the counting time, and t_d is the decay time from the end of irradiation to the start of spectrum measurement.

Rearranged equation (1), one obtains

$$c_m = \frac{PA_r\lambda}{\eta\theta\zeta m h L (1 - e^{-\lambda t_i})(1 - e^{-\lambda t_c}) e^{-\lambda t_d} \int_{E_{thres}}^{E_{max}} \varphi(E) \sigma(E) dE} \quad (2)$$

Normally, energy differential photon flux $\varphi(E)$ cannot be measured directly. However, assisted by Monte Carlo simulations from computational nuclear physics, one can derive photon flux by simulated photon yield $Y(E)$, average beam current I_{beam} of the LINAC, and single electron charge q_e as below:

$$\varphi(E) = Y(E) I_{beam} / q_e \quad (3)$$

With $\varphi(E)$ and cross section data $\sigma(E)$, one can compute the integral in the denominator of equation (2). Therefore, it is possible to calculate the concentration of target isotopes without any reference material based on equation (2). This procedure is regarded as quasi-absolute method (QAM) because photon flux is not assessed from any direct experimental measurement, but from Monte Carlo simulation.

2.2. Relative Methods

Both the sample and the reference are irradiated with the same photon beam in the relative methods. Their decay spectra are measured with the same gamma ray detector. According to equation (2), for the same characteristic energy line of the same isotope in the sample and in the reference, one has

$$c_s = \frac{P_s A_r \lambda}{\eta \theta \zeta m_s h L \varphi_s \sigma (1 - e^{-\lambda t_i}) (1 - e^{-\lambda t_{cs}}) e^{-\lambda t_{ds}}} \quad (4)$$

$$c_r = \frac{P_r A_r \lambda}{\eta \theta \zeta m_r h L \varphi_r \sigma (1 - e^{-\lambda t_i}) (1 - e^{-\lambda t_{cr}}) e^{-\lambda t_{dr}}} \quad (5)$$

Where the subscript S means the parameters are of the sample, R means the parameters are of the reference. In addition, the integral of energy differential photon flux $\varphi(E)$ and cross section $\sigma(E)$ is replaced by the product of integral photon flux φ and effective cross section σ as

$$\int_{E_{thres}}^{E_{max}} \varphi(E) \sigma(E) dE = \varphi \sigma \quad (6)$$

Let us divide equation (4) with equation (5) to obtain the general equation of the relative method:

$$c_S = c_R \cdot \frac{P_S}{P_R} \cdot \frac{m_R}{m_S} \cdot \frac{\varphi_R}{\varphi_S} \cdot \frac{(1 - e^{-\lambda_i t_{cR}})}{(1 - e^{-\lambda_i t_{cS}})} \cdot \frac{e^{-\lambda_i t_{dR}}}{e^{-\lambda_i t_{dS}}} \quad (7)$$

On the right side of equation (6), the only unknown parameter is the photon flux ratio φ_R / φ_S . As mentioned, relative methods are fallen into three categories according to the way of getting the flux ratio with different positioning of the monitors: mass-based (MB), external monitor method (EM) and internal monitor method (IM). Here we discuss the internal monitor method in details; calculations of other procedures can be found in Ref [20].

The internal monitor (e.g. Sc solution) is added and mixes evenly in the sample and reference in order to represent photon fluxes in the sample and the reference. From equation (7), for internal monitor i , one has

$$\frac{c_{iS}}{c_{iR}} = \frac{P_{iS}}{P_{iR}} \cdot \frac{m_R}{m_S} \cdot \frac{\varphi_R}{\varphi_S} \cdot \frac{(1 - e^{-\lambda_i t_{cR}})}{(1 - e^{-\lambda_i t_{cS}})} \cdot \frac{e^{-\lambda_i t_{dR}}}{e^{-\lambda_i t_{dS}}} \quad (8)$$

Rearrange equation (8), we can get the photon flux ratio f as

$$f = \frac{\varphi_R}{\varphi_S} = \frac{c_{iS}}{c_{iR}} \cdot \frac{P_{iR}}{P_{iS}} \cdot \frac{m_S}{m_R} \cdot \frac{(1 - e^{-\lambda_i t_{cS}})}{(1 - e^{-\lambda_i t_{cR}})} \cdot \frac{e^{-\lambda_i t_{dS}}}{e^{-\lambda_i t_{dR}}} \quad (9)$$

By plugging the flux ratio of equation (9) back into equation (7), one can reach the final formula for internal monitor method:

$$c_S = c_R \cdot \frac{c_{iS}}{c_{iR}} \cdot \frac{P_S}{P_R} \cdot \frac{P_{iR}}{P_{iS}} \cdot \frac{e^{-\lambda_i t_{dR}}}{e^{-\lambda_i t_{dS}}} \cdot \frac{e^{-\lambda_i t_{dS}}}{e^{-\lambda_i t_{dR}}} \cdot \frac{(1 - e^{-\lambda_i t_{cR}})}{(1 - e^{-\lambda_i t_{cS}})} \cdot \frac{(1 - e^{-\lambda_i t_{cS}})}{(1 - e^{-\lambda_i t_{cR}})} \quad (10)$$

One remarkable thing in equation (10) is: all the mass data are eliminated. Therefore, people do not need to measure the mass of the sample and reference in the experiments. This is an inherent advantage of the internal monitor method.

3. Experiments and Simulations

3.1. Sample, Reference and Monitors

The irradiation target included the sample, the reference, and the photon flux monitors. The sample was the Standard reference material 1648a (urban particulate matter) from NIST [21]. The reference material was the fly ash (certification campaign BCR “City Waste Incineration Ash”) from the Federal Institute for Materials Research and Testing in Germany (BAM) [22]. Both of them have well-known certified elements and are widely used in instrumental analytical research. High purity nickel foils used as external photon flux monitor were bought from Alfa Aesar Company. 1% scandium solution ($\text{Sc}(\text{NO}_3)_3$) was added to both the sample and the reference as the internal photon flux monitor. Initially, the sample and the reference were in powder form. Then we mixed them with cellulose evenly and employed a mechanical compressor to press them into two dime-shaped pellets. Each pellet is in 1 cm diameter and 1mm thickness. Pellets and Ni foils were wrapped together by Al foils to serve as the irradiation target.

3.2. Experimental Setup for Irradiation

Figure 1 shows the experimental setup for photon activation. The electrons are initially produced by the hot cathode and then accelerated by a series of alternating RF electric fields in the LINAC. After focused by magnetic fields, they created an electron beam about 3mm radius at the water-cooling beam window. After the window, electrons hit a custom designed electron-photon converter (or radiator). The converter was made by 3mm thickness of tungsten which induced the bremsstrahlung radiation of incoming electrons and generated a cone of high energy X-rays after the converter. A 3-inch-thick aluminum absorber (or hardener) is positioned directly after the converter to absorb the residual electrons and filter the low energy photons. Most of residual high energy photons hit directly with the target behind the hardener and cause photonuclear reactions inside the sample, the reference, and the monitors.

Fig. 1. Experimental setup for irradiation of the sample and the reference

3.3. Data Acquisition and Data Processing

After the irradiation, the radioactive target was “cooled” down (allowed to decay short-lived radioactivity) over night before we assay the sample, the reference, and the monitors into a high purity germanium detector (HpGe). The schematic setup of electronics in the measurement is shown in Figure 2.

Fig 2. Data acquisition system for gamma spectra measurement.

Three point sources (Co-60, Ba-133 and Cs-137) were stacked together to calibrate the detector since the characteristic energy lines of those sources span a very large energy region. The software for the spectra collection is MPANT from Fast ComTech Company [23]. MPANT automatically saved the spectrum into mp3 format. After the data acquisition, we saved the spectra as mp file for peak analysis with Gamma-W [24]. Gamma-W can quickly read the peaks and subtract the background accordingly. The output file of Gamma-W included all the peak information, such as energy (keV), deviation of the energy (keV), peak area (counts), and deviation of peak area (counts). One also had the information about the mass of samples, irradiation time, decay time and counting time from

measurements. All these data were transferred to a web-based spectra analysis program for relative methods — Photon Activation Analysis System (PAAS) [25]. PAAS read the energy lines and automatically assigned a certain unstable nuclide to each energy line. Besides that, the concentration values of the isotopes of interests were given with standard deviations according to the equations of relative methods in Section 2.

3.4. Cross Section and Simulations

For quasi-absolute method (QAM) in PAA, one needs to know the energy-differential cross section of the isotope of interest and the photon flux in the object. The photon flux was simulated by Geant4 version 4.10 patch 02 [26] exactly based on the geometry and material of the experimental setup in Figure 1. The class library of the high energy physics (CLHEP) used in this version is 2.1.4.1. [27]. The Geant4 toolkit is installed on a computer server (<http://photon.cmt.anl.gov>) running 64 bits GNU/Linux. The physics list of simulations includes all the electromagnetic processes, such as ionization, multiple scattering, positron annihilation, photoelectric effects, Compton scattering, pair production, and bremsstrahlung radiation. A large number of electrons (10 million) were applied in the simulations in order to reduce the statistical uncertainty significantly. The left side of Figure 3 shows the photon profile after the converter (beam comes from below). The right side of Figure 3 depicts the simulation of electron-photon yield in the sample.

The energy-differential cross sections of the isotopes are retrieved from the Experimental Nuclear Reaction Database [28]. These records are in EXchange FORmat (EXFOR) format (except that cross section of $^{68}\text{Zn}(\gamma, p)^{67}\text{Cu}$ is adapted from KAERI simulation with GNASH code [29]) and have been validated by a consortium of organizations, including the International Atomic Energy Agency (IAEA), the National Nuclear Data Center (NNDC), and the Center for Photonuclear Experiments (CDFE).

Fig. 3. Simulation result on electron-photon yield with Geant4 (log scale).

4. Results and Discussions

Na, Ca, Zn, and Rb were selected to compare the results of different procedures of PAA. They were selected because (1) they have known concentration values both in the sample and the references; (2) they have energy-differential cross section in EXFOR database; (3) their product nuclides in photonuclear reactions have very clear (interference and pile-up can be ignored) characteristic energy lines. In the discussion below, we assume the certified value as the true value. The accuracy is indicated by the ratio of the difference (between measurement value and the true value) to the true value. The indicator of precision is described by the ratio of a value to its uncertainty. Both of them are shown in percentages.

Table 1. Comparison between the certified values and the results from quasi-absolute method

Elements	Reaction	$E(KeV)$	$T_{1/2}$	Net Peak Area P	Certified C ($\mu g/g$)	QAM C_q ($\mu g/g$)	Accuracy%
Na	$^{23}Na(\gamma,n)^{22}Na$	1274.53	950d8h20m	1058 $\pm$ 37	4240 $\pm$ 60(1.42%)	2630 $\pm$ 380(14.45%)	-37.97%
Ca	$^{48}Ca(\gamma,n)^{47}Ca$	1297.09	4d12h51m40s	6026 $\pm$ 79	58400 $\pm$ 1900(3.25%)	42800 $\pm$ 7400(17.29%)	-26.71%
Zn	$^{66}Zn(\gamma,n)^{65}Zn$	1115.55	244d5h6m40s	2648 $\pm$ 61	4800 $\pm$ 270(5.63%)	3470 $\pm$ 410(11.82%)	-27.71%
Zn	$^{68}Zn(\gamma,p)^{67}Cu$	184.577	2d13h50m	8721 $\pm$ 121	4800 $\pm$ 270(5.63%)	3860 $\pm$ 390(10.10%)	-19.58%
Rb	$^{85}Rb(\gamma,n)^{84}Rb$	881.61	32d18h23m20s	1011 $\pm$ 48	51.0 $\pm$ 1.5(2.94%)	30.8 $\pm$ 3.6(11.69%)	-39.61%

Table 1 is the comparison between the certified values and the results from quasi-absolute method (QAM). The accuracy is shown in the last column and the precision values are given in the brackets. One can see that the precision and accuracy of QAM results are not practical from the analytical point of view at present, despite the fact that its calculation is straightforward and it eliminates the assistance of reference materials as well. It should be noted that the two different reaction channels of the same element *Zn* gave very close results in Table 1.

The lack of accuracy and precision of QAM may come from a combination of several factors, such as that cross section data are not accurate enough, simulated photon flux is higher than the real situation in the experiments, and the complexity in calculation equations introduced many uncertainties [15, 19]. However, quasi-absolute method has its merit in the future practice: after systematic adjustment of photon flux and with more accurate measurements on experimental parameters, it should be feasible to apply the quasi-absolute method to actual radioanalytical work for certain elements that we have reliable data on photo-nuclear cross sections. The quasi-absolute method has its value in rapid-result field application where large uncertainty is adequate.

Table 2. Comparison between the certified values and the results from relative methods

Elements	Reaction	Net Peak Area P	Certified $C(\mu\text{g/g})$	Reference $C(\mu\text{g/g})$	Relative Methods $C(\mu\text{g/g})$		
					MB	EM	IM
Na	$^{23}\text{Na}(\gamma, n)^{22}\text{Na}$	1058±37	4240±60(1.42%)	16500±400(2.42%)	4040±230(5.69%)	4110±240(5.84%)	4170±300(7.43%)
Ca	$^{48}\text{Ca}(\gamma, n)^{47}\text{Ca}$	6026±79	58400±1900(3.25%)	101600±1800(1.77%)	56500±1700(3.01%)	57600±1700(3.65%)	58400±3090(5.64%)
Zn	$^{66}\text{Zn}(\gamma, n)^{65}\text{Zn}$	2648±61	4800±270(5.63%)	13000±500(3.85%)	3890±210(5.40%)	3970±220(6.05%)	4020±280(7.20%)
Zn	$^{68}\text{Zn}(\gamma, p)^{67}\text{Cu}$	8721±121	4800±270(5.63%)	13000±500(3.85%)	3780±160(4.23%)	3850±160(5.71%)	3900±240(6.39%)
Rb	$^{85}\text{Rb}(\gamma, n)^{84}\text{Rb}$	1011±48	51.0±1.5(2.94%)	139±3(2.16%)	47.0±4.1(8.72%)	47.9±4.2(8.77%)	48.6±4.1(8.44%)

Table 3. Accuracy and precision comparison among different procedures of PAA

Elements	Reaction	Quasi-absolute Method	Relative Methods		
			MB	EM	IM
Na	$^{23}\text{Na}(\gamma, n)^{22}\text{Na}$	-37.97%±14.45%	-4.72%±5.69%	-3.07%±5.84%	-1.65%±7.43%
Ca	$^{48}\text{Ca}(\gamma, n)^{47}\text{Ca}$	-26.71%±17.29%	-3.25%±3.01%	-1.37%±2.95%	0.00%±5.65%
Zn	$^{66}\text{Zn}(\gamma, n)^{65}\text{Zn}$	-27.71%±11.82%	-18.96%±5.40%	-17.29%±5.54%	-16.25%±7.21%
Zn	$^{68}\text{Zn}(\gamma, p)^{67}\text{Cu}$	-19.58%±10.10%	-21.25%±4.23%	-19.79%±4.16%	-18.75%±6.41%
Rb	$^{85}\text{Rb}(\gamma, n)^{84}\text{Rb}$	-39.61%±11.69%	-7.84%±8.72%	-6.08%±8.77%	-4.71%±8.44%

Table 2 shows the results from relative methods. Table 3 compares all the procedures in accuracy and precision.

One can note that both the accuracy and precision from the relative methods are much better than those of the QAM.

This originated from the fact that relative methods remove the dependency of cross sections by introducing the reference materials. For the same characteristic energy line of the same isotope in the sample/reference, lots of parameters just simply cancel out in calculations. Their precisions are in the same range because the sources of uncertainties are primarily from the uncertainty of concentration of the isotope of interest in the reference and counting statistics of the net peak area P . Among those three relative procedures, the internal monitor method has higher accuracy because the internal monitor evenly distributed in the sample/reference. It presents the photon flux better than other methods since it is “internal” and shares the same photon flux with the target isotopes. In addition, the eliminating of mass in calculation certainly reduces the complexity of experiments and increases the quality and reliability of the procedure. However, for some samples which are not easily processable (e.g. too hard to grind to powders), internal monitor cannot be applied: one has to use external monitors or even conduct analysis without monitor.

5. Conclusions

We made a comparison of the quasi-absolute method and traditional relative methods of PAA within the same irradiation setup. Experimental results indicate that: (1) the relative procedures generated better values despite the fact that the absolute measurement has the advantage in straightforward calculation and eliminating the reference materials. (2) In relative methods, the procedure with internal flux monitor yields higher quality of the analytical results.

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