Survey of Standardization Methods of Instrumental Neutron Activation Analysis: A Review

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ABSTRACT

The quest for a versatile standardization method for Instrumental Neutron Activation Analysis Technique has been the major driving force behind many research works for the past few decades. The key factor is the desire to overcome the limitation of dependence on multi-elemental standards without sacrificing analytical accuracy and experimental simplicity. Appraisal of the different approaches towards standardization of Neutron Activation Analysis has been carried out; re-visiting the strengths and weaknesses of each standardization method. k_0-standardization method is proposed to be the most suitable for its robustness and for providing remedy to the drawbacks in other methods. The basic principle of Neutron Activation Analysis and the superior qualities of k_0-standardization has been reviewed.

Introduction

For over seventy-five years since Hevesy and Levi did the experiments that are marked as the birth of Neutron Activation Analysis (NAA), several attempts have been made at standardizing the measurements even though the principles of the technique are well understood [7]. For quantification of the radioactive products, different methods are used including: the relative, the absolute, the single comparator (k-factors) and the k_0 methods. However, none of the first three approaches towards standardization of NAA satisfied all the desired qualities of experimental simplicity, analytical accuracy and flexibility (with respect to activation and counting conditions). The quest for a more versatile standardization method that meets these three criteria has culminated in the k_0-based NAA methodology which is gaining wide acceptance in Nuclear Analytical laboratories worldwide [8, 11]. The development of the k_0-NAA methodology has gone through various stages including: building of the nuclide data library from measurements carried out in expert laboratories, development of procedures for characterization of irradiation facilities, outlining of spectrometer calibration procedures, computerization and development of dedicated software for spectral analysis [6]. The procedures for the characterization of the irradiation channels, calibration of the spectrometers and development of the experimental protocols have been described elsewhere [3, 5, 21, 23, 26].

Survey of Standardization Methods of NAA

For quantification of the radioactive products using the prevalent relative method, the samples and standard materials containing known amounts of the elements of interest are irradiated simultaneously to eliminate the influence of varying neutron flux. Both are also counted under the same experimental conditions to avoid detector efficiencies calculations. The physical parameters for the element of interest being identical in both the analyte and standard cancel out in the concentration calculations reducing the equation for calculating the elemental concentration to its simplest form.

Where A = activity of sample (sam) and standard (std), C = concentration of the element and W = weight of the sample (sam) and standard (std).

$$C_{sam} = C_{std} \frac{W_{std}}{W_{sam}} \frac{A_{sam}}{A_{std}}$$

<div align="right">1</div>

The measured peak area which is proportional to the induced radioactivity is also linearly proportional to the amount of element present and the neutron dose (fluence rate and irradiation time). The relative method uses the proportionality between the amount of an element present in a sample and the area of a measured photopeak to determine the concentration of the element in the sample. This proportionality constant depends on many experimental and physical parameters but the determination of these parameters and their relations to the constant are avoided by irradiation and measurement of a calibration sample. The ratio of the areas of the photopeak corresponding to the element of interest in the two measured spectra is used to calculate the concentration [5]. The major advantage of this approach is that it eliminates the need for accurate determination of neutron fluxes and detector calibration, then one needs to correct the difference in decay between the two [15].

The major problem with the relative method is its reliance on the use of suitable Standard Reference Materials (SRMs), single or multi-element standards prepared in-house or supplied by agencies like the National Institute of Standards and Technology (NIST), United States or the International Atomic Energy Agency (IAEA), Vienna, Austria, and other bodies distributing Certified Reference Materials (CRMs). However, re-investigation of these materials have revealed departures from the certified values due to several possible reasons among which are batch inhomogeneity, contamination, matrix related problems, and degradation with time. Secondly, quantitative analysis is impossible for unexpected elements because no standards are provided. The lack of standards for such unexpected element also makes the calculation of detection limits (maximum possible concentrations) impossible. More important is the ever-increasing demand for these limited reference materials due to wider applications of NAA in research and industry [2, 13, 33]. The departures from certified values of Certified Reference Materials (CRMs) and its attendant consequences on the accuracy of elemental concentrations determined by routine analysis using relative method necessitated a new method that does not rely on the use of reference materials.

The fact that NAA is entirely based on well-understood physical principles and can be completely mathematically described led to a procedure for 'absolute' standardization. This alternative procedure was developed in the late 1950s from mathematical modeling approach for absolute standardization of the quantification of elemental concentration. For nuclide irradiated in a neutron flux where both thermal and epithermal neutron fluxes are present and describing the (n,γ) reaction rate in the Høgdahl convention in which the reaction cross-section σ is inversely proportional to the neutron velocity v $[\sigma(v)\sim 1/v]$ in the thermal neutron energy region. Also for an ideal case in which the epithermal neutron flux per unit of energy is inversely proportional to the neutron energy $[\Phi epi\sim 1/E]$, the reaction rate will be given by

$$R = (\sigma_{th}\Phi_{th} + I_0\Phi_{epi})$$ [18]

Then the mass w of the element to be determined in the irradiated sample can be well approximated by the equation:

$$w = \frac{N_p}{t_m}.\frac{M}{N_A.\theta.(\sigma_{th}\Phi_{th} + I_0\Phi_{epi}).\gamma.\varepsilon_p.S.D.C}$$ 2

Ignoring few correction factors such as the sample's thermal and epithermal neutron self-shielding and gamma ray self-absorption, which are negligible for small sample size as well as the deviation of the epithermal neutron flux distribution from the ideal 1/E law approximated by $1/E^{1+\alpha}$ shape (α).

The list of the symbols used is as follows:

N_p = net peak area of the measured gamma-line (corrected for pulse losses)

t_m = measurement time

N_A = Avogadro's number

M, θ, σ_{th}, γ are the atomic mass, the relative isotopic abundance of the target isotope, the 2200 ms^{-1} (n,γ) thermal neutron cross-section, and the absolute gamma-ray intensity (emission probability) for the analytical gamma-line.

Φ_{th} = thermal neutron flux

Φ_{epi} = epithermal neutron flux

I_0 = effective resonance integral

ε_γ = full-energy peak detection efficiency for measuring E_γ

S = 1 – exp(–λt_{irr}), is the saturation factor with t_{irr} = irradiation time

D = exp(–λt_d), is the decay factor with t_d = decay time

C = [1 – exp(–λt_m)]/λt_m, is the correction factor for the nuclide decay during counting with t_m = counting time

$$\lambda = \frac{\ln 2}{T_{\frac{1}{2}}}$$

λ = decay constant given by with $T_{\frac{1}{2}}$ = half-life of the nuclide.

This equation 2 allows the determination of the neutron flux parameters from the irradiation of a suitable set of flux monitors with and without cadmium. Hence the equation can be solved by an absolute measurement of the induced activity if the disintegration scheme of the isotope of interest is known and the full-energy peak detection efficiency of the detector is also well known.

However, this method cannot be considered as practical as too many uncertainties are introduced, since it was based on nuclear data obtained from the literature which are prone to systematic errors due to the uncertainties associated with these constants. So this "absolute" (parametric) method was not very useful because of its inherent limitations, which led to lower accuracy. Even after appreciable refinement of these data, the resulting accuracy of "absolute" NAA is still lower than the one obtained from relative standard NAA [18, 24, 31].

Further attempts at standardization of NAA led to the introduction of single-comparator method by Girardi and other co-workers in 1965. "It is almost similar to the parametric method but replaces the nuclear constants for each analyte with experimentally determined constants, termed the k-factors" [13]. Given that

$$Detected\ count\ rate = \frac{Net\ peak\ \text{count}}{Live\ time} = \frac{N_p}{t_m}$$ 3

$$Full\ \text{energy peak efficiency of detector}\ \varepsilon_\gamma = \frac{Detected\ \text{count rate}}{Source\ \text{emission rate}}$$ 4

Theoretically, the induced activity A on a sample of mass m irradiated in a reactor under thermal and epithermal neutron flux is given [13] as

$$A = (\phi_{th}\sigma_{th} + \phi_{epi}I_0).m.\frac{N_A}{M}.\theta.S.D.C \qquad\qquad 5$$

While experimentally, the activity A (in disintegration per second) of an irradiated sample for any nuclide with a gamma emission probability γ when counted for a duration t_m by a detector of efficiency ε_γ is given as

$$A = \frac{N_p}{t_m.\gamma.\varepsilon_\gamma} \qquad\qquad 6$$

Where N_p is the net photo-peak counted at specified energy for the nuclide of interest.
From equations (5) and (6) we have

$$\frac{N_P}{S.D.C.m} = (\phi_{th}\sigma_{th} + \phi_{epi}I_0).\frac{N_A}{M}.\theta.t_m.\gamma.\varepsilon_\gamma \qquad\qquad 7$$

If we designate the specific activity (counts per gram) for the irradiated sample as A_{sp} given as

$$A_{sp} = \frac{N_P / t_m}{S.D.C.m} \text{ and } I = \frac{N_P / t_m}{S.D.C} \qquad\qquad 8$$

Where m = mass of the irradiated sample in gram.
From equations (7) and (8),

$$A_{sp} = (\phi_{th}\sigma_{th} + \phi_{epi}I_0).\frac{N_A}{M}.\theta.\gamma.\varepsilon_\gamma \qquad\qquad 9$$

$$A_{sp} = \phi_{epi}\sigma_{th}(f + Q_0).\frac{N_A}{M}.\theta.\gamma.\varepsilon_\gamma \qquad\qquad 9b$$

By definition of k-factors as introduced by Girardi and co-workers in 1965, the ratio of specific intensities (A_{sp}) for any given nuclide compared with that of gold as the co-irradiated comparator is given as

$$k = \frac{A_{sp}}{A_{sp(Au)}} = \frac{\phi_{epi}.\sigma_{th,a}.(f+Q_0)_a.N_A.M_{Au}.\theta_a.\gamma_a.\varepsilon_{\gamma,a}}{\phi_{epi}.\sigma_{th,Au}.(f+Q_0)_{Au}.N_A.M_a.\theta_{Au}.\gamma_{Au}.\varepsilon_{\gamma,Au}} = \frac{\sigma_{th,a}.(f+Q_0)_a.M_{Au}.\theta_a.\gamma_a.\varepsilon_{\gamma,a}}{\sigma_{th,Au}.(f+Q_0)_{Au}.M_a.\theta_{Au}.\gamma_{Au}.\varepsilon_{\gamma,Au}} \qquad 10$$

$$k = \frac{M_{Au}\theta_a\sigma_{th,a}\gamma_a\varepsilon_{\gamma,a}(f+Q_0)_a}{M_a\theta_{Au}\sigma_{th,Au}\gamma_{Au}\varepsilon_{\gamma,Au}(f+Q_0)_{Au}}$$

leading to   11

which yields the k-factor for a given nuclide of interest for co-irradiation of sample and comparator carried out in a stable neutron source, hence Φ_{epi} cancel out in the ratio [13, 16].
Where f = thermal to epithermal neutron flux ratio, (Φ_{th}/Φ_{epi}) and
Q_0 = the ratio of resonance integral to thermal cross-section, (I_0/σ_{th})

$$m = \frac{I}{A_{sp}}$$

From equation (8)   and $A_{sp} = kA_{sp(Au)}$ from equation (10) hence
once the k-factor for a given nuclide of interest is determined from the intensities as above, the mass (m) of the irradiated sample could be obtained as follows:

$$m = \frac{I}{kA_{sp(Au)}} \qquad\qquad 12$$

This single-comparator method based on the experimental determination of k-factors depending on nuclear constants by irradiating known weight of single element with a neutron flux monitor proved to be more reliable and compared well with the level of accuracy of the relative standardization method. However, there was a shortcoming in its applicability to a strictly defined local irradiation and counting conditions due to the experimental parameters in the last part of the k-factor in equation 11 represented by

$$\left[\frac{\varepsilon_{\gamma,a}(f+Q_0)_a}{\varepsilon_{\gamma,Au}(f+Q_0)_{Au}}\right]$$ [13, 31]. This implies this approach is reactor- and detector-dependent.

In an attempt to overcome this limitation, the single comparator method has evolved over the years leading to the introduction of the universal k_0 factor, the key expression in the k_0-standardization of NAA, an acceptable alternative single comparator method which combine the flexibility of the "absolute" with the accuracy of the relative standardization.

This method termed k_0-standardization has solved the three major problems of NAA standardization: the tedious preparation of synthetic standards from pure substances for the relative method, many inaccuracies introduced by using ambiguous absolute nuclear data in the absolute method and the inflexibility of the earlier single comparator method (which is strictly bound to a given set of local

irradiation and counting conditions). It has also increased applicability to panoramic analyses. This standardization method can also be used to investigate nuclear parameters and nuclear reaction rates [12, 31].

The K$_0$-Method

The k$_0$-standardization method was designed to replace the uncertain nuclear data in the absolute technique by composite nuclear constants – the k$_0$-factors, which are experimentally determined with high accuracy. This determination is done as for k-factors, which are then transformed into k$_0$'s by lifting out the experimental parameters [8].

From equation 11 an expression for k$_0$, which is a composite nuclear constant independent of irradiation and counting conditions could be obtained as

$$k_{0,Au}(a) = \frac{M_{Au}\theta_a\sigma_{0,a}\gamma_a}{M_a\theta_{Au}\sigma_{0,Au}\gamma_{Au}}$$

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k$_0$-factors are thus generally applicable on condition that the activation analyst recombines them with the parameters of the local irradiation and counting conditions, in this way generating 'his' k-factors; from this point of view, the k$_0$-method is a flexible single-comparator technique [8, 13].

So in k$_0$-NAA, the normalization of the analytical result is based on the k$_0$-factors associated with each gamma line in the activation spectrum, and the neutron flux characterization is usually based on the Au-Zr triple bare monitor method ($^{95}Zr-^{97}Zr-^{198}Au$), Zr being an important monitor for epithermal flux [13, 22, 28].

Generally, the adoption of k$_0$ standardization method for routine analysis has several advantages including, elimination of problems of non-availability of suitable standards for certain elements as well as the removal of the inaccuracy caused by poorly prepared standards [20]. Perhaps the greatest advantage of k$_0$ method is the flexibility it allows. The k$_0$ factors are reactor- and detector-independent; they are universal, so the same values are accepted and being used by a growing number of NAA users all over the world. Secondly, the k$_0$ standardization method gives more accurate results than the absolute method since the unnecessary build-up of uncertainties in the underlying physical constants is avoided. It has been shown that the k$_0$-factors are also insensitive to changes in their nuclear data components. k$_0$ method of NAA also saves the analyst time and labour that would have been used in counting and analysing multiple standards [4]. Hence increasing throughput of NAA laboratories. k$_0$-NAA standardization has been found especially useful in studies in which the sample matrix composition varies considerably in an unpredictable way, or where no matrix-matching reference materials are available [19]. The k$_0$ factors associated with each gamma line in the activation spectrum replace a series of nuclear constants such as cross-sections and gamma ray emission probabilities [27]. And they are experimentally determined by co-irradiation of a standard and a single comparator.

Theoretical Framework for the K$_0$-Naa Standardization Method

The standard energy for tabulation of thermal neutron cross-section (σ_0) is that of room temperature (293.6 K or 20.43 °C), corresponding to a neutron energy 0.0253 eV or to a neutron velocity of 2200 m/s. However, in many reactors, the temperature (T) in the irradiation channels is not 20.43 °C, but at higher temperature. Westcott developed a method for converting σ_0 to $\sigma(T)$, which is the effective cross-section at the actual temperature (T) of the irradiation channel and introduced the so called Westcott's factor g(T). This parameter g(T) represents the departure of the cross-section from the 1/v law in the thermal region (g(T) = 1 if nuclide obeys the 1/v law in this energy region) [17].

The k$_0$-method was formulated in the theoretically less rigorous Høgdahl convention whose applicability is restricted to (n,γ) reactions for which Westcott's g-factor is equal to unity, i.e. for which the cross-section varies as $\sigma(v)\sim 1/v$ (v- neutron velocity) in the thermal neutron energy region. This holds for most isotopes and permits the simple Høgdahl convention to be used for reaction rate calculations. But the use of the Høgdahl convention rules out the handling of "non-1/v" reactions (Westcott's g-factor differs from unity). The k$_0$-concept can be adapted for "non-1/v" reactions by turning to the more general but also "more sophisticated" Westcott-formalism at the expense of keeping the basic equations of the k$_0$-method simple. This more general Westcott-formalism demands that the values of Westcott's g-factors are available and that an additional neutron spectrum parameter, the neutron temperature is known [10, 34].

The essence of a comparator method is co-irradiation of the samples to be analysed with a suitable element of known mass (comparator), and combining this with the results of gamma-ray spectrometry. Various precalibrations of the detector and irradiation facility with the use of appropriate nuclear data enable one to compute the concentration of any element [24]. Elements such as Au that have well defined nuclear constants are used as monostandards because the nuclear data for the comparator must be known with high accuracy. Usually gold, zirconium, or other suitable elements can be chosen.

"On irradiation with thermal neutrons, ^{197}Au undergoes an (n,γ) reaction to give ^{198}Au (half-life = 2.7 d) emitting well characterised 411.8 keV γ–rays" [14, 29].

The k$_0$ factor of an analyte isotope/gamma-line relative to the gold comparator is defined (equation 13) as

$$k_{0,Au}(a) = \frac{M_{Au}\theta_a\sigma_{0,a}\gamma_a}{M_a\theta_{Au}\sigma_{0,Au}\gamma_{Au}}$$

where M_x, θ_x, σ_x, γ_x are the atomic mass, the relative isotopic abundance of the target isotope, the 2200 ms^{-1} (n,γ) thermal neutron cross-section, and the absolute gamma-ray intensity (emission probability) for the analytical gamma-line. The k$_0$ factors for the majority of the elements that can be determined via NAA were experimentally measured with high accuracy and are tabulated in the literature [1, 12, 24].

The equation for computation of concentration c$_a$ of analysed element is:

$$C_a (in\ ppm) = \frac{\left(\dfrac{N_P / t_m}{SDCW}\right)_a}{\left(\dfrac{N_P / t_m}{SDCW}\right)_{Au}} \times \frac{1}{k_{0,Au}(a)} \times \frac{\varepsilon_{P,Au}}{\varepsilon_{P,a}} \times \frac{f + Q_{0,Au}(\alpha)}{f + Q_{0,a}(\alpha)} \times 10^6$$

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Ignoring the corrections for the effects of neutron self-shielding and γ-ray self-attenuation for real samples with finite size which are considered to be negligible for small sample size used in most cases. Otherwise the formula for computation of the analyte mass fraction would become

$$C_a = \frac{\left(\dfrac{N_P / t_m}{SDCW}\right)_a}{\left(\dfrac{N_P / t_m}{SDCW}\right)_{Au}} \times \frac{1}{k_{0,Au}(a)} \times \frac{\varepsilon_{P,Au}}{\varepsilon_{P,a}} \times \frac{G_{th,Au} f + G_{epi,Au} Q_{0,Au}(\alpha)}{G_{th,a} f + G_{epi,a} Q_{0,a}(\alpha)}$$

15

Given that $G_{th,Au} = 1$, $G_{epi,Au} = 1$ for gold foil, while $G_{th,a} \approx 1$ and $G_{epi,a} \approx 1$ for small sample size. Where $G_{th,x}$ is the thermal neutron self-shielding correction factor and $G_{epi,x}$ is the epithermal neutron self-shielding correction factor.

The first term of equation 15 describes the results of the gamma-ray spectrometry measurements of the analytical radionuclide and the comparator, the second term is the corresponding k_0 factor, the third term is related to the full energy peak efficiency calibration of the detector, and the fourth term accounts for the contribution of the epithermal activation.

Where $\varepsilon_{p,x}$ = full-energy peak detection efficiency for measuring E_γ

f is the thermal-to-epithermal neutron flux ratio, (Φ_{th}/Φ_{epi}).

$Q_0 = I_0/\sigma_0$ with σ_0 (n, γ) cross-section for thermal neutrons (velocity 2200 m s^{-1}) and

$$\int_{E_{Cd}}^{\infty} \frac{\sigma(E)}{E} dE,$$

I_0 = resonance integral for epithermal neutrons =

$E_{Cd} = 0.55$ eV = Cadmium cut-off energy.

α = measure of the nonideal epithermal neutron flux distribution, approximated by a $1/E^{1+\alpha}$ behavior.

W = irradiated sample/comparator weight in gram.

The subscripts "a" and "Au" refer to the analyte and the co-irradiated gold monitor [^{197}Au(n,γ)^{198}Au, E_γ = 411.8 keV] respectively. All other terms and index-notations have their usual meanings as defined earlier [1, 12, 24].

The rate of production of radioactive atoms from stable atoms of interest is related to the activation rate of gold through the k_0-constant. Generally the relative rate of production depends on the specific irradiation facilities and gamma-ray detectors used. The k_0-constant in the 2nd term of equations 14 & 15 represents the part of that rate that is independent of irradiation facility and detector, and plays an important part in the computation of concentration in k_0-INAA. The other parts of this rate are the irradiation facility and detector dependent components represented by the 4th and the 3rd terms of equation 15 respectively. So knowing the k_0-constants, each sample needs to be co-irradiated with a gold comparator only, rather than with as many comparators (or standards) as there are elements to be determined in the sample. Errors in standard preparation are thus excluded. In exchange, systematic errors in the k_0-constants will affect all results obtained. However, these errors have been shown to be small. The general agreement is that the errors in the constants are smaller than 3.5 %. Sample and gold comparator may have different shapes, neutron and gamma absorption characteristics. Correction procedures for these differences are also considered to be integral parts of the k_0-method. The constituents of the k_0-constant (M, θ, σ and γ, as in equation 13) could be found in literature separately, but in most cases with poor reliability. The k_0-constants were therefore measured directly experimentally with high accuracy for more than 130 isotopes relative to a gold comparator and compiled into an electronic database along with other relevant nuclear data in some specialized institutes at Budapest and Ghent [24, 25]. The acceptability of the k_0-method coupled with the large data set and measurements involved in its implementation necessitated computerization of the procedures, leading to the development of several spectral analysis software.

Conclusion

The flaw of the relative method was its dependence on multi-elemental standards, on the other hand absolute standardization developed in the late 1950s eliminated the need for any standards, thus producing experimental simplicity but no accuracy, while the single comparator method (as introduced by Girardi and co-workers in 1965) produced accuracy but very rigid with respect to the irradiation and counting conditions, hence no flexibility. The launching in 1975 of the concept of the generalized k_0-factors and subsequent implementation of the k_0-standardization in many NAA laboratories provided remedy to all these drawbacks. However, implementation of k_0-standardized NAA comes at a price of accurate characterization of the neutron flux parameters in all the irradiation channels and full calibration of the detectors, but the benefits to be derived make these stringent requirements worthwhile [1, 3, 30]. The samples only needs to be co-irradiated with the comparator and flux monitors, usually ($^{95}Zr-^{97}Zr-^{198}Au$) 'triplet' as flux monitor, Zirconium foils being an important monitor for epithermal flux and Gold foil or few millimeters of wire of other suitable materials as the comparator [13].

Among many others, adoption of k_0-method has the following advantages:

1. It reduces dependence on multi-elemental standards.

2. It removes the restriction of unavailability of suitable reference material for determination of "unexpected" elements.

3. The k_0-based INAA method does not require a prior knowledge of elements present in the sample. Hence its applicability to panoramic analyses.

4. It is cost-effective since no cost will be incurred in purchasing the dedicated k_0-IAEA spectral analysis software (freely distributed to member states by IAEA) and chemical standards.

5. It also saves the analysts time and labour that would have been used in counting and analyzing multiple standards, hence it has the potential of increasing throughput of these laboratories.

6. Less analysis will also result in less exposure of personnel to radiation.

7. Reduction in radioactive wastes generated in NAA laboratories as fewer standards are used.

Perhaps the only downside to the introduction of the k_0-method in NAA laboratory is the initial time-consuming task of calibration of all detectors and characterisation of all irradiation channels to be used. Subsequently, only newly introduced detectors and irradiated channels needs to be calibrated.

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