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DEVELOPMENT OF INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS METHODS FOR ASSESSMENT OF IODINE AND SELECTED ELEMENTS IN GHANAIAN FOODS USING LOW POWER RESEARCH REACTORS

BY

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A Thesis submitted to the Department of Physics, Faculty of Science, University of Cape Coast in partial fulfilment of the requirement for the award of a Doctor of Philosophy Degree

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DECLARATION

Candidate's Declaration

I hereby declare that this thesis is the result of my own original work and that no part of it has been presented for another degree in this university or elsewhere.

Candidate's Signature. ..... Date. 2006-11-25

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Supervisors' Declaration

We hereby declare that the preparation and presentation of the thesis were supervised in accordance with guidelines on supervision of thesis laid down by the University of Cape Coast.

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ABSTRACT

Various types of instrumental neutron activation analysis (INAA) method were developed for the determination of Iodine and 30 major, minor, and trace elements in samples of Ghanaian foods. Most of the elements were analysed by conventional INAA with anticoincidence counting. A number of INAA methods were evaluated for selenium and iodine determinations. The methods included: conventional and Pseudo-Cyclic INAA (PCINAA) with Compton suppression gamma-ray spectrometry for selenium. The INAA methods developed for iodine determinations were Conventional Flux and Epithermal INAA (EINAA) using Compton suppression gamma-ray spectrometry. The relative method of standardization was used for quantification of all the elements. Additionally, quantification of iodine and some selected elements was carried out using k_0-NAA standardization method. The Nisle unified formulation was investigated for its applicability to the k_0-NAA method and compared with the well known Hogdahl convention and Westcott formalism.

Precision and accuracy were evaluated through the analysis of standard reference materials (SRMs). The measured values were found to be in good agreement with the certified values; generally within ±10%. Detection limits were calculated and found to vary from 1.0 $ng.kg^{-1}$ for antimony and gold to 400 $mg.kg^{-1}$ for sulphur. The overall uncertainty associated with the measurement of Iodine using both the relative and the k_0 methods of standardization were evaluated. In general, most of the Ghanaian food items were found to contain the required concentration of iodine and other elements of nutritional importance.

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DEDICATION

IN MEMORY OF MY PARENTS

OPAYIN KWAME NYARKO AND MADAM ABA ADAYIWA

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LIST OF ABREVIATIONS AND SYMBOLS

A	Mass number
A_b	Activity of bare sample
A_{cd}	Activity of cadmium-covered sample
A_{sp}	Specific counts rate
AC	Anticoincidence counting
b	barns ($10^{-24}cm^2$)
C	Measurement factor [$= \frac{1-e^{-\lambda t_c}}{\lambda t_c}$]
Cd	Epi-cadmium (related to irradiation under cd-cover)
COI	Correction factor for true-coincidence effects
CONV	Conventional counting
CS	Compton suppression
CSS	Compton suppression spectrometry
D	Decay factor ($=e^{-\lambda t_d}$)
DDW	De-ionized distilled water
DUSR	Dalhousie University SLOWPOKE-2 facility
E	Neutron energy
E_γ	Gamma ray energy
E_{cd}	Effective Cd cut-off energy (=0.55 eV under standard conditions)
EINAA	Epithermal instrumental neutron activation analysis
E_0	0.0253eV (Maxwellian) neutron energy corresponding to v_0

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E_r	Resonance energy
E_T	kT = Energy corresponding to characteristic temperature of the Maxwellian distribution
F_1	Nisle's factor that characterizes the 1/E component of a flux spectrum
f	Sub-cadmium (thermal)-to-epithermal neutron flux ration $(= \frac{\phi_{th}}{\phi_{epi}})$
F_{att}	Correction factor for gamma-attenuation
F_{cd}	Correction factor for Cd-transmission of epithermal neutrons
FNAA	Fast neutron activation analysis
G_e	Correction factor for epithermal neutron self-shielding
G_{th}	Correction factor for thermal neutron self-shielding
GAEC	Ghana Atomic Energy Commission
GHARR-1	Ghana Research Reactor-1
$g(T_n)$	Westcott-g factor at (Maxwellian) neutron temperature T_n
h	Planck's constant
INAA	Instrumental Neutron Activation Analysis
I_{abs}	Neutron absorption resonance integral of an element
I_0	Resonance integral for a 1/E epithermal spectrum
$I_0(\alpha)$	Resonance integral for a $\frac{1}{E^{1+\alpha}}$ epithermal spectrum
k	Boltzman's constant = 8.6165×10^{-5} eV/K

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M	Molar mass
MSI	(Westcott) Modified Spectra Index ($=r(\alpha)\sqrt{T_n/T_0}$)
MW	Megawatts
N	Number of cycles
N_A	Avogadro's number
NAA	Neutron Activation analysis
N_i	Number of nuclei of the parent material
$N_2(t)$	Number of nuclei of product material at time t
NIST	National Institute of Standards and Technology
NNRI	National Nuclear Research Institute
n	Thermal (subcadmium) neutron density n(v)neutron density per unit of velocity interval at neutron velocity v
P_A	Number of counts in the full-energy peak
$P(F1,T_n)$	Nisle effective cross section factor
PCINAA	Pseudo-cyclic instrumental neutron activation analysis
PCEINAA	Pseudo-cyclic epithermal instrumental neutron activation analysis
Q_0	Resonance integral (1/E) to 2200 ms^{-1} cross section ($=I_0/\sigma_0$)
$Q_0(\alpha)$	Resonance integral ($\frac{1}{E^{1+\alpha}}$) to 2200 ms^{-1} cross section ratio ($=\frac{I_0(\alpha)}{\sigma_0}$)
R	(n,γ) reaction rate per nucleus

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R_{cd}	Cd-ratio $(=\frac{(A_{sp})_{bare}}{(A_{sp})_{Cd}})$
RTC	Radiation Technology Centre
S	Saturation factor $(= 1 - e^{-\lambda t_i})$
SRM	Standard Reference Materials
TNAA	Thermal neutron activation analysis
$s_0(\alpha)$	Modified reduced resonance integral
$t_{1/2}$	Half-life
t_c	Counting time
t_d	Decay time
t_i	Irradiation time
T_n	(Maxwellian) neutron temperature
T_0	(Maxwellian) neutron temperature at 293.59K
U_I	Uncertainty associated with element i
v	Neutron velocity
v_{cd}	Neutron velocity corresponding to E_{cd} energy
v_0	(Maxwellian) neutron velocity responding to 2200 $m.s^{-1}$
W	Mass of sample
w	Mass of element
Z	Atomic mass number
α	Epithermal neutron flux shape factor (parameter describing the $\phi_{epi}(E) \sim 1/E^{1+\alpha}$ neutron flux distribution)

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CHAPTER 1

INTRODUCTION

This work is presented in 6 chapters to deal with specific topics. Chapter 1 deals with introduction, objectives and scope of the work while chapter 2 deals with the literature review. Chapter 3 covers the theory of INAA, standardization methods of INAA, interaction of gamma ray with detector materials and gamma ray spectrometry. Experimental, results and discussion and conclusions and recommendations are covered under chapters 4, 5 and 6 respectively.

The NAA technique was discovered in 1936 when Hevesy and Levi (1936) first recognized that nuclear reactions might be used for quantitative analysis of elements, after they exposed a rare earth element (REE) salt to neutrons emitted from Radium Beryllium (RaBe) source. Since then nuclear analytical techniques have become a versatile analytical tool for multi-element analysis. With the development of high-resolution radiation detectors, automated transfer systems and computerized multi-channel analyzers, activation analysis has become a very sensitive analytical tool especially for simultaneous multielement determination.

Basically, in activation analysis a sample is irradiated in a flux of elementary particles, such as neutrons, protons, *etc.*, or in radiation fields such

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as bremstralung (photons) and the intensity of induced radioactivity is measured with an appropriate detector-analyzer system.

Neutron activation analysis in general

Among the activation analysis techniques, neutron activation analysis (NAA) is the most common type where neutrons are employed as the bombarding particles to induce radioactivity. Target nuclei in the sample interact with the neutrons by capture reactions, most commonly (n,γ) reactions, whereby a radionuclide (*i.e.* radioactive isotope) may be formed. A radionuclide has a characteristic half-life and mode of decay. During the decay process a nuclide may emit positrons, alpha, beta and/or gamma-rays or involved in electron capture or internal conversion. The majority (about 90%) of the nuclides formed by the (n, γ) process undergo beta decay which is most often associated with the emission of one or more gamma rays as the product nuclide de-excites to a more stable state (De Sooete *et al*, 1972; Kruger 1971) as shown in Fig.1.1

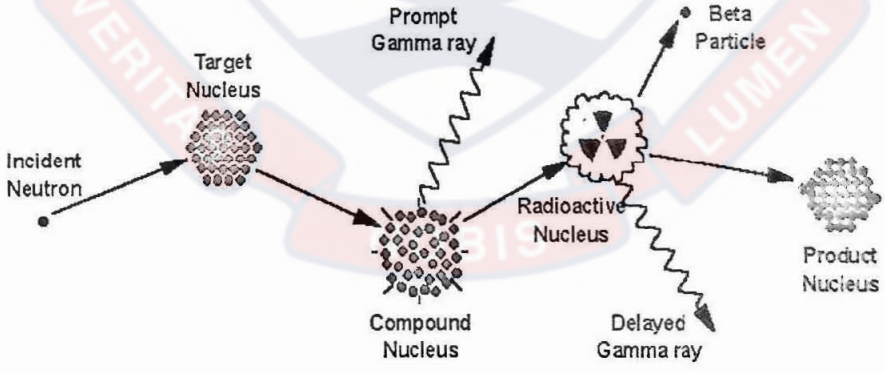

Fig.1.1: A typical Activation process of a nucleus (Glascock, 2003)

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The radioactive isotope formed may disintegrate by emission of gamma-ray within 10^{-10} s. This gamma-ray is referred to as prompt gamma-ray and the activation analysis employing this gamma-ray for analysis is termed prompt gamma activation analysis. The most common gamma-ray energies used in NAA are those called delayed gamma-rays. The emission of these gamma-rays can occur in milliseconds to years after the bombardment of the nuclide has taken place. The technique using these gamma-rays although can be called delayed gamma-ray neutron activation analysis but almost always referred to simply as neutron activation analysis.

In general, the energy of a gamma-ray is characteristic of a nuclide. The gamma-ray energies ranging between 70 and 3100 keV are commonly used for multielement determination by NAA. Neutron activation analysis can be performed in a variety of ways depending on the nature of the background matrix.

In some cases, the element of interest is concentrated from an interfering matrix prior to irradiation with neutrons; this technique is then termed preconcentration neutron activation analysis. On the other hand, if the irradiation is done before separation of the desired elements, the procedure is known as radiochemical neutron activation analysis. The application of these techniques to natural matrices involves a number of steps such as digestion of the sample followed by wet chemical separations. Therefore, both PNAA and RNAA are time-consuming and could be inconvenient for routine analysis. Furthermore, in RNAA special precautions (such as addition of carriers) have to be taken in order to correct for errors due to loses of the elements of interest

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whereas PNAA does not take advantage of the capability of reagent blank-free determinations of NAA. Moreover, RNAA has an added disadvantage of not being able to make use of short-lived nuclides due to relatively long experimental times involved. However, PNAA and RNAA can be very useful for analyzing certain matrices (De Sooete *et al*, 1972; Ehmann and Vance 1991).

Instrumental neutron activation analysis

In contrast to PNAA and RNAA where chemical separations are carried out, INAA, (*i.e.*, no pre- or post-irradiation chemical separation) or non-destructive NAA techniques offers the advantage of multielement analysis without any physical destruction of the sample (Kruger, 1971; Ehmann and Vance, 1991). When high-resolution detectors are used, the specificity of INAA is usually excellent as the purity of the nuclide measured can be checked by its characteristic half-life and the energy of the gamma-ray emitted. Thus the primary advantage of INAA over most of the other analytical techniques are its non-destructive nature, freedom from reagent blanks, excellent selectivity and sensitivity, high accuracy and precision, and capability of simultaneous measurement of multielement concentrations.

One of the problems generally encountered in INAA is high background activity arising from the scattering of photons; this phenomenon is called the Compton Effect. Another problem in INAA is the masking of the element of interest due to high activities of other elements in the sample with high cross sections for thermal neutrons. In the presence of a high background activity, photopeaks with small number of counts diminish in relative size due

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to random fluctuation of the high background counts. In fact, in order to obtain an accurate and precise result, one has to be able to detect the photopeaks of interest at high activities. Therefore INAA methods that enhance the relative activity of nuclides of interest with respect to the background are needed.

In recent years, INAA has been utilized in developing nuclear analytical methods for solving diverse types of analytical problem. Numerous INAA methods have been developed and subsequently applied to study concentrations of trace elements in several matrices (Chattophadhyay, 1974; DeSilva, 1981; Chatt *et al.*, 1981; Landsberger *et al.*, 1990; Rao, 1995; Sullivan, 1998)

Importance of iodine determination

Iodine is an essential micronutrient required mainly for the production of thyroid hormones; it occurs in foods both naturally and artificially as additives (Saxby, 1986). It is an important constituent of the thyroid hormones and is present in most tissues. Iodine deficiency results in enlargement of the thyroid because of hypertrophy and hyperplasia of the thyroid cells. This enlargement, or goiter, is generally considered a classic manifestation of lack of adequate iodine intake (Iodine in Food, 1974). Controlling the amount of iodine present in food is therefore important in order to ensure adequate consumption as well as to prevent excessive consumption. Suitable, sensitive and reliable methods are needed to measure iodine concentrations in foods down to $\mu g.kg^{-1}$ levels.

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In Ghana, goiter is a major problem affecting a cross section of the population especially women and children. In Northern Ghana, people are particularly vulnerable to iodine deficiency disorders (IDD). In recent years, there have been national drives to combat the problem of IDD and all efforts are being made by governmental and non-governmental agencies as well as scientists (Nyarko *et al.*, 2002) to estimate the average daily dietary intake (ADDI) of iodine. All these efforts are geared towards recognition of the population group at risk and to identify various natural sources of iodine.

Objective of thesis

Iodine determination in foods and diets is generally difficult due to the low iodine levels, loss of iodine during sample preparation by most analytical techniques and cumbersome analytical procedures. With INAA, the problem of loss of iodine due to sample preparation is avoided. It is simple, sensitive and offers short-turn around time. The overall objectives of this thesis work were therefore to use nuclear analytical techniques to identify various foods, which are rich in iodine and can therefore be used to help combat the IDD problem in Ghana and to determine essential and toxic elements in some selected Ghanaian food items. Due to the high concentrations of elements like Cl, Mn, Mg, and Na in Ghanaian foods, suitable nuclear methods aimed at reducing interferences from these elements are needed. Nuclear analytical methods developed and used in this work included reactor flux INAA and epithermal INAA in conjunction with conventional and anti-coincidence counting techniques. In order to determine low-levels of iodine in Ghanaian foods, there is a need to develop methods, which will improve the sensitivity,

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precision, accuracy and detection limits of measurements. Pseudo-cyclic INAA and EINAA methods were therefore developed and used in conjunction with anti-coincidence counting for low level iodine and short-lived nuclides determination in food samples. The k_0 standardization and relative methods of INAA were developed at the Dalhousie University SLOWPOKE-2 Reactor facility and the Ghana Research Reactor-1 Center at the National Nuclear Research Institute of the Ghana Atomic Energy Commission for quantification of iodine and other elements on routine basis in various types of sample. Another specific objective of this work was to compare the different INAA methods for the determination of iodine in foods. Different INAA methods were developed for the determination of short, short-to-medium and long-lived nuclides in foods using both conventional and Compton suppression gamma-ray spectrometry.

Summary of Objectives

The objectives of this thesis project are:

1. To develop various INAA methods such as reactor flux INAA in conjunction with conventional as well as anti-coincidence counting techniques for the determination of low levels of iodine and some major, minor and trace elements in Ghanaian food samples;
2. To do a critical evaluation of the k_0 and the relative methods of quantification using low power research reactors for the analysis of iodine and some essential and toxic elements in food samples. The evaluation of the k_0 methods will include (a) the Hogdahl convention (b) Westcott formalism and (c) Nisle unified formalism;

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3. To develop pseudo-cyclic and/or epithermal irradiation in conjunction with conventional and Compton Suppression counting methods, evaluate and use for the analysis of food samples on a routine basis at GHARR-1 Centre and SLOWPOKE-2 facility; and
4. To evaluate epithermal irradiation in conjunction with conventional and Compton Suppression counting and k_0 standardization methods.

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CHAPTER 2

LITERATURE REVIEW

Types of instrumental neutron activation analysis

Although there are several types of neutron sources (reactors, accelerators, and radioisotopic neutron emitters) one can use for NAA, nuclear reactors with their high fluxes of neutrons from uranium fission offer the highest available sensitivities for most elements. Different types of reactors and different positions within a reactor can vary considerably with regard to their neutron energy distributions and fluxes due to the materials used to moderate (or reduce the energies of) the primary fission neutrons. However, as shown in Fig. 2.1, most neutron energy distributions are quite broad and consist of three principal components namely, thermal, epithermal, and fast (Glascock, 2003).

Thermal neutron activation analysis

The thermal neutron component consists of low-energy neutrons (energies below 0.5 eV) in thermal equilibrium with atoms in the reactor's moderator. At room temperature, the energy spectrum of thermal neutrons is best described by a Maxwell-Boltzmann distribution with a mean energy of 0.025 eV and a most probable velocity of 2200 $m.s^{-1}$. In most reactor irradiation positions, 90-95% of the neutrons that bombard a sample are thermal neutrons. In general, a 1 MW reactor has a peak thermal neutron flux of approximately $1x10^{13}$ $cm^{-2}s^{-1}$. Activation analysis employing this type of

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neutrons for sample irradiation is termed thermal neutron activation analysis but generally referred to as NAA

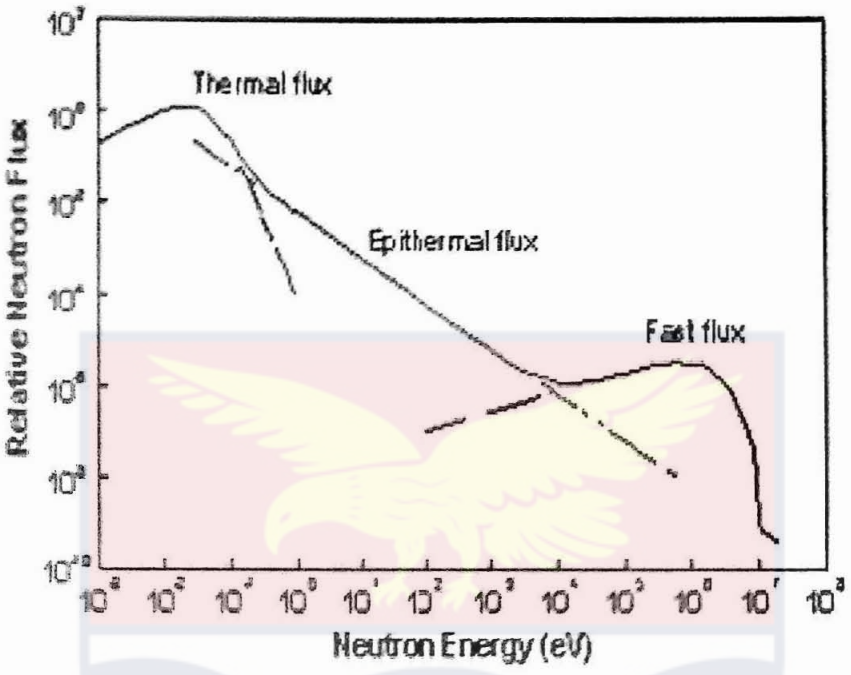

Fig.2.1. A typical reactor neutron energy spectrum showing the various components used to describe the neutron energy regions (Glascock, 2003)

Epithermal neutron activation analysis

The epithermal neutron component consists of neutrons (energies from 0.5 eV to about 0.5 MeV), which have been only partially moderated. A 1-mm thick cadmium foil absorbs all thermal neutrons but allows epithermal and fast neutrons above 0.5 eV in energy to pass through. In a typical unshielded reactor irradiation position, the epithermal neutron flux represents about 2% the total neutron flux. Both thermal and epithermal neutrons induce (n,γ) reactions on target nuclei. An NAA technique that employs only epithermal neutrons to induce (n,γ) reactions by irradiating the samples being analyzed inside either

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cadmium or boron shield is called epithermal neutron activation analysis.

Fast neutron activation analysis

The fast neutron component of the neutron spectrum (energies above 0.5 MeV) consists of the primary fission neutrons, which still have much of their original energy following fission. Fast neutrons contribute very little to the (n, γ) reaction, but instead induce nuclear reactions where the ejection of one or more nuclear particles as well as (n, p), (n, n'), and (n, 2n), are prevalent. In a typical reactor irradiation position, about 5% of the total flux consists of fast neutrons. The NAA technique that employs nuclear reactions induced by fast neutrons is called fast neutron activation analysis (Alfassi, 1990)

The reactions that occur with fast neutrons of energies usually in the MeV range should be considered in two ways; (1) The use of these reactions for the determination of some elements, and (2) the possible interference of these reactions in the determination of some elements by the (n,γ) reaction owing to the formation of the same radionuclide. These can only be solved by the use of double irradiation, *i.e.*, irradiating the sample bare (without Cd cover) and then irradiated inside a Cd or B filter followed by calculation of the contribution from each element. The same treatment is usually adopted when using (n,p) and (n,∝) reactions in the determination of certain elements. (Glascock, 2003)

The main advantage of these reactions is that they produce nuclides that are different from those produced by (n,γ) reactions. Consequently, this

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may lead to faster determinations when producing a short-lived nuclide rather than the long-lived one normally produced by the (n,γ) reaction. In other cases, these reactions may enable the determination of elements that cannot be measured *via* (n,γ) reactions because the produced radionuclide is only a β emitter.

Cyclic and Pseudo cyclic neutron activation analysis

Cyclic Instrumental Neutron Activation Analysis technique is used to enhance the sensitivity of short-lived nuclides by improving counting statistics. This is done by repetitive irradiation-transfer-counting process of a sample for a suitable number of cycles and the gamma-ray spectra accumulated for the analysis. Cyclic method was first introduced by Andrez, 1969 to determine F *via* ^{16}N (half-life = 7.4s) with the reaction ^{19}F $(n,\alpha)^{16}N$ using a pneumatic transfer system.

Later, Spyrou *et al.*, 1974; Grass *et al.*, 1977 and Chatt *et al.*, 1981 applied CINAA techniques for trace element analysis using reactor neutron sources and fast pneumatic transfer systems. Since then, other workers have determined short-lived nuclides in diverse samples using this approach (Chatt *et al.*, 1988; DeSilva and Chatt, 1988; Zhang, 1997).

The cyclic activation techniques often require expensive automated equipment, which is not commonly available in most nuclear analytical laboratories. In addition, high dead-time and pulse pile-up corrections are necessary to account for the high-count rate. Alternatively, Pseudo-cyclic Instrumental Neutron activation analysis method can be used at nuclear facilities where these automated systems are unavailable. The PCINAA is

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based on the principles of CINAA but using the facilities available for conventional INAA. The PCINAA method usually involves manual transfer of samples from receiver to a detector–analyzer system for the determination of up to five elements in several RM and SRMs using short-lived nuclides (Chattopadhyay and DeSilva, 1979; Zhang, 1997; Shi *et al.*, 1999). In this work, three types of PCINAA methods are developed for determination of short-to-medium-lived nuclides.

1. Short irradiation-delay-counting times for the same sample for n number of cycles. With this method, samples become more active after each irradiation and therefore dead-time and pulse pile-up effects increase as the number of cycles increases.
2. Short irradiation-long delay – short counting times; several hours of decay after irradiation makes the irradiated sample virtually inactive and therefore dead-time and pulse pile-up effects are virtually nonexistent. The disadvantage of this method is long throughput time of analysis and if the nuclide of interest has long-lived isotope, then this method becomes undesirable.
3. Short-irradiation-delay-counting times with different capsules of the same sample. Here n number of the same sample is prepared and irradiated one after the other and the spectra accumulated on one another. This method eliminates high dead-time and pulse pile-up effects and reduces sample analysis time. This method is sometimes referred to as commutative INAA. It can also be used to determine the homogeneity of a sample.

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General food analyses

Human existence and its survival predominantly depend on the inhalation of ambient air, intake of clean water, and ingestion of nutritionally adequate as well as contaminant-free food. Nutritional importance of many trace elements is well established (World Health Organization Report of a WHO/FAO/IAEA, WHO, Geneva, 1994). Essential trace elements play a very important role in various physiological and metabolic processes of the body. Appropriate intakes of these elements are required for the above processes, since deprivation can lead to diseases (Oskarsson and Sandstoerm, 1995). On the other hand, excessive intakes of some essential elements may adversely affect the human biomedical functions (Prasad, 1993). There is also an interest in understanding the role of certain elements in flavour and toxicology of foods (Contis, 2001). For these reasons, there is an increasing interest in the determination of mineral content of foods and diets even at very low levels. Recent advances in analytical techniques with improved sensitivity have opened up this new scope to scientists (Gharib *et al.*, 2001).

There are three main reasons for obtaining better information on the trace element levels in foods and diets (Stewart, 1980). The first reason is to measure the concentrations of as many elements as possible with improved sensitivity, accuracy and precision. The second reason arises from the need to trace the flow of elements through food supply. The third reason is the necessity to provide better knowledge-based sources of safe foods (Contis, 2001). Nuclear analytical methods can be conveniently applied to all these areas (Iyengar, 1986; Valcovic, 1975; Buss, 1983; Underwood, 1986; Contis, 1993; Tandon, 1995)

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Elements of health interest have historically been divided into two major groups depending upon their levels, namely the mineral elements (>10 $mg.kg^{-1}$) and trace elements (<10 $\mu g.kg^{-1}$) (Wolf, and Harnly, in: Chralambous, 1984). However, modern analytical techniques have pushed detection limits down from $\mu g.kg^{-1}$ to $ng.kg^{-1}$ range. Many techniques have been used for measuring elemental levels in foods at $\mu g.kg^{-1}$ to percentage levels. These include atomic absorption spectrometry (AAS), inductively coupled plasma (ICP) coupled to atomic emission spectrometry (ICP-AES) and mass spectroscopy (ICP-MS), neutron activation analysis (NAA), X-ray fluorescence and ion selective electrodes. Several excellent examples of general food analysis including trace elements have been published (Saxby, in: King, 1984; Manning, in: King, 1984; King, 1984; Charalambous and Inglett, 1983; Alfassi, in Alfassi, 1991; Chatt, 1988).

Of all the instrumental techniques, ICP, NAA and XRF are most widely used. In general, ICP and AAS methods require sample in a liquid state and a reagent blank correction. On the other hand, various types of NAA can be used for both solid and liquid samples. In addition, NAA offers easy sample preparation, freedom from reagent blanks, high specificity, improved sensitivity, high accuracy, rare interferences, and seldom matrix effects.

The determination of trace elements at low levels in food samples by INAA sometimes suffers from high background activities induced by the activation products of elements such as Na, K, Mn, Br and Cl. This effect is mostly dominant at the lower energy region of the gamma-ray spectrum where the photopeaks exhibit poor signal-to-noise ratio due to Compton scattering. A Compton suppression gamma-ray spectrometry system can be used to lower

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the background for obtaining better counting statistics, higher precision and lower detection limits.

Procedures usually adapted to enhance sensitivity of various NAA methods involve the optimization of irradiation, decay and counting times, the use of cyclic and pseudo-cyclic modes, the employment of loss-free counters, large detectors, low-background shields, and chemical separation either before or after irradiation. While the Compton suppression system is not new, very few groups have judiciously employed this technique for trace element determination in foods by NAA (Suzuki and Harai, 1990; Cumming *et al.*, 1988).

The Compton suppression is unique in that while it reduces the Compton Effect, it is also an excellent shield for external background due to the thick sodium-iodide detector used as an annulus (Landsberger, 1994). Recently, Zhang, 1997 demonstrated the capabilities of the Compton suppression gamma ray spectrometry system by analyzing over 30 elements in several biological samples.

Iodine in foods and dietary intake

Iodine is an essential trace element for animals and humans. It forms an indispensable part of the thyroid hormones, *i.e.*, thyroxine (T_4) and 3,5,3' - triiodothyronine (T_3). In all vertebrates including man, a constant supply of these hormones is necessary for proper development of the brain and for the body growth, and to keep the level of basal metabolism and of functional activity of most organs normal. In the human body, it forms an essential component of thyroxine, the main hormone produced by the thyroid gland.

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The excessive consumption of certain foods like cabbage, cauliflower, cassava and reddish can cause iodine deficiency. These foods contain substances, which react with the iodine present in the food and make it unsuitable for absorption (Assessment of iodine deficiency disorders and monitoring their elimination, WHO, 2001).

Iodine occurs in foods mainly as inorganic iodide, which is readily and completely absorbed from the gastrointestinal tract. Other forms of iodine in foods are reduced to iodide before absorption. Absorbed iodide is distributed throughout the body *via* the circulatory system. A portion (approximately 30%) is removed by the thyroid for hormonal synthesis. Iodine intake in excess of requirement is excreted primarily through the urine (Assessment of iodine deficiency disorders and monitoring their elimination, WHO, 2001).

Dietary iodine is absorbed from gastro-intestinal tract into the blood. The amount of iodine present in the body of an adult is estimated to be about 25 µg. Most of it is concentrated in the thyroid gland, where it is stored in the form of thyroglobulin, a complex of protein and iodine. To ensure an adequate supply of thyroid hormones, thyroid must trap about 60 µg of iodine per day (Underwood, 1977). The recommended dairy dietary intake (DDI) of iodine for man is 50 $\mu g.day^{-1}$ from 0-6 months, 90 $\mu g.day^{-1}$ from 6 months to 6 years, 120 $\mu g.day^{-1}$ from 7 years to 10 years, 150 $\mu g.day^{-1}$ during adolescence and adulthood and 200-300 $\mu g.day^{-1}$ during pregnancy and lactation (Trace Elements in Human Nutrition and Health, 1966). In the presence of goitrogens in the diet, the intake should be increased to 200-330 $\mu g.day^{-1}$ for adults. Goitrogens are found in a number of staple foods in developing countries, including cassava, maize, bamboo shoots, sweet potatoes, lima beans and

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millets. Wolf (1967) suggested that iodine intake by humans of 2000 $\mu g.day^{-1}$ should be regarded as excessive or potentially harmful. When these physiological requirements are not met in a given population, series of functional and developmental abnormalities occur, including thyroid function abnormalities. When iodine deficiency is severe (*i.e.*, iodine intake is <20 $\mu g.day^{-1}$), endemic goiter and cretinism occur together with endemic mental retardation, decreased fertility rate and increased perinatal death and infant mortality. These complications are grouped under the general heading of iodine deficiency disorders (Hetzel *et al.*, 1987)

It is estimated that there are about 1.6 billion people (approximately 30% of the world population) are affected by IDD including 655 million with goiter, 26 million with severe brain damage and 5.6 million with overt endemic cretinism. Data released by WHO-Nutrition Unit, Global Provenance of Iodine Deficiency Disorders, 1993, indicates that approximately 140 million people in Europe are at risk of IDD today and that 97 million have goiter. Consequently, iodine deficiency constitutes a major public health issue. It presents one of the most common preventable causes of mental impairment in the world today. In the developing countries, IDD is more severe because they live in iodine-deficiency environment characterized by soil from which iodine has been leached by glaciations, high rainfall or flood (Trace Element in Human Nutrition, 1996). This means that, all the food grown in such soil is low in iodine so that iodine deficiency will persist unless there is dietary diversification or some form of iodine supplement is given (Trace Element in Human Nutrition, 1996). The major part of essential iodine enters living organisms *via* food chain. However, the accurate data on the iodine

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concentration in foods and diets are rather scarce, the main reason being analytical difficulties associated with the element determination, especially at low levels.

Survey of iodine determination in biological materials

It is generally recognized that the concentration of iodine in most biological tissues is low. Accurate determination of iodine in food requires very sensitive methods of analysis with freedom from reagent contamination. The estimation of iodine in complex foodstuff is difficult because in most analytical techniques the sample must be digested and concentrated. A Survey of iodine determination in different matrices and its associated advantages and disadvantages are presented in Table 2.1

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Table 2.1: A survey of analytical methods used for the determination of iodine in biological matrix

Sample Matrix	Method used	Reference	Advantages/ Disadvantages
Food	Modified Ehnslie-Calwell dry ash procedure (heating samples at 100°C followed by ashing at 500°C. Iodine is determined by titration with sodium thiosulphate in the presence of starch.	AOAC (1970)	Method is good for samples with relatively high Iodine content. Is tedious, lead to partial losses of iodine during ashing
Protein bound iodine in tissue, blood, plasma, milk	The technique involves precipitation, washing, and oxidation of the protein, distillation of the iodine and calorimetric iodine determination	Barker (1948), Sandell and Kolthooff (1937), Binnert (1954)	Good for fluid or liquid samples
Food and Drugs	The method consist of oxidation of micro-quantities of iodide in the to iodine, xylene extraction of the iodine and subsequent spectro-photometric determination	Oslson (1961), Menschen-Freund (1956)	Can determine low amount of iodine (0.01-0.1µg)

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Table 2.1: Continued

Sample Matrix	Method used	Reference	Advantages/Disadvantages
Blood serum	"Wet ashing" the sample with choleric acid	Zak *et al.* (1912)	Destructive and loss of iodine
Air dried plant materials	Cerium-arsenic reaction after oxidation of the dry sample in schöniger combustion flask and subsequent collection of the iodide in 1M NaOH	Cuthbert and Ward (1964)	The speed of sample oxidation and the small sample size are the chief advantages
Urine, stool, tissue, biological materials	Samples homogenized in the wet state and lyophilized to ensure uniform sampling. The lyophilized samples (~30mg) is digested with chloric acid and the sample size adjusted to make the total iodine content between 0.01-0.06 µg per sample. After complete digestion the analysis is continued in a manner described by Zak	Benotti *et al.* (1965)	Time consuming and lost of iodine due to ashing

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Table 2.1: Continued

Sample Matrix	Method Used	Reference	Advantages/Disadvantages
Food, natural products and water	Studies of optimal conditions for the automated determination of low iodine concentrations by the Sandell-Kolthoff reaction. Use of Auto Analyzer System to determine µg/liter amounts of iodine.	Keller *et al.* (1973)	Capable of deterring low levels of iodine but time consuming
Seawater	Platinum electrode techniques of Potter and White (1957)	Berkley and Thompson (1960)	Compares favourable with the catalytic Sandell-Kolthoff reaction method
Animal and plant materials	Ion-selective electrode analysis (used to estimate microgarm quantities of iodine)	Hoover *et al.* (1971)	No ashing, no interference. It is simple and rapid. Compares favourable with AOAC method
Row Milk	Simple electrochemical method. The method uses solid state-ion-selective electrode principle	Curtis (1973)	Requires technical competence. Gives relative values of iodine

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Table 2.1: Continued

Sample Matrix	Method Used	Reference	Advantages/Disadvantages
Several body compartments and hyperthyroid human subjects	NAA techniques. Measurement of specific radioactivity of iodine in plasma, urine, feces and thyroid glands	Riviere *et al.* (1965) Conttino *et al.* (1967)	Higher sensitivity, specificity, precision and accuracy. Initial equipment cost is very expensive
Vegetables, biological fluids	NAA	Ohio (1971). Heurtebise (1971)	Higher sensitivity and specificity, good precision and accuracy. Initial equipment cost is very expensive
Proposed Iodine determination is some matrices	Gas-Chromatography	Hasty (1971)	Used in analysis of food has not been evaluated

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Table 2.1: Continued

Sample Matrix	Method Used	Reference	Advantages/Disadvantages
Foodstuffs	Different NAA methods	Kucera, Randa and Soukal (2001)	Low detection limits, good precision and accuracy
Health food, food and Salts	EINAA	Chen (2003), Serfor-Armah *et al.* (2003), Nyarko *et al.* (2002)	Low detection limits, good precision and accuracy, and non destructive
Cow Milk	NAA, catalytic acceleration and specific electrode measurement	Binnerts (1989)	Found NAA to be more reliable than the rest
Food and Water	Kinetic method based on catalytic reaction of Ceric	Longvah and Deosthale (1998)	Ashing of samples leads to loss of iodine
Urine	Simple Microplate Method	Ohashi *et al.* (2000)	Rapid monitoring of iodine
Foodstuffs	Quartz crystal microbalance method	Yao, Chen and Wei (1999)	Cheap equipment cost but sample should be in liquid form
Foodstuffs	Improve Micro-method	Patnaik, (1934)	Lost of iodine

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Iodine deficiency disorders in Ghana

In Ghana, goiter is a major problem affecting a cross section of the population especially women. Children are also at risk of IDD due to lack of knowledge on the levels of iodine in Ghanaian foods and diets. In the northern Ghana, people are particularly vulnerable to IDD. The most recent IDD survey carried out in Ghana from 1991-1994 among school children of ages between 10-19 years and women of reproductive age 15-45 years from twenty-seven districts showed that the Bongo district in the Northern Region had the highest prevalence rate of 56.5% while Shama Ahanta district in the Western Region, had the least at 7.6%. Http://www.edu/~internut/countries/ghana/ghanaiodine.html. (1995)

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CHAPTER 3

THEORY OF NEUTRON ACTIVATION ANALYSIS, STANDARDIZATION METHODS AND GAMMA-RAY SPECTROMETRY

Neutron activation analysis is extensively used for the determination of major, minor and trace elements in fields like archaeology, biomedicine, cosmology, ecology, forensic science, geochemistry, material science, nuclear technology, zoology, etc. (Morrisson and Porterm, 1972; Davis *et al.*, 1982; Stone *et al.*, 1988; Cheng *et al.*, 1994; Frontasyeva and Steinnes, 1995; Luten *et al.*, 1997). In comparison with other analytical techniques such as AAS, ICPMS/AES, electrovolumetric methods etc., NAA has high sensitivity and selectivity for a large number of elements (Muramatsu *et al.*, 1989; Meon and Dams, 1995). In NAA, the proportionality between the amount of an element present in a sample and the area of a measured photopeak is used to determine the concentration of the element in the sample. The proportionality constants depend on many experimental and physical parameters.

The quantification of an element in a sample can be carried out *via* three main methods of standardization, namely absolute, relative and single comparator (k_0). These k_0 are applied in various facilities for the multi-element analysis of samples. The relative method is prevalently applied in most laboratories.

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Activation Equation and Principles of Standardization

If a stable nuclide is exposed to a thermal neutron flux, it may capture a neutron to produce a radioactive isotope of that element. This can be represented by a simplified equation as:

$$ {}^{A}_{Z}Y + {}^{1}_{0}n = {}^{A+1}_{Z}X + \gamma \qquad [3.1] $$

If n_i is the number of nuclides of a given stable isotope exposed to thermal neutron flux ϕ for a time t_r, σ_i the activation cross section for the (n,γ) reaction and $N_i(t_r)$ is the number of the radionuclide formed, then the rate of reaction is given by:

$$ \frac{dN_i(t_r)}{dt} = \text{Rate of production} - \text{rate of radioactive decay} \qquad [3.2] $$

Production rate $= \phi\sigma_i n_i$

And rate of radioactive decay $= \lambda N_i(t_r)$

Therefore, $\frac{dN_i(t_r)}{dt} = \phi\sigma_i n_i - \lambda N_i(t_r)$ [3.3]

Integrating equation 3.3 yields

$$ N_i(t) = \frac{\phi\sigma_i n_i(1 - e^{-\lambda_i t})}{\lambda_i} \qquad [3.4] $$

$\lambda_i = 0.693/t_{1/2}$

The activity $A_i(t)$ at any time t during the irradiation period according to equation 3.4 is given by

$$ A_i(t) = \lambda_i N_i(t) = \phi\sigma_i n_i(1 - e^{-\lambda_i t}) \qquad [3.5] $$

Where n_i is expressed as:

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$$n_i = \frac{m_i \theta_i N_A}{M_i}$$

At the end of the irradiation period the activity is given by;

$$A_i(t_i) = \frac{\phi \sigma_i \theta_i m_i N_A (1 - e^{-\lambda i t_r})}{M_i} \quad [3.6]$$

If counting is delayed for time t_d after irradiation then the activity at the end of the delay is:

$$A_i(t_d) = A_i(t_i) e^{-\lambda_i t_d} \quad [3.7]$$

If after the delay, the sample is counted for a time t_c, then the number of disintegrations that occurred during the counting period is obtained from equation 3.7 as:

$$N_i = \int_0^{t_c} A_i(t_d) e^{-\lambda_i t_d} dt = \frac{A_i(t_d)(1 - e^{-\lambda_i t_c})}{\lambda_i} \quad [3.8]$$

From equations [3.6], [3.7] and [3.8]

$$N_i = \frac{\phi \sigma_i \theta_i m_i N_A (1 - e^{-\lambda_i t_r})(1 - e^{-\lambda_i t_c}) e^{-\lambda_i t_d}}{\lambda_i M_i} \quad [3.9]$$

Suppose $\varepsilon(E_i)$ is the photopeak detection efficiency for the gamma ray energy E_i and total counts recorded by the detector (the photopeak area) is P_A then N_i can be expressed as;

$$N_i = \frac{P_A}{\varepsilon_i(E) \gamma_i} \quad [3.10]$$

From equation [3.9] and [3.10] we obtain

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$$m_i = \frac{P_A \lambda_i M_i}{\phi \sigma_i \theta_i N_A \varepsilon_i(E) \gamma_i (1 - e^{-\lambda_i t_r})(1 - e^{-\lambda_i t_c}) e^{-\lambda_i t_d}} \quad [3.11]$$

If W is the weight of the sample used, then the concentration or the amount ρ of a nuclide i in the sample is given by:

$$\rho = \frac{m}{W_i} = \frac{P_A \lambda_i M_i}{\phi \sigma_i \theta_i N_A \varepsilon_i(E) \gamma_i G (1 - e^{-\lambda_i t_r})(1 - e^{-\lambda_i t_c}) e^{-\lambda_i t_d} W} \quad [3.12]$$

Equation [3.12] can be written as:

$$\rho = \frac{m_i}{W} = \frac{[P_A / t_c] M_i}{\phi \sigma_i \theta_i \gamma_i \varepsilon(E_i) N_A SCDW} \quad [3.13]$$

Standardization methods

Absolute (parametric) standardization

This method of quantification is based on equation [3.13] above. By measuring P_A for known timing parameters, *viz.* t_i, t_d, and t_c, the amount of the element present, ρ can be calculated. A reliable determination of ρ requires prior knowledge of accurate values of ϕ, σ, θ, ε and λ. Since these parameters are not usually known with a high degree of accuracy, the absolute measurement does not always provide reliable results; hence it is not used in many laboratories.

Relative standardization

In the relative standardization method, a chemical standard (index std) with a known mass w of the element is co-irradiated with the sample of known mass W. When short-lived radionuclides are employed both the standard and sample are irradiated separately under the same conditions, usually with a monitor

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of the same neutron fluence rate and both are counted under the same geometrical arrangements with respect to the gamma-ray energy. It is assumed that the neutron flux, cross section, irradiation times and all other variables associated with counting are constant for the standard and the sample at a particular sample-to-detector geometry. For low-power research reactors such as the MNSR and SLOWPOKE, there is no need for a neutron monitor anytime samples are irradiated since neutron flux in the irradiation sites are fairly stable over a long period of time. The neutron activation equation then reduces to:

$$\rho_{sam} = \frac{[(P_A/t_c)CD]_{sam}[\rho W]_{std}}{[P_A/t_c)CD]_{std}.W_{sam}} \qquad [3.14]$$

Where $(P_A/t_c)_{std}$ and $(P_A/t_c)_{sam}$ are the counting rates for standard and sample respectively, ρ_{std} and ρ_{sam} are the concentrations of the standard and the element of interest respectively, C_{std} and C_{sam} are the counting for standard and sample , D_{std} and D_{sam} are decay factors for the standard and sample respectively.

Equation 3.14 can be rewritten as:

$$\rho_{sam} = \frac{[(P_A/t_c)CD]_{sam}}{CD_{std}.W_{sam}SA} \qquad [3.15]$$

Where SA is defined as $\frac{\left[\frac{p_A}{t_c}\right]_{std}}{[\rho W]_{std}}$ and is the sensitivity of the element.

Using the number of counts under the photopeak area from standardized irradiation and counting conditions, the concentration of the element of interest can be determined.

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Single Comparator (k_0-method) standardization

The k_0-standardisation also known as the single comparator method of NAA is based on the fundamental equation for the calculation of the reaction rate R defined as:

$$R = \int_0^\infty \sigma(v)\phi(v)dv \qquad [3.16]$$

Integration of equation [3.16] yields

$$R = \phi(v)\sigma(v) = \phi\sigma = nv_0\sigma_{eff} \qquad [3.17]$$

When the k_0-method was originally proposed in 1975 it was formulated in the Stoughton-Halperin convention (Simonits *et al.* 1975). In the first practical experimental work dealing with the k_0-method, the Hogdahl convention was used instead (Blaauw *et al.* 1991). This method involves the simultaneous irradiation of the sample and a single nuclide standard such as ^{197}Au.

The activation equation from equation [3.13] using the k_0 method with Au as comparator standards can be written in the form:

$$\rho = \frac{\left(\frac{P_A/t_c}{SCDW}\right)_i}{\left(\frac{P_A/t_c}{SCDW}\right)_{Au}} \frac{M_i\phi_{Au}\gamma_{Au}\sigma_{effAu}\varepsilon_p(E_{Au})}{M_{Au}\phi_{ii}\gamma_i\sigma_{effi}\varepsilon_p(E_i)} \qquad [3.18]$$

Accurate knowledge of the nuclear data, the detector efficiencies and the specific activities of the nuclides in the sample and the monitor are needed for the determination of the concentration in the sample. The application of the k_0-method avoids the problem associated with preparation of individual standards for each element to be determined.

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Types of the k_0 formulations

Hogdahl Convention

According to the Hogahl convention 1962, the (n,γ) reaction rate R (in s^{-1}) per nucleus can be split into two terms:

$$R = \int_0^{v_{cd}} \sigma(v)\phi(v)dv + \int_{v_{cd}}^{\infty} \sigma(v)\phi(v)dv \qquad [3.19]$$

For those (n,γ) reactions with resonance below 0.55 eV (which means the Westcott factor (g(T) =1), the following description can be applied for velocities below v_{cd}:

$$\sigma(v) = \sigma_0 v_0 / v \qquad [3.20]$$

Substituting equation [3.20] into the left-hand integration part of equation [3.19] yields

$$\int_0^{v_{cd}} \sigma(v)\phi(v)dv = \int_0^{v_{cd}} \frac{\sigma_0 v_0 \phi(v)}{v} dv = \sigma_0 v_0 \int_0^{v_{cd}} n(v)dv \qquad [3.21]$$

where $n(v) = \dfrac{\phi(v)}{v}$

Defining the conventional sub-cadmium neutron flux ϕ_{th} by

$\phi_{th} = v_0 \int_0^{v_{cd}} n(v)dv$ and substituting ϕ_{th} into the left-hand integral of equation [3.21] yields

$$\int_0^{v_{cd}} \sigma(v)\phi(v)dv = \sigma_0 \phi_{th} \qquad [3.22]$$

The right-hand integral in equation 3.19 can be written as:

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$$\int_{v_{cd}}^{\infty} \sigma(v)\phi(v)dv = \int_{E_{cd}}^{\infty} \frac{\sigma(E)\phi(E)}{E} dE \text{ as a function of neutron energy} \quad [3.23]$$

Defining the infinitely dilute resonance integral I_0 as:

$I_0 = \int_{E_{cd}}^{\infty} \frac{\sigma(E)}{E} dE$ and the epithermal or intermediate neutron flux per unit in E

neutron energy interval ϕ_{epi} as $\phi(E)$, then equation [3.23] becomes:

$$\int_{v_{cd}}^{\infty} \sigma(v)\phi(v)dv = \int_{E_{cd}}^{\infty} \frac{\sigma(E)\phi(E)}{E} dE = I_0\phi_{epi} \quad [3.24]$$

Substituting equations [3.22] and [3.24] into equation [3.19] yields:

$$R_H = \sigma_0\phi_{th} + I_0\phi_{epi} \quad [3.25]$$

Equation 25 can be written in the form:

$$R_H = \phi_{th}\left(\sigma_0 + \frac{I_0\phi_{epi}}{\phi_{th}}\right) = nv_0\left(\sigma_0 + \frac{I_0\phi_{epi}}{\phi_{th}}\right) \quad [3.26]$$

Defining the flux ratio $f = \frac{\phi_{th}}{\phi_{epi}}$ and $Q_0 = \frac{I_0}{\sigma_0}$, equation 3.26 can be transformed

to: $R_H = \phi\sigma_{eff} = nv_0\sigma_0(f + Q_0)$ [3.27]

Using [3.18] and [3.27] and defining k_0 as: $k_0 = \frac{\theta_i\sigma_{0i}\gamma_i M_{Au}}{\theta_{Au}\sigma_{0Au}\gamma_{Au}M_i}$ the concentration ρ

of an element in a sample using the Hogdhal convention can be written as:

$$\rho = \frac{\left(\frac{P_A/t_c}{SCDW}\right)_i}{\left(\frac{P_A/t_c}{SCDW}\right)_{Au}} \cdot \frac{1}{k_0} \cdot \frac{(f + Q_{0Au})}{(f + Q_{0i})} \cdot \frac{\varepsilon_p(E_{Au})}{\varepsilon_p(E_i)} \quad [3.28]$$

If the term $Q_0(\alpha)$ is introduced into equation [3.28] to take care of the α-corrected

Q_0 value, then equation [3.28] becomes.

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$$\rho = \frac{\left(\frac{P_A/t_c}{SCDW}\right)_i}{\left(\frac{P_A/t_c}{SCDW}\right)_{Au}} \cdot \frac{1}{k_0} \cdot \frac{[f + Q_0(\alpha)_{Au}]}{[f + Q_0(\alpha)_i]} \cdot \frac{\varepsilon_p(E_{Au})}{\varepsilon_p(E_i)} \quad [3.29]$$

The expression for $Q_0(\alpha)$ is given as:

$$Q_0(\alpha) = \frac{Q_0 - 0.429}{E_r^\alpha} + \frac{0.429}{(2\alpha + 1)E_{cd}^\alpha} \quad [3.30]$$

The Hogdahl-convention is restricted to only nuclides that follow the "1/v" (n,γ) reaction rates.

The flux ratio f can be determined using the cadmium ratio (R_{cd}) method as:

$$f = \frac{\phi_{th}}{\phi_{epi}} = Q_0(\alpha)(R_{cd} - 1) \quad [3.31]$$

The cadmium ratio at the irradiation site is defined as

$$R_{cd} = \frac{A_0}{F_{cd} A_{cd}} \quad [3.32]$$

Using two monitors such as Au and Zr, the epithermal neutron flux shape factor α can be obtained from iteration using equation 3.33 as:

$$(R_{cd} - 1)_{Au} Q_0(\alpha)_{Au} = (R_{cd} - 1)_{Zr} Q_0(\alpha)_{Zr} \quad [3.33]$$

Westcott Formalism

For the k_0-NAA standardization method to cover all nuclides, the modified Westcott-formalism [Westcott, 1960] was proposed to deal with "non-1/v" (n,γ) nuclides (De Corte et al, 1993, 1994).

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The modified Westcott-formalism takes into account a non-ideal non-1/E epithermal neutron flux distribution, which is approximated to the shape $1/E^{1+\alpha}$. Westcott defined the reaction rate per nucleus R as:

$$R_W = nv_0\sigma_0[g(T_n) + r(\alpha)\sqrt{T_n/T_0}.s_0(\alpha)] \quad [3.34]$$

Where $\sigma_{eff} = \sigma_0[g(T_n) + r(\alpha)\sqrt{T_n/T_0}.s_0(\alpha)]$

And $s_0(\alpha) = s_0 E_{rr}^{-\alpha}$ (with $s_0 = 2\pi^{-1/2}Q_0$) [3.35]

The concentration ρ of an element in a sample using the Westcott-formalism with Au as a comparator standard can therefore be written in the form:

$$\rho = \frac{\left(\frac{P_A/t_c}{SCDW}\right)_t}{\left(\frac{P_A/t_c}{SCDW}\right)_{Au}} \cdot \frac{1}{k_0} \cdot \frac{g(T_n)_{Au} + r(\alpha)\sqrt{T_n/T_0}.s_0(\alpha)_{Au}}{g(T_n)_t + r(\alpha)\sqrt{T_n/T_0}.s_0(\alpha)_t} \cdot \frac{\varepsilon_p(E_{Au})}{\varepsilon_p(E_t)} \quad [3.36]$$

The expression that can be used to obtain the parameter from the cadmium ratio of a monitor *i.e.* Au, which follows a "1/v" (n,γ) reaction with $g(T_n) = 1.007$ is written in the form (De Corte et al, 1993)

$$r(\alpha)\sqrt{T_n/T_0} = \frac{g(T_n)}{R_{cd}F_{cd}\left(\frac{g_{1/v}(T_n)(lev)^\alpha}{K(1+2\alpha)E_{cd}^\alpha} - \frac{2}{\sqrt{\pi}}W'(\alpha) + s_{0,1/2}(\alpha)\right) - s_{0,1/2}(\alpha)} \quad [3.37]$$

Where $W'(\alpha) = W'E_r^{-\alpha}$ with W' dependent on reactor type and irradiation site, and $K = \frac{1}{4(\pi E_{cd}/E_0)^{1/2}}$; K =2.293 for a 1-mm Cd thickness (Westcott et al., 1958).

The Westcott ($g(T_n)$) factor has been evaluated to give Maxwellian neutron temperature T_n at the irradiation site by co-irradiation of Lu and a 1/v monitor (De

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Corte *et al.*, 1993). The expression for calculating $g_{Lu}(T_n)$ in order to obtain T_n is given by the following expression.

$$g_{Lu}(T_n)=\left(\frac{A_{spLu}/k_{0,AdLu}}{A_{spV}/k_{0,AdV}}\right)\frac{\varepsilon_{p,Lu}}{\varepsilon_{p,V2}}\left(g_{V}(T_n)+r(\alpha)\sqrt{T_n/T_0}\,s_{0,V}(\alpha)\right)-r(\alpha)\sqrt{T_n/T_0}\,s_{0,Lu}(\alpha) \qquad [3.38]$$

The $g(T_n)$ function is evaluated either theoretically (Kim *et al*, 1975) or experimentally (De Corte *et al*, 1994)

Nisle unified Formulation

The calculation of reaction rates is basic to nuclear reactor design. Absorption, capture, fission or scattering events all take place at rates that vary with the energy of the reacting particles. To simplify the calculations, the concepts of effective cross section values have been introduced in various ways but all of them lack unity to various degrees (Nisle 1963). Nisle therefore proposed a unified formulation for the specification of neutron flux spectra in reactors. The unifying principle consisted of two basic ideas: (i) a conventional flux that is measurable by physically realizable detectors and (ii) An effective cross section convention.

An important feature of this system is that nowhere does the cadmium ratio appear in the formulation. It is entirely unnecessary either to the formulation of the system or to the measurement of the parameters specifying any particular case. The essence of the integral method for measuring neutron flux parameters is an energy-average value of an energy-dependent cross-section of a detector material. The Nisle unification method is accomplished by adoption of some

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already existing concepts and abandonment of the "cadmium ratio" concept in favour of one in which the neutron spectrum is divided into Maxwellian and non-Maxwellian components.

The main features of the Nisle formulation are:

(a) It is based on integrated reaction rates,

(b) Flux parameters are measurable by physically realizable detectors,

(c) It is applicable to any cross-section function of energy,

(d) The reactor flux spectrum is expressed in terms of Maxwellian and non-Maxwellian components,

(e) It forms the basis for a universal flux detector capable of measuring all three parameters, neutron temperature, equivalent 1/E component, and conventional flux with a single foil or wire by a single irradiation, and

(f) The resonance integral concept is generalized.

In this formulation, it is assumed that:

(i) The neutron flux distribution can be represented by two major components the Maxwellian and the 1/E components, joined by a transition region,

(ii) Each component can be characterized by a single parameter $E_T = kT$ for the Maxwellian and F_1 for the 1/E component and

(iii) The transition region is related to the Maxwellian by parameter μ, which is relatively constant for a large class of reactors.

For this formulation, the reaction rate R per nucleus is defined as:

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$$R_N = nv_0\sigma_0 P(F_1, T_n) \quad [3.39]$$

Where $P(F_1, T_n)$ is the effective cross section factor (σ_{eff}) that can be calculated from accurate cross section data or obtained from experiments.

The expressions for the neutron flux distributions for Maxwellian ϕ_M and non-Maxwellian $\phi_{n\text{-}M}$ (Nisle 1963) is given as:

$$\phi_M = \frac{nv_0}{[(1+2F_1)(\mu E_T)]^{-1/2}} \quad [3.40]$$

$$\phi_{n-M} = \frac{nv_0 2F_1(\mu E_T)^{-1/2}}{[(1+2F_1)(\mu E_T)]^{-1/2}} \quad [3.41]$$

$$f = \frac{\phi_M}{\phi_{n-M}} = \frac{1}{2F_1(\mu E_T)^{-1/2}} \quad [3.42]$$

$$F_1 = \frac{1}{2f(\mu E_T)^{-1/2}} \quad [3.43]$$

The relationship between the three methods with regards to the effective cross section is:

$$\sigma_{eff} = \sigma_0[f + Q_0(\alpha)] = \sigma_0[g(T_n) + \sqrt{\frac{T_n}{T_0}} s_0(\alpha)] = \sigma_0[P(F_1, T_n)] \quad [3.44]$$

The effective cross section for the Nisle formulation can be obtained from experiment as:

$$P_X(F_1, T_n) = \frac{A_x/N_x\sigma_x}{A_{1/v}/N_{1/v}\sigma_{1/v}} \quad [3.45]$$

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The Generalized k_0-NAA formulation

The general expression for calculating the concentration of an element in any sample using the k_0-NAA method is written in the form. (De Corte *et al.*, 1992)

$$\rho = \frac{\left[\frac{P_A / t_c}{SCDW}\right]^i}{\left[\frac{P_A / t_c}{SCDw}\right]^{Au}} \cdot \frac{1}{k_0} . EPI . \frac{\varepsilon_p^{Au}}{\varepsilon_p^i} \qquad [3.46]$$

Where EPI is defined as

$$EPI = \frac{\left[\sigma_{eff} / \sigma_0\right]^{Au}}{\left[\sigma_{eff} / \sigma_0\right]^i} \qquad [3.47]$$

Therefore for the three conventions, the EPI values are defined as:

$$EPI_H = \frac{f + Q_c(\alpha)_{Au}}{f + Q_c(\alpha)_i},$$

$$EPI_W = \frac{g(T_n)_{Au} + \sqrt{T_n/T_0}\, s_0(\alpha)_{Au}}{g(T_n)_i + \sqrt{T_n/T_0}\, s_0(\alpha)_i},$$

$$EPI_N = \frac{P_{Au}(F_1, T_n)}{P_i(F_1, T_n)}$$

Computerization or measurement of any of the above EPI values for any particular element (i) will allow the calculation of the concentration of that element using equation [3.46]. Akaho and Nyarko (2002) established the relationship between the three EPI factors

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Epithermal instrumental neutron activation analysis based on k_0-standardization method

El Nimr *et al.*, (1981) investigated the applicability of the k_0-standardisation concept in ENAA for 32 isotopes. They concluded that, the k_0-comparator method could be extended and applied in general to epi-cadmium (n,γ) activation analysis. Since then this method has not been judiciously applied to real sample matrix.

The concentration of an element in a sample co-irradiated under cadmium cover together with the comparator standard can be evaluated from gamma-counts measured on a calibrated detector by using the equation

$$\rho = \frac{\left[\frac{P_A / t_c}{SCDW}\right]^{i}_{Cdsample}}{\left[\frac{P_A / t_c}{SCDw}\right]^{Au}_{CdComparato}} \cdot k_{0e} \cdot \frac{F_{cd}^{Au}}{F^{i}_{cd}} \cdot \frac{\varepsilon_p^{Au}}{\varepsilon^{i}_{p}} \cdot \frac{Q'_0(\alpha)}{Q'_0} \cdot \frac{Q_0(\alpha)}{Q_0} \qquad [3.48]$$

Where F_{cd} is the cadmium epithermal neutron transmission factor (mostly ≤1), $Q_0 = (I_0/\sigma_0)$ and $Q_0(\alpha) = [I_0(\alpha)/\sigma_0]$ are the ratios of the resonance integral to the 2200 $m.s^{-1}$ cross section, valid for 1/E and a $1/E^{1+\alpha}$ epithermal neutron flux distribution respectively. k_{0e} is the compound nuclear constant independent of the irradiation and counting conditions, which is defined theoretically as:

$$k_{0e} = \frac{M^{Au}\theta^{i}\gamma_{i}I^{i}_{0}}{M^{i}\theta^{Au}\gamma^{Au}I_{0}^{Au}} \qquad [3.49]$$

and from definition of k_0:

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$$k_{0e} = k_0 \cdot \frac{Q'_0}{Q_0^{Au}} \qquad [3.50]$$

Thus, when substituting the k_{0e} factor in equation 3.48 as a function of k_0 and Q_0 as given by equation [3.50] it can be concluded that, in principle, the k_{0e} comparator method becomes just an extension of the well- k_0-method. Equation 3.45 becomes established

$$\rho = \frac{\left[\frac{P_A / t_c}{SCDW}\right]^i_{sample}}{\left[\frac{P_A / t_c}{SCDw}\right]^{Au}_{cd} k_0} \cdot \frac{F_{cd}^{Au}}{F'_{cd}} \cdot \frac{\varepsilon_p^{Au}}{\varepsilon'_p} \cdot \frac{Q_\alpha^{Au}(\alpha)}{Q'_0(\alpha)} \qquad [3.51]$$

Irradiating a sample and the comparator standard in a 1-mm-thick Cd shield, the concentration of an element (i) in the sample can be determined using equation [3.51].

Interaction of gamma-ray with detector material

Although a large number of possible interaction mechanisms are known for gamma-rays in matter, only three major types play an important role in radiation measurement. They are (i) photoelectric absorption (ii) Compton scattering and (iii) pair production. All of these processes lead to partial or complete transfer of the gamma-ray photon energy to the electron energy in the detector material. The result is sudden and abrupt changes in the gamma-ray photon history, in that the photon either disappears entirely or is scattered through a large average angle (Knoll, 1986).

In the photoelectric absorption process, an incoming gamma-ray photon undergoes an interaction with an absorber atom in which the photon completely

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disappears. In its place, the atom from one of its bound shells ejects an energetic photoelectron. The interaction is with the atom as a whole, and cannot take place with free electrons. For gamma-rays of sufficient energy, the most probable origin of the photoelectron is the most tightly bound or the K shell of the atom. The photoelectron appears with energy given by

$$E_e = h\nu - E_b \qquad [3.52]$$

In addition to the photoelectron, the interaction also creates an ionized absorber atom with a vacancy in one of its bound shells. This vacancy is quickly filled through capture of free electrons from the medium and/or rearrangement of electrons from other shells of the atom. Therefore, one or more characteristic X-ray photons may also be generated. Although in most cases, these X-rays are reabsorbed close to the original site through photoelectric absorption involving less tightly bound shells, their migration and possible escape from irradiation detectors can influence their response. In some fraction of the cases, the emission of an Auger electron may substitute for the characteristic X-rays in carrying away the atomic excitation energy.

The interaction process of Compton scattering takes place between the incident gamma-ray photon and an electron in the absorbing material. It is most often the predominant interaction mechanism for gamma-ray energies typical of radioisotope sources. In Compton scattering, the incoming gamma-ray photon is deflected through an angle θ with respect to its original direction. The photon transfers a portion of its energy to the electron (assumed to be initially at rest), which is known as a recoil electron. Because all angles of scattering are possible,

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the energy transferred to the electron can vary from zero to a large fraction of the gamma-ray energy. The expression, which relates to the energy transfer and the scattering angle for any given interaction can be simply derived by writing simultaneous equations for the conservation of energy and momentum.

It can be shown that:

$$h\nu' = \frac{h\nu}{1 + \frac{h\nu}{m_0c^2}(1 - Cos\theta)} \qquad [3.53]$$

Where

m_0c^2 is the electron rest mass energy (0.511 MeV).

For small scattering angles θ, very little energy is transferred. Some of the original energy is always retained by the incident photon even in the extreme of $\theta = \pi$ (180^0). The probability of Compton scattering per atom of the absorber depends on the number of electrons available as scattering targets and therefore increases linearly with Z.

If the gamma energy exceeds twice the rest mass energy of an electron (1.02 MeV), the process of pair production is energetically possible. As a practical matter, the probability of the interaction remains very low until the gamma-ray energy approaches twice this value and therefore pair production is predominately confined to high-energy gamma-rays. In this interaction (which must take place in the coulomb field of the nucleus), the gamma ray photon disappears and is replaced by an electron-positron pair. All the excess energy carried in by the photon above the 1.02 MeV required to create the pair goes into kinetic energy shared by the positron and the electron. Because the positron will

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be subsequently annihilated after slowing down in the absorbing medium, two annihilation photons are produced as secondary products of the interaction. The subsequent fate of this annihilation radiation has important effect on the response of the gamma-ray detectors.

Gamma-ray spectrometry

Long before the advent of the first commercially available thallium activated sodium iodide (NaI(Tl)) scintillation detectors in the 1950s, counting of irradiated samples were carried out using proportional counters following chemical separation. In the 1950s, when research reactors were made available worldwide, gamma-ray spectroscopy underwent rapid development making multi-elemental analysis possible. However, NaI(Tl) detectors lacked good energy resolution. Around 1960, the first lithium drifted germanium (GeLi) detectors appeared making an improvement in the energy resolution by a factor of about 20 to 30. From that time, gamma-ray spectroscopy has witnessed great improvement leading to manufacture of high efficiency and high-resolution High-Purity Germanium detectors (HPGe).

Compton suppression spectrometry

Since the early 1990's Compton suppression counting has been effectively utilized in NAA especially, in environmental studies and food analyses to lower detection limits for several elements (Lansberger *et al.*, 2005). Compton suppression spectrometry has developed into a well-established position in low-level counting. Compton suppression spectrometry is attractive because of the

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reduction in the Compton continuum, cosmic and natural background. In NAA, CSS can help by increasing the sensitivity of measurement and also substantially reducing spectral interferences.

The principle of the CSS system is based on the Compton effect described as follows, When a gamma-ray interacts with the main detector, the Compton Effect may occur, in which a recoil electron and a scattering photon are created sharing the initial gamma-ray energy. The recoil electron has a short range and deposits its energy in the main detector, while the scattered photon is more likely to escape the main detector. In a normal detection system, the signal from the recoil electron is recorded as a contribution to the background since the energy of the recoil electron is lower than the original gamma-ray energy. In the CSS system, photons passing the main detector are detected by the surrounding NaI(Tl) or the photomultipliers (PMT) detectors. If both the main and NaI(Tl) or PMT detectors record the signals within a specific time interval, the signal is eliminated under the assumption that the signals result from Compton scattering. Through this phenomenon, the background of Compton continuum in the spectrum is reduced to a level much lower than in the normal spectrum, which drastically improves the analytical sensitivity. The capabilities of the CSS methods in multi-elemental NAA and the analytical comparison with large and well-type detectors have been made (Lin *et al.*, 1997; Bode, 1997). The Compton Effect spans the largest energy range of interaction as compared to the photoelectric and the pair production.

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Uncertainty budget calculation

Every analytical result should be reported with its corresponding uncertainty. This trivial statement is being now a day understood and applied in major publications. The problem is, however, the way uncertainties are computed by different laboratories. Following the clear instructions given in the "Guide to the Expression of Uncertainties in Measurements" (1995) issued by the International Organization for Standardization, all analytical processes should be taken from the sampling of the original material to the final measurement.

Chemical measurement process consist of sampling and sample preparation, measurement of the test portion, evaluation of the measurement results (data reduction), and reporting of measurement results in terms of an estimate of the analyte amount (measurand) and its uncertainty (Currie 1995). The approach of reporting the estimate of the measurand together with a measurement uncertainty is different from the classical evaluation of the mean value of repetitive measurements and its standard deviation (in neutron activation analysis NAA, even the counting statistics from only single measurement were sometimes used to estimate the standard deviation).

Uncertainty (of measurement) is defined (Guide to the Expression of Uncertainty in Measurements ISO 1995; Quantifying Uncertainty in Analytical Measurement, EURACHEM, 1995) as "a parameter associated with the results of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand". Its quantification is of utmost importance in all types of measurements, and therefore the analytical community developed guidelines (Guide to the Expression of Uncertainty in Measurements,

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ISO, 1995; Guide to the Expression of Uncertainty in Measurement, 1992) with examples for some analytical techniques for better understanding of this evaluation methodology. Recently, the International Atomic Energy Agency (IAEA) took the initiative in developing such guidelines with examples for selected nuclear and nuclear related measurement techniques including NAA; in the form of a technical document (TECDOC). (Quantifying Uncertainty in Nuclear Analytical Measurements, 2004)

Basic rules for the quantification of uncertainty

The first step of the uncertainty specification process is a clear statement of what is being measured and the relation between it and the parameters on which it depends. Then, sources of uncertainty are identified for each part of the analytical process or each parameter, followed by estimation of the size of each uncertainty. At this stage, approximate values suffice; significant values can be refined in subsequent stages. The next stages involve conversion of each uncertainty component to a standard deviation and calculation of combined uncertainty. The significant uncertainty components are identified and re-evaluated, if needed, and then the final combined uncertainty is calculated. The final step of the uncertainty quantification is calculation of the expanded uncertainty by means of a coverage factor (Guide to the Expression of Uncertainty in Measurements, ISO, 1995; Quantifying Uncertainty in Analytical Measurement, EURACHEM, 1995)

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Survey of uncertainty sources in NAA

In general, the sources of standard uncertainty u_i can be grouped according to the individual steps of analysis into four categories: (1) preparation of the sample and comparator (standard, k_0-factors, neutron fluence rate monitor); (2) irradiation; (3) gamma-ray spectrometry measurement; and (4) radiochemical separation, if performed.

Uncertainty components in Relative method

Since the same irradiation and counting times are generally used for the samples and standards, the NAA equation using the relative method can be simplified as:

$$\rho = \frac{(Np)_a D_{st} w}{(Np)_{st} D_a W} \qquad [3.54]$$

Since equation [3.54] involves only multiplication and division of quantities, the combined standard uncertainty $u\rho(c_m)$ can be calculated according to equation

$$u\rho(c_m) = c_m \sqrt{u_1^2 + u_2^2 + u_3^2} \qquad [3.55]$$

where the individual components of $u\rho(c_m)$ are expressed as relative standard uncertainties of the uncertainty sources in a particular analytical steps entering the variables in equation [3.54]. Table 3.1 shows the quantities to be considered.

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Table 3.1: Origin and typical magnitude of uncertainties in INAA

Uncertainty Component	Origin	Typical standard uncertainty
U_1	Sample and Comparator preparation	
u_{1a}	Mass determination of a sample	0.015% to 0.1% for 100mg sample
u_{1b}	Mass determination of comparator	0.075% to 0.75%
u_{1c}	Mass change of samples due to moisture uptake during weighing	Negligible to 1%
u_{1d}	Concentration of comparator (standards), purity and stochiometry of chemicals used for the preparation of standards and/or the uncertainty of k_0-values	0.1% to several %
u_{1e}	Variation of isotopic abundance	Negligible for most elements
u_{1f}	Blank variation and the necessary correction	0.1% to several %

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Table 3.1: Continued

Uncertainty	Origin	Typical standard uncertainty
U_2	Irradiation	
u_{2a}	Irradiation geometry difference	<0.1% to 0.5%
u_{2b}	Neutron self-shielding/scattering difference	~ 0.1% in most cases
u_{2c}	Timing of irradiation	Negligible to 0.3%
u_{2d}	Nuclear reaction interferences	Negligible to ~ 1%
u_{2e}	Neutron spectrum variation in time and space	Negligible to ~ 1%
u_{2f}	Volatilization losses during irradiation	Negligible to several % (for Hg. etc.)

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Table 3.1: Continued

Uncertainty	Origin	Typical standard uncertainty
U_3	γ-ray spectrometry measurement	
u_{3a}	Counting statistics	Usually 0.2%-30%
u_{3b}	Counting geometry difference	~ 0.1% to 20%
u_{3c}	Pulse-pileup losses (random coincidence)	~ 0.1% to 0.5%
u_{3d}	True coincidence (cascade summing)	~1%(irrelevant in the relative method)
u_{3e}	Dead-time effects	Negligible, necessary for short-lived nuclides
u_{3f}	Decay timing effects	Negligible, necessary for nuclides ($T_{1/2}$< 1m)
u_{3g}	Timing of counting	Negligible in most case
u_{3h}	γ-Ray self-shielding	~ 0.1% to 0.5%, especially for E_γ<100 keV
u_{3i}	γ-Ray interferences	~ 0.3% to 1%
u_{3j}	Peak integration method	~ 0.5% to several % (for multiple)
u_{3k}	Blank correction (due to counting room/shielding background)	Negligible in most cases

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Uncertainty components in k_0-NAA

De Corte performed the first uncertainty evaluation for the k_0-NAA in 1987. The general formulae for error propagation was applied and led to "an overall uncertainty of less than 4.0%. (De Corte, 1987). Nowadays a simpler model of error propagation is recommended in the EURACHEM document "Quantifying Uncertainty in Analytical methods". It is based on the "Universally applicable spreadsheet techniques" developed by Kragten, (1994). According to the GUM, the total expanded uncertainty is composed by uncertainties statistically evaluated, (cf. Type A uncertainty), and by uncertainties based on relevant information available or scientific judgement (cf. Type B uncertainty).

The relevant k_0-NAA constants taken from the literature (De Corte, 1987) and the corresponding uncertainties (cf. Type B) are:

$t_{1/2}$ - half-life	$u(t_{1/2})$
E_r – resonance energy	$u(E_r)$
Q_0 – resonance integral to thermal cross section ratio	$u(Q_0)$
k_0 constants	$u(k_0)$
The standard uncertainty of the coincidence correction factors, COI was estimated by De Corte	u(COI)=1.5%
The gold composition in the IRMM-530R Al-0.1%Au alloy is reported in the IRMM-530R certificate	u(Au) = 1.0%

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The neutron flux parameters

f – thermal to epithermal neutron flux ratio $u(f)= 2.1\%$

α - Deviation from the 1/E epithermal flux distribution $u(\alpha) = 11\%$

Type A Uncertainties

ε_p – detector efficiency of gamma ray energy $u(\varepsilon_p) = 1.5\%$

w – mass of sample $(w)= < 0.1\%$

Np – Net peak area $u(Np)=0.1\text{-}10\%$

All these parameters allow the computation of the uncertainty associated with the determination of the elemental concentration using the k_0-NAA method. The concentration ρ_i of an element in a sample can be written as:

$$\rho_i = \frac{A_{sp}}{F_c . k_{0(i)} G_s} \quad [3.56]$$

The first order approximation of the combined uncertainty $u(\rho_I)$ can be described as follows:

$$u(\rho_i)^2 = u(Np_i)^2 + u(G_s)^2 + u(F_c)^2 \quad [3.57]$$

Where; $Fc = 10^{-6} A_{spm}/G_m$

$$A_{spi} = \frac{Np}{S.D.C.t_c.w.COI} \quad [3.58]$$

$$G_i = [f + Q_{0i}(\alpha)].\varepsilon_i \quad [3.59]$$

Considering the known uncertainties presented, one gets the equation:

$$u(\rho_i)^2 = u(Np_i)^2 + (2.6)^2 \quad [3.60]$$

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This indicates that the k_0-NAA systematic uncertainty is generally of the order of 2.6% using low-power research reactor. Following GUM's recommendation the experimental result ρ is then reported based on "n" replicates with corresponding uncertainty as follows:

$$\rho(k=2) = \rho_i \pm 2\sqrt{\frac{u(Npi)^2}{n} + (2.6)^2} \qquad [3.61]$$

Where the individual components of $\mu\rho(c_m)$ are expressed as relative standard uncertainties of the uncertainty sources in a particular analytical step entering the variables in equation [3.55]. Table 3.1 shows the quantities to be considered.

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CHAPTER 4

EXPERIMENTAL

This chapter deals with the experimental work. The equipment, chemicals and standards used are given. The procedures for sample preparation, irradiation, measurement, counting, and the methods of analysis are described below.

Sampling and sample preparation

Individual food samples were obtained from local markets and farms in the three northern (Upper East and West, Northern) and three southern regions (Greater Accra, Central and Western) of Ghana. Some of the samples were freeze-dried for 48-72 h using a tray type (Christ LMC-1) freeze-dryer at the Ghana Research Reactor-1 Centre, Department of Nuclear Engineering, National Nuclear Research Institute of Ghana Atomic Energy Commission. The rest of the samples were oven dried at 40°C for 48 h using a Gallenkamp oven situated at the same location. The dried samples were homogenized and then sterilized using 50 kCi Gamma Irradiator at the Radiation Technology Centre of GAEC. The samples were shipped to the Dalhousie University SLOWPOKE-2 Reactor facility.

Six replicate samples of each food item and standard reference materials were weighed directly into pre-cleaned 2.0-mL polyethylene vials, which were

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capped and heat-sealed. The sample size varied from 350 and 850 mg depending upon the density of the material. The vials were then placed in 7.0-mL polyethylene irradiation vials which were again capped and heat-sealed. All vials were obtained from the Olympic Plastic Company, USA. The vials were pre-cleaned by washing them first with distilled water and then soaked in 1:4 reagent grade HCl for 24 h, then rinsed with distilled deionized water. The vials were further soaked in 1:4 ultrapure HNO_3 for 24 h. They were then rinsed thoroughly with DDW and air-dried in a clean fumehood. The moisture content of the samples were determined using the conventional oven drying method. An empty moisture pan and two filter discs were placed in a drying oven at 100-102°C for 5 min. The pans and discs were then removed, placed in a desiccator for an additional 5 min, and weighed. About 2.0 g of each sample was put in one disc and covered with the second disc, put on the dried pan, and placed in an oven for 16-18 h. Samples were removed from the oven, cooled in a desiccator, and weighed to a constant weight. The moisture contents were then calculated. Moisture contents of the samples were found to be in the range 1% to 95% depending on sample type.

Quality assurance and quality control

To ensure good quality assurance, 12 iodine standards were prepared and irradiated by the TINAA and EINAA methods. The number of counts in each of them was noted. The results were statistically evaluated; *i.e*, the mean, variance and the standard deviation were calculated. The process was repeated twice after two

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weeks decay and the same principles applied to results. The errors in the analyses were found to be within two standard deviations. The realistic detection limits were calculated for ^{128}I. This procedure was applied to the rest of the elements determined in this work.

Validation of the analytical methods

The analytical methods were validated by the analyses of standard reference materials obtained from National Institute of Standards and Technology USA

Elemental comparator standards

Apart from bromine, chlorine and iodine, comparator standards of all other elements used in this work were prepared from single element standard solutions. The element comparator standards were made from Plasma Emission Spectroscopy Standards Solutions supplied by Seigniory Chemical Products (SCP) Canada, Ltd. The standards had certified purity of >99.99% and had concentrations of between 100 and 10,000 $mg.kg^{-1}$. Stock solutions of about 0.1 and 1000 mg.kg-1 were prepared from the standard solutions. Volumes of 100 µL of the working standard solutions were pipetted onto 2.0-mL polyethylene vials half filled with high-purity sucrose (obtained from Koch Light Laboratories, USA). Twelve replicate standards of each element were prepared. Few drops of Deionized distilled water were added to form a homogeneous mixture and then dried under an infrared lamp before heat-sealing the cap.

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The comparator standards of Cl and I were made from Fisher Scientific Company Certified ACS grade potassium chloride (KCl) and potassium iodide (KI) solids. "Analar" grade of Potassium bromide (KBr) supplied by BDH Chemicals Canada Limited was used for preparing Br comparator standard. One half of a gram of BDH "Analar" grade potassium hydroxide (KOH) per liter was added to the above standard solutions. The KOH made the solutions slightly basic and thus stabilized the iodide in solution, a procedure developed at the Dalhousie University SLOWPOKE-2 Facility. After preparation of the solutions, the comparator standards were made in the same manner as for the other elements.

The elemental Au standard used in the k_0 calculations was made from plasma Emission Spectroscopy standard solutions supplied by Seigniory Chemical Products (SCP) Canada Ltd. The standard had a certified purity of >99.999% and had a concentration of 1000 ppm. A working stock solution of 10 ppm was prepared by dilution. A volume of 100 µL was taken from the standard solution and pipetted into 2.0 mL polyethylene vial half filled with finely ground sucrose (obtained from Koch Light Laboratory, USA). Ten replicate standards were prepared. Few drops of deionized distilled water DDW were added to form a homogeneous mixture and dried before heat sealing the cap.

Analysis of Standard Reference Materials

Five SRMls obtained from National Institute of Standards and Technology, USA, namely, Bovine liver NIST 1577b, Oyster tissue NIST 1566b,

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Peach leaves NIST 1547, non fat milk powder NIST 1549, and Apple leaves NIST 1515 were weighed directly into 2.0-mL vials. To obtain reproducible geometry, all vials were half filled. The mass of the samples ranged between 350 and 850 mg depending on the density of the sample

Neutron spectrum characteristics of SLOWPOKE-2 and GHARR-1 reactors

A 0.1% Au-Al wire, obtained from Reactor experiment USA and or 1000 ppm Au solution obtained from SPEX industries Inc., Canada were used for the neutron flux characterization. About 5.5 mg weight of the Au-Al wire was used. Gold standards of about 5.0 µg were prepared from the 1000 ppm high purity spectroscopic grade Au solution. Thin Zr and Ni foils of masses between 4.5 and 15.0 mg were prepared. The targets of lutetium were prepared from spectroscopic grade $Lu(NO)_3$ solution into polyethylene vials.

Sample irradiation, counting and analysis

Samples were either irradiated in the inner irradiation sites of either DUSR or GHARR-1 when thermal neutron activation analysis was preferred. Epithermal irradiations were carried out in the outer irradiation sites of the reactors. DUSR operated at quarter powers of 4.0 kW and GHARR-1 at half-full power of 15.0 kW at thermal neutron fluxes of 2.5×10^{11} $cm^{-2}s^{-1}$ and 5.0×10^{11} $cm^{-2}s^{-1}$, respectively. Most of the irradiations were carried out using DUSR in the pneumatic irradiation inner site #2 and outer sites #9 (Cd-lined) and #10. Samples were irradiated in the inner #1 and outer #6 of the GHARR-1.

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Irradiation conditions were categorized under three conditions namely, short, medium and long. The experimental conditions for the short-lived nuclides using thermal neutrons and anti-coincidence counting was 10 s irradiation, 20 s decay and 40 s counting times and for the medium-lived, 1 min irradiation, 2 min decay and 10 min counting times were used. Nuclear data for the elements determined are presented in Table 4.1 (De Corte et al, 2003; Practical Aspect of Operating a Neutron Activation Analysis Laboratory, IAEA-TECDOC, 1990). EINAA and anti-coincidence counting methods were developed for determination of iodine in three SRMs and 79 food items with 30 min irradiation, 5 min decay and 20 min counting times. Other elements determined by EINAA and anticoincidence counting included As, Au, Br, I, Sb, Sr and U. The conditions for the determination of these long-lived nuclides were 3 h irradiation, between 3 days to 1 month decay and 10 h counting times.

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Table 4.1: Continued

Element	Target Isotope	Isotopic abundance (%)	Thermal cross section σ_{th} (barns)	Integral Resonance I_0 (barns)	Product Nuclide	Half-life	Gamma-ray energy keV	γ-yield (%)
Eu	^{151}Eu	47.80	5900	5564	^{152}Eu	133.33y	1408.0	20.85
Fe	^{58}Fe	0.28	1.31	1.28	^{59}Fe	44.5d	1099.25	56.5
							1291.6	43.2
Hf	^{178}Hf	27.30	53.0	1039	^{179}Hf	18.68 s	214.5	82.0
	^{180}Hf				^{181}Hf	42.39d	482.21	50.60
I	^{127}I	100	4.04	100	^{128}I	24.99 min	442.90	16.90
K	^{41}K	6.73	0.423	0.29	^{42}K	12.36 h	1524.58	18.80
La	^{139}La	99.91	9.34	11.6	^{140}La	40.27h	487.02	44.27
							1596.2	95.4
Lu	^{176}Lu	2.60	2100	1160	^{177}Lu	6.71d	208.36	11.0
Mg	^{26}Mg	11.01	0.0372	0.024	^{27}Mg	9.46 min	1014.43	28.60
Mn	^{55}Mn	100	13.2	13.9	^{56}Mn	2.58 h	1810.72	27.19
Mo	^{98}Mo	24.13	0.131	6.96	^{99}Mo	65.94h	140.51	90.7
							739.58	12.13
Na	^{23}Na	100	0.513	0.303	^{24}Na	14.96 h	1368.6,	100
							2754	99.94
Rb	^{85}Rb	72.17	0.050	1.16	^{86m}Rb	1.02 min	555.4	98.19
	^{85}Rb	72.17	0.494	7.31	^{86}Rb	18.66d	1076.6	8.76
S	^{36}S	0.02	0.16	0.18	^{37}S	5.05 min	3103.98	94.00
Sb	^{121}Sb	57.3	6.33	209	^{122}Sb	2.7d	564.2	62.00
Sc	^{45}Sc	100	9.60	-	^{46m}Sc	18.75 s	142.53	62.00
Se	^{76}Se	9.00	21.0	16	^{77m}Se	17.45 s	161.93	52.40

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Table 4.1: Continued

Element	Target Isotope	Isotopic abundance (%)	Thermal cross section σ_{th} (barns)	Integral Resonance I_0 (barns)	Product Nuclide	Half-life	Gamma-ray energy keV	γ-yield (%)
Sm	^{154}Sm	22.70	7.74	33.3	^{155}Sm	22.3 min	104.18	28.82
Sn	^{124}Sn	5.79	0.116	6.97	^{125m}Sn	9.52 min	332.10	99.57
Sr	^{86}Sr	9.86	0.770	3.17	^{87m}Sr	2.81 h	388.4	82.26
Ta	^{181}Ta	99.99	20.4	679	^{182}Ta	114.5d	1221	27.10
Ti	^{50}Ti	5.40	0.171	0.115	^{51}Ti	5.76 min	320.08	93.1
	^{232}Th	100	7.26	83.7	^{233}Th	22.53 min	86.53,	2.60
Th							459.3	1.40
	^{232}Th	daughter	daughter	daughter	^{233}Pa	27.0d	312.01	36.00
U	^{238}U	99.27	2.75	284	^{239}U	23.47 min	74.66	50.0
	^{238}U	daughter	daughter	daughter	^{239}Np	2.36d	277.6	14.2
V	^{51}V	99.75	4.79	2.63	^{52}V	3.75 min	1434.08	100
W	^{186}W	28.6	38.7	530	^{187}W	23.9h	479,57	21.13
							685.74	26.39
Zn	^{68}Zn	18.80	0.0699	0.233	^{69m}Zn	13.76 h	438.63	94.8
	^{64}Zn	48.6	0.726	1.42	^{65}Zn	243.9d	1115.55	50.70

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Pseudo cyclic activation analysis

The elemental comparator standards, SRMs, and food samples were irradiated in the pneumatic irradiation inner site #2 of the DUSR facility operating at quarter power of 4.0 kW and a thermal neutron flux of 2.5×10^{11} $cm^{-2}s^{-1}$. The stability, homogeneity, and reproducibility of the DUSR neutron flux have previously been reported (Holzbecher *et al.*, 1985). Ten seconds irradiation, 20 s decay and 40 s counting times were selected. Fifty seconds delay time between cycles was allowed. Counting of samples was done using the CSS system operating in the anticoincidence mode.

Detector full-energy photopeak efficiency determination

Two mixtures of radionuclide solutions (Product codes QCYB41 and QCY48) from Amersham Bucher GmbH & Co were obtained for efficiency calibration of Ge(Li) and Compton suppression spectrometers. The QCYB41 mixture contains the following radionuclides: ^{133}Ba, ^{57}Co, ^{139}Ce, ^{85}Sr, ^{137}Cs, ^{54}Mn, ^{65}Zn and ^{88}Y while QCY48 consists of ^{241}Am, ^{109}Cd, ^{57}Co, ^{139}Ce, ^{203}Hg-, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{60}Co, and ^{88}Y. One hundred µL of each solution was diluted to 1 000 µL with DDW in a 2.0 mL pre-cleaned polyethylene vials. The solutions were then counted at different distances from the top of the detectors. These distances varied between 0 and 10.0 cm from the surface of the conventional Ge(Li) detector, and from 0 to 4.0 cm for the HPGe detector of the CSS counting system.

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The principal detectors used in this work, in both conventional and Compton suppression gamma-ray spectrometry, consisted of an EG & G Ortec high purity germanium (HPGe) p-type coaxial detector with a crystal diameter of 51.2 mm and length of 65.2 mm. The detector had a peak-to-Compton ratio of 93:1, a relative efficiency of 25% with respect to the standard NaI(Tl) detector and resolution of 1.8 keV at 1332.5 keV photopeak of ^{60}Co. The guard detector used in the CSS system consisted of a 10"X10" Na(Tl) annulus with five photomultiplier tubes (PMTs) supplied by Teledyne. The peak-to-Compton plateau ratio of this system was 582:1 at 662 keV photopeak of ^{137}Cs, using the IEEE convention of the number of counts per channel in the Compton plateau (358-382). The principles and techniques of Compton suppression gamma-ray spectrometer are described in details elsewhere (Knoll, 1986; Landsberger 2005). The block diagram of the Compton suppression counting system used in this work is shown in Fig. 4.1. The HPGe detector was inserted into one end of the annular guard detector and the NaI(Tl) plug at the other end, Two separate power supplies were used for the two guard detectors to ensure equal gains. The major difference between the anticoincidence and the conventional spectrometer is that the former has two electronics. If the conventional gamma-ray spectrum was to be acquired, a simple switch was used to change it from the anticoincidence setting. The detailed description of this system is documented (Zhang, 1997, Beazkley, 1987). The samples were counted at the main detector surface for the PCINAA of the short-lived nuclides and at 2.6 cm for all other elements.

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At the GHARR-1 Centre in Ghana, the gamma-ray spectrometry used consisted of HPGe detector model GR 2518 with an 8K Ortec Maestro Multichennel Analyzer (MCA) card and emulation software. The detector operated on a bias voltage of (-ve) 3000 V, and had a resolution of 1.8 keV (FWHM) for ^{60}Co gamma ray energy of 1332 keV. By means of the MCA card, the spectra intensities of the samples were accumulated for a preset time. The samples were counted at a distance of 7.2 cm from the top of the detector surface.

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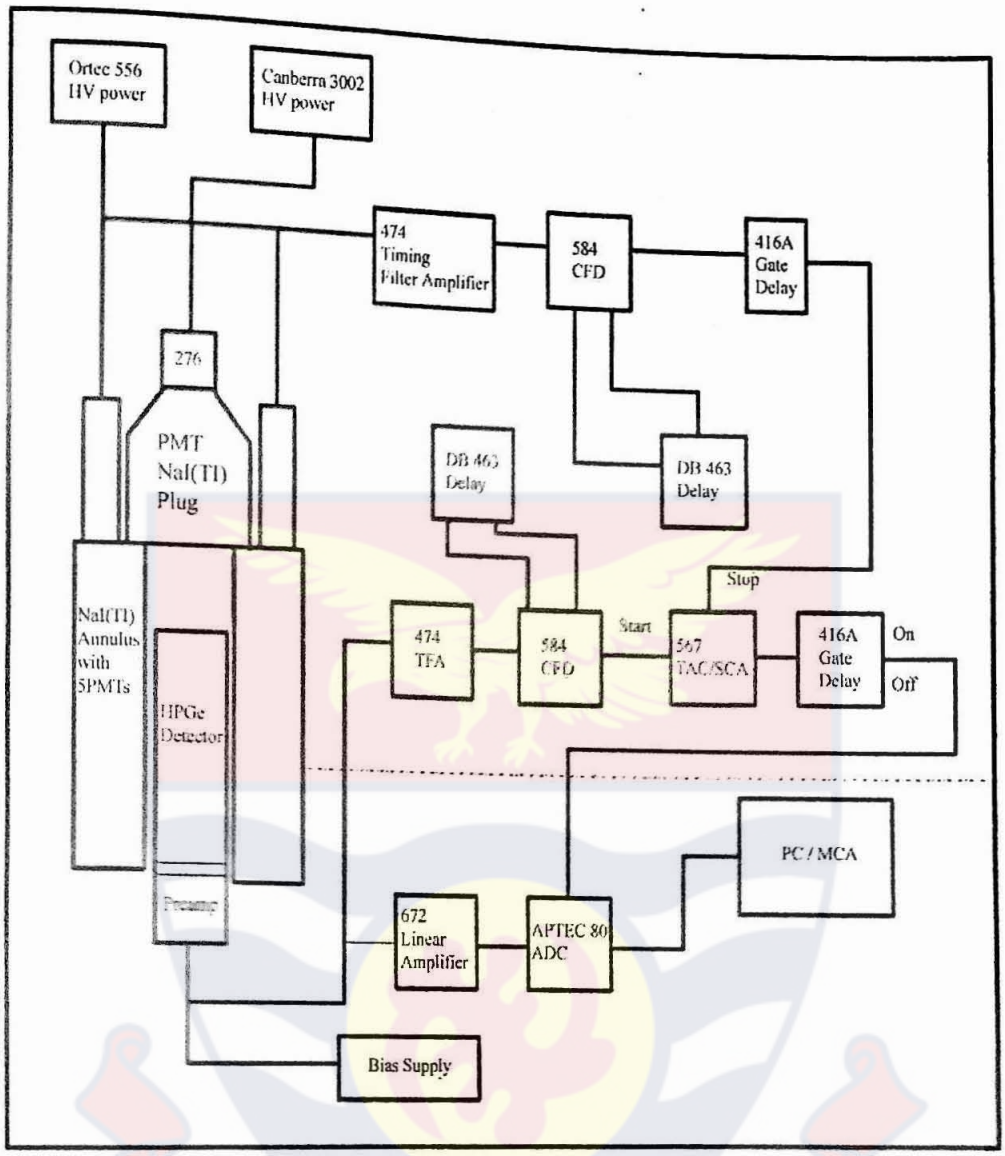

Fig. 4.1: Block diagram of the Compton suppression gamma-ray spectrometry system used in this work

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Relative method

After irradiation and counting of the samples, under well-defined experimental conditions, the net counts were obtained by integrating the photopeak area using the Gaussian function. The peak areas obtained from the comparator standards were used to calculate the sensitivities of the elements. These sensitivities were then used to calculate the concentrations of the elements in the sample.

k_0-Method

The flux parameters of Hogdhal, and Wescott were determined by irradiating Au-Al wires and Zr foils bare and Cd covered. Neutron temperatures for the Westcott formalism were obtained by irradiating $Lu(NO)_3$ solution bare and Cd covered. Both the bare and Cd-covered (1-mm thickness) were irradiated in one outer and one inner irradiation sites of DUSR and GHARR-1. The effective cross sections of the elements of interest using the Nisle formulation were obtained experimentally or by calculations. Irradiation times were varied from 30 min to 2 h. After appropriate cooling, samples were assayed using the Gamma-ray detectors at the facilities already described. The samples and the single comparator Au standards were also irradiated in the characterized irradiation sites. The peak areas of each photopeak of interest were obtained and used for the calculation of the elemental concentration using the appropriate equation.

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CHAPTER 5

RESULTS AND DISCUSSION

In this chapter, the experimental results are presented and discussed. For simplicity and easy understanding of the data, the English names and local names of the food items were used instead of their scientific names.

Comparator standards and quality assurance

Internal quality assurance and control charts

Elemental comparator standards were prepared and analyzed by different INAA methods. These were then used to determine the sensitivities for the elements, which in turn were used to calculate the concentrations of the elements by the relative method. A number of standards were analyzed over the duration of the experiment, which serve as a means of monitoring the performance of the experiments

The control charts for iodine using PCEINAA with five cycles of irradiations and anticoincidence counting are shown in Fig.5.1. Each major horizontal division is one unit of standard deviation (σ). The upper and lower warning limits are given by $\pm 2\sigma$. Results more than $\pm 3\sigma$ from the mean are beyond the control limit and considered to be influenced by determination errors.

Sensitivities of all elements determined were obtained in a similar manner using the appropriate INAA method and gamma-ray spectrometry.

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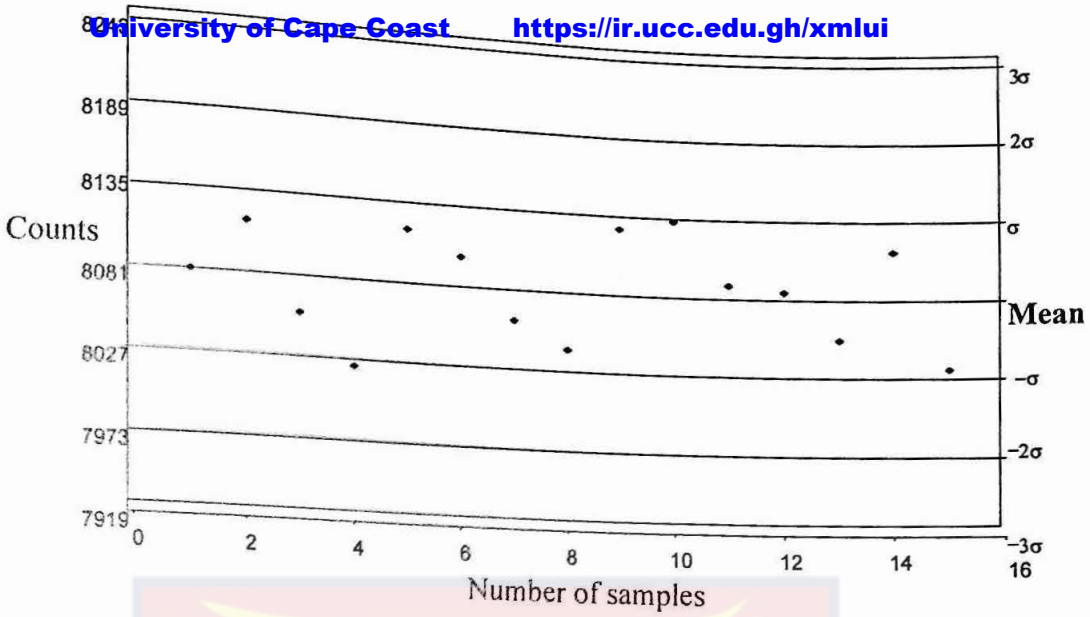

Fig. 5.1 Control chart of iodine sensitivities using five cycles EINAA and anticoincidence counting (t_i=30 min t_d= 5 min t_c= 20 min)

SLOWPOKE-2 and GHARR-1 neutron flux spectrum characteristics of Hogdahl convention, Westcott formalism and Nisle unified formulation

The nuclear data (Meons *et al.*, 1984; De Corte *et al.*, 1994, 2003; Practical Aspect of Operating a Neutron Activation Analysis Laboratory, 1995) required for calculation of the neutron flux parameters are listed in Table 5.1. Table 5.2 shows the measured parameters of the three conventions for the inner #2 and outer #10 of DUSR and inner #1 and outer #6 of GHARR-1 irradiation sites. The values obtained for Hogdahl convention and Westcott formalism are in good agreement with those obtained by Acharya and Chatt, (2003) for the DUSR facility. In the same way, the neutron flux parameters for Hogdahl convention, Westcott formalism and Nisle are in good agreement with those obtained by Akaho and Nyarko, (2002) for the GHARR-1 facility. The computed EPI values of Hogdahl convention, Westcott formalism and Nisle unified formulation for the monitor ^{197}Au for irradiation sites # 2 of DUSR are given in Table 5.3. The computed EPI values were used in calculating the concentration of the elements

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of interest using equation 2.6. Since there were no significant differences in the EPI values, any of the three conventions can be used for the calculation of the concentration of the elements of interest. As reported by Akaho and Nyarko, (2002), the generalized k_0-NAA standardization equation holds for all kinds of nuclides whether it is a 1/v or non-1/v nuclide.

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Table 5.1: Nuclear data of nuclides used for the neutron flux characterization

Target Isotope	Q_0	S_0	E_r	F_{cd}	W'	g (20^0C)	Product Isotope	$t_{1/2}$	Eγ (keV)	k_{0Au}
^{197}Au	15.7	17.24	5.65	0.991	0.055	1.007	^{198}Au	2.695d	411.8	1
^{94}Zr	5.306	5.503	6260	1	0	1	^{95}Zr	64.02d	724.2; 756.7	9.32×10^{-5}
^{96}Zr	251.6	283.4	338	1	0	1	^{97}Zr	16.24h	743	1.149×10^{-4}
^{176}Lu	1.4804	1.67	0.158	-	-	-	^{177}Lu	6.7d	208	0.0714

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Table 5.2: SLOWPOKE-2 and GHARR-1 neutron flux parameters of Hogdhal convention, Westcott formalism and Nisle formulation

Flux Parameter	SLOWPOKE-2		GHARR-1	
	Ch. # 2 (inner)	Ch. # 10 (outer)	Ch. # 1 (inner)	Ch. #6 (outer)
α	-0.0422 ± 0.0051	-0.0098 ± 0.0033	-0.104 ± 0.011	-0.0261± 0.0020
f	18.9 ± 0.6	56.9 ± 2.5	18.8 ± 0.7	49.5 ± 3.2
MSI= $r(\alpha)\sqrt{T_n/T_0}$	0.0449 ± 0.0021	0.01497 ± 0.0012	0.0418 ± 0.0011	0.0159 ± 0.0016
T_n(K)	312.0 ± 5.5	306.0 ± 4.0	300.0 ± 4.5	293.0 ± 3.5
$P(F_1,T_n)$	1.2585 ±0.14	1.3244 ± 0.15	1.8695 ± 0.17	1.2938 ± 0.12
F_1	0.0083 ± 0.0003	0.0028 ± 0.0001	0.0082 ± 0.0005	0.0031 ± 0.0001

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Table 5.3: Nuclear data and EPI values used in the k_0 NAA calculations

Target Isotope	Formed Isotope	Half–life ($t_{1/2}$)	γ-ray energy keV	k_o- Values	EPI_W	EPI_H	EPI_N
^{75}As	^{76}As	26.32 h	559	$4.97X10^{-2}$	0.9266	0.9266	0.924
^{138}Ba	^{139}Ba	83.1 m	165	$1.05X10^{-3}$	1.8210	1.8208	1.8203
^{81}Br	^{82}Br	35.3 h	776	$2.76X10^{-2}$	0.7484	0.7484	0.7471
^{50}Cr	^{51}Cr	27.7 d	320	$2.62X10^{-3}$	1.9179	1.9178	1.9273
^{37}Cl	^{38}Cl	37.2 d	1642	$1.97X10^{-3}$	1.8699	1.8699	1.8679
^{164}Dy	^{165}Dy	2.33 h	94.7	$3.57X10^{-1}$	1.9614	1.9613	1.9611
^{58}Fe	^{59}Fe	44.5 d	1099	$7.77X10^{-5}$	1.8471	1.8470	1.8481
^{127}I	^{128}I	24.9 m	442.8	$1.12x10^{-2}$	0.740	0.7402	0.7421
^{41}K	^{42}K	12.36 h	1525	$9.46X10^{-4}$	1.7940	1.7940	1.1781
^{55}Mn	^{56}Mn	2.58 h	847	$4.96X10^{-1}$	1.8140	1.8140	1.8142
^{23}Na	^{24}Na	14.66 h	1369	$4.68X10^{-2}$	1.8847	1.8846	1.8848
^{121}Sb	^{122}Sb	2.72 d	564	$4.38X10^{-2}$	0.6136	0.6138	0.6133
^{50}Ti	^{51}Ti	5.76 m	320	$3.74X10^{-4}$	1.8536	1.8535	1.8530
^{51}V	^{52}V	3.75 m	1434	$1.96X10^{-1}$	1.8915	1.9150	1.9120
^{64}Zn	^{65}Zn	244 d	1116	$5.72X10^{-3}$	1.6758	1.6758	1.6755

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Detector full-energy photopeak efficiency calibration

The full-energy photopeak efficiencies of the counting systems were obtained from using two radionuclide standard solutions. These were used to calculate the efficiencies of the Compton suppression gamma-ray spectrometry system in the conventional and the anticoincidence modes and the GeLi #1 detector at the DUSR facility at different counting positions. Table 5.4 shows the efficiency values at 2.6 cm from the top of the surface of the CSS system operating in the anticoincidence mode where most of the counting of the samples was carried out. Fig.5.2. shows the efficiency curves for three different geometric positions from the surface of the CSS system in AC mode. It was observed that, the gamma-rays of radionuclides like Ba and Co were suppressed in the AC because they emit cascading gamma-rays and were not included in the efficiency calibration. The efficiency calibration of the HPGe detector used in this work at GHARR-1 centre was obtained by Osae *et al*, 1999. The efficiencies of all the gamma-ray energies of the elements determined were obtained using the fitted curves.

Table 5.4: Efficiency values at 2.6 cm from the surface of the HPGe detector of the Compton suppression system in anticoincidence mode

Radionuclide	Energy(keV)	γ-yield (%)	Efficiency
^{241}Am	59.54	35.70	0.027578
^{109}Cd	88.03	3.61	0.0554543
^{57}Co	122.10	85.20	0.028323

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Table 5.4: Continued

Radionuclide	Energy (keV)	γ-yield (%)	Efficiency
^{139}Ce	165.90	80.10	0.027310
^{203}Hg	279.20	81.46	0.009830
^{113}Sn	391.70	64.89	0.020008
^{65}Sr	514.00	99.27	0.010172
^{137}Cs	661.60	85.21	0.009362
^{88}Y	898.00	93.52	0.002428
^{60}Co	1172.00	99.90	0.001738
^{60}Co	1333.00	99.98	0.001544
^{88}Y	1836.00	99.36	0.001192

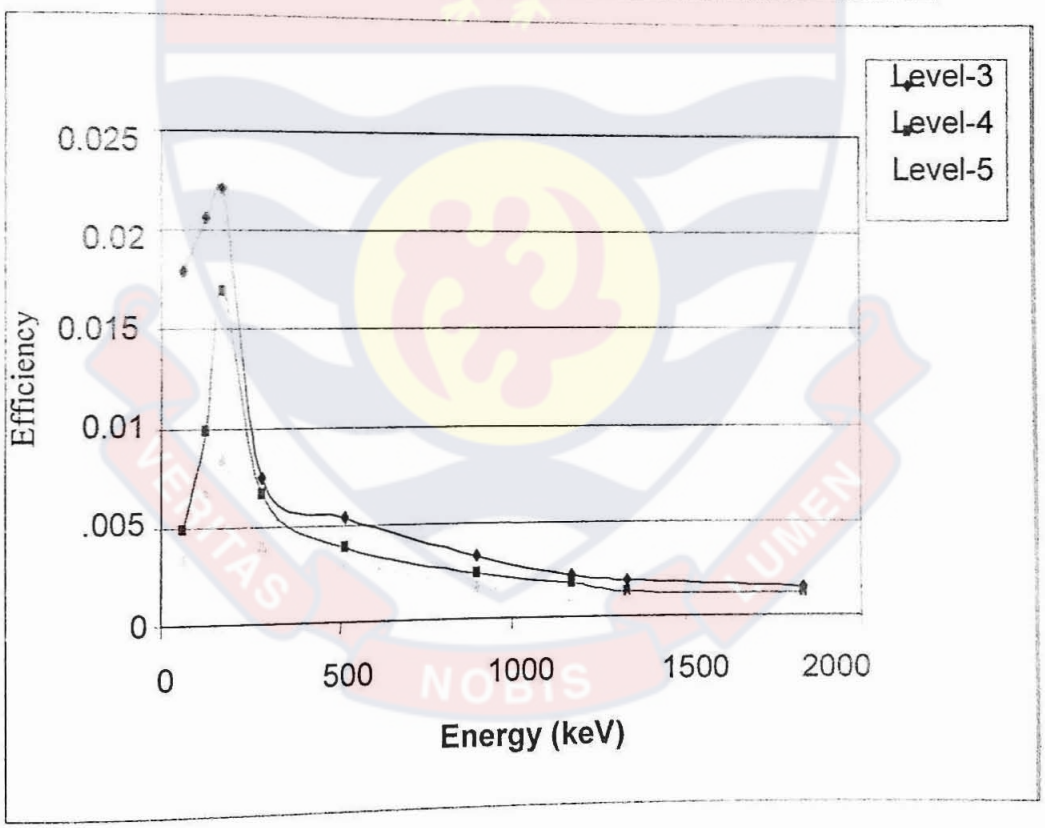

Fig.5.2. Efficiency curves at 1.0, 1.5 and 2.6 cm from the surface of the HPGe of the Compton suppression spectrometry in AC mode

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Determination of iodine in foods using various INAA methods

A combination of different types of INAA methods have been developed for the determination of iodine in various food items from Ghana. The methods involved conventional reactor flux INAA in conjunction with conventional and Compton suppression gamma-ray spectrometry using both the relative and the k_0 standardization methods. The other methods are epithermal INAA (EINAA), pseudo-cyclic INAA (PCINAA) and pseudo cyclic EINAA (PCEINAA) with conventional and Compton suppression gamma-ray spectrometry using the relative and k_0 standardization methods. The sensitivities and detection limits obtained under different experimental conditions are presented in Table 5.5. The detection limits L_D were calculated using the formula proposed by Currie, 1968.

$$L_D = 2.71 + 3.29\sqrt{\mu_B}$$

Where μ_B is the background counts. The detection limits varied from sample to sample depending on the background activity.

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Table 5.5: Sensitivities and detection limits of iodine under different experimental conditions

Scheme t_i-t_d-t_c	Method of Analysis	Sensitivity (counts/µg)	Detection Limits (µgkg^{-1})
5-5-20	INAA-CONV.	1595	200-500
5-5-20	INAA-AC	1045	120-300
30-5-20	EINAA-CONV.	2810	50-250
30-5-20	EINAA-AC	1440	25-180
30-5-20	PCEINAA-CONV.(N=4)	10970	20-140
30-5-20	PCEINAA-AC (N=1)	1440	25-180
30-5-20	PCEINAA-AC (N=2)	2590	15-50
30-5-20	PCEINAA-AC (N=3)	4380	5-10
30-5-20	PCEINAA-AC (N=4)	6130	1-5
30-5-20	PCEINAA-AC (N=5)	8081	0.5-4

The lowest detection limits were obtained using PCEINAA with anticoincidence counting up to a maximum of 5 cycles. Due to high dead-times, after 5 cycles of irradiation, the background activities became undesirably high and led to insignificant reduction in detection limits. The use of the same sample material in different capsules allows the accumulation of spectra for a number of samples without the increased in background activities and therefore yielded lower detection limits than repetitive irradiations of the same sample material. This method eliminated high dead-time effects. The detection limits for 5 cycles were about three times lower than one-short irradiation in all cases due to significant improvement in sensitivity of the elements. The PCEINAA with

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anticoincidence counting method was found to be the best method for determination of low levels of iodine. The optimum delay time between the end of one cycle and the start of irradiation of the next cycle was 40 min.

Detection limits of the order of 90-350 μgkg^{-1}, 21-130 μgkg^{-1} and 11-70 μgkg^{-1} in biological materials have been reported by Suilivan, 1998; Zhang, 1997 and Yonezawa *et al.*, 2003 respectively using PCINAA-AC and EINAA-AC. Kucera *et al.*, 2001 used RNAA and obtained detection limit of 1.0 μgkg^{-1} in biological sample. In this work, the lowest detection limit of 0.5 μgkg^{-1} was obtained using PCEINAA-AC with 5 cycles of irradiation-delay-counting. This is an improvement of about 10 orders of magnitude over reported values using INAA and 2 orders of magnitude better than RNAA. The spectra for NIST 8415 Whole Egg Powder for one-short EINAA and 5 cycles PCEINAA using anticoincidence counting is shown in Fig.5.3. As seen from the figure, the background activities increase as the number of cycles increased but the increased in sensitivities far exceeded the corresponding increase in the background leading to lower detection limits.

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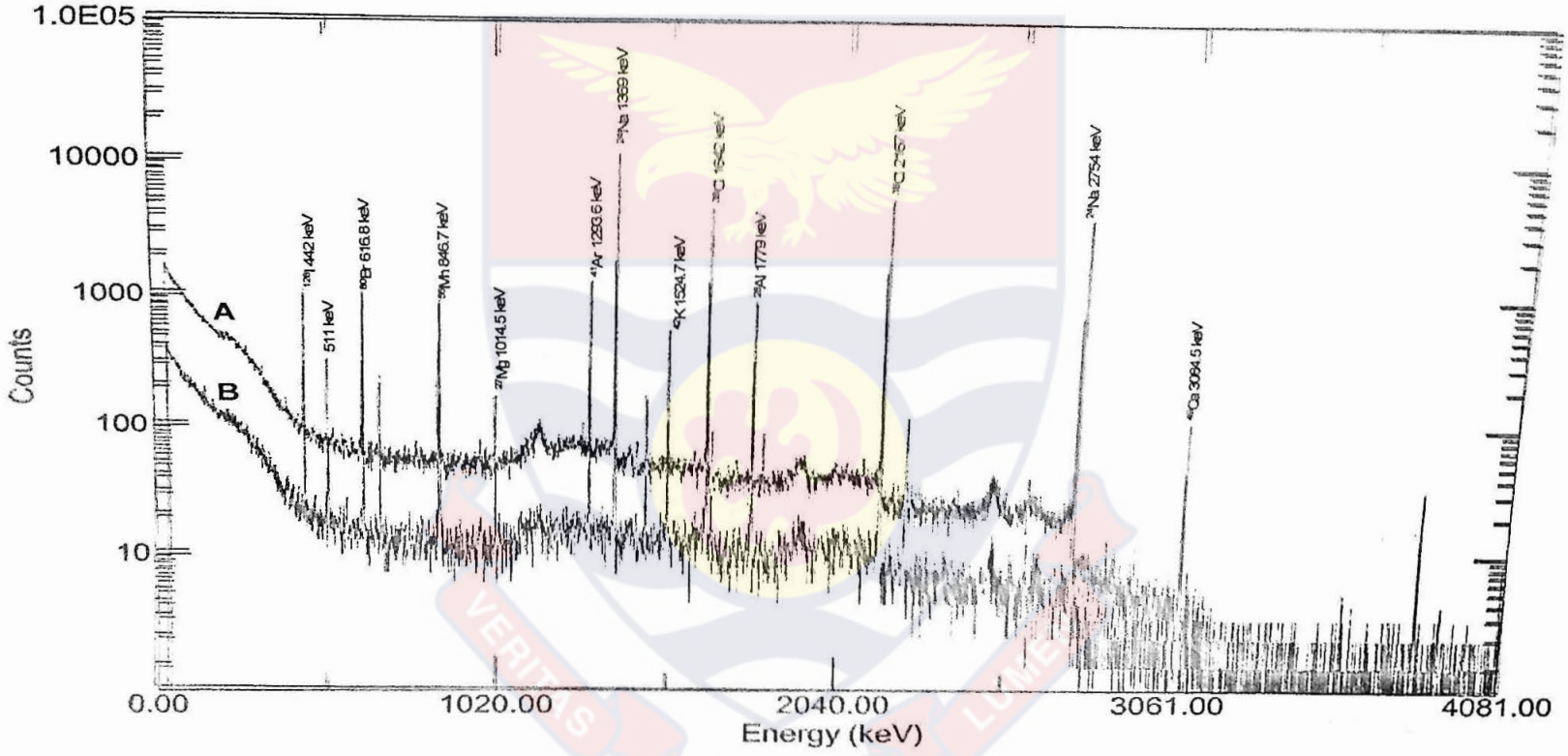

Fig. 5.3: Spectra of NIST SRM whole egg powder using Compton suppression gamma-ray spectrometry (t_i=30 min, t_d=5 min, t_c=20 min), A=One shot EINAA, B=Five cycles PCEINAA

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The uncertainty associated with the determination of iodine using both the relative method and the k_0 standardization methods have been evaluated and discussed under uncertainty calculations. The trueness of the method was checked by analyzing 5 National Institute of Standards and Technology SRMs The results shown in Table 5.6 are in good agreement with the certified values.

Table 5.6: Iodine concentration in SRMs by PCEINAA-AC

Certified Reference Material	This Work (μgkg^{-1})	Certified Value (μgkg^{-1})
NIST 8415 Whole Egg Powder	1850 ± 220	1970 ± 460
NIST 1549 Non-Fat Milk Powder	3245 ± 40	3380 ± 20
NIST 8418 Wheat Gluten	62 ± 15	60 ± 13
NIST 1547 Peach Leaves	270 ±20	(300)
NIST 1515 Apple leaves	330 ± 20	(300)

The iodine content of 79 Ghanaian food items using the methodologies described above are presented in Table 5.7. As expected, iodine levels in seafoods were higher than in all other food items. The levels of iodine in Ghanaian foods were also comparable to that in similar food items determined elsewhere (Fisher and Carr, 1974; Dodd and Digbe, 1993). The lowest concentration of iodine occurred in stable foodstuffs, cereals and meat.

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Table 5.7: Iodine levels in Ghanaian food items using different INAA methods and Compton Suppression spectrometry

Local or English Name	Iodine concentration (μgkg^{-1}) dry weight			
	Range	Mean	Uncertainty	Elsewhere*
Cereals and grain products				
Sorghum	<1.0-87	33.7	1.7	
Maize	5.5-105	41.4	2.2	43
Yellow maize	<1.0-56	8.1	0.4	
Millet	7.5-88	38.6	1.8	
Flour	15-135	55.3	2.9	
Wheat	35-180	73.2	3.4	44
Rice	8.7-80	31.4	1.5	39
Bread	55-670	241	15.3	
Mean		65.3		65
Legumes and nuts				
Dawadawa (Local name)	<1.0-77	35.6	4.3	
Groundnut	26-134	66.5	7.8	
Beans	85-450	233	58	245
Bambara beans	25-130	76.5	3.2	
Soya beans	3.7-86	48.6	2.5	
Agushie (Local name)	<1.0-89	58.4	2.6	
Prekese (Local name)	10-99	66.0	5.9	
Cola nuts	<1.0-86	45.3	2.0	
Tiger nuts	55-195	120.5	7.6	
Coconut (fresh)	45-205	127.5	5.7	
Mean		87.8		

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Table 5.7: Continued

Local or English Name	Iodine concentration (μgkg^{-1}) dry weight			
	Range	Mean	Uncertainty	Elsewhere*
Vegetables				
Cabbage	10-89	34.6	1.9	260
Okra	35-100	68.3	4.4	
Carrots	65-255	165	18	202
Cassava leaves	250-2450	1720	150	
Cocoyam leaves	75-360	240	15	
Garden Egg	54-186	122	9.4	
Tomatoes	87-280	167	11.6	196
Spring Onion	185-650	314	22	
Green Pepper	35-200	89.2	4.2	
Onion	38.4-270	185	9.5	204
Pepper	34.0-79.6	55.4	3.6	
Mushroom	95-450	290	2.1	
Mean		288		385
Meat, eggs and game				
Beef	95-330	281	95	
Mutton	10-120	82.7	10.4	
Eggs	1600 - 2100	1890	120	
Snail	<1.0-15	5.53	1.2	
Grasscutter	<1.0-23	11.3	1.7	
Goat Meat	33-174	82.5	3.7	
Guinea fowl	<1.0-87	46.6	2.8	
Chicken	<1.0-19	5.1	0.15	

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Table 5.7: Continued

Local or English Name		Iodine concentration (μgkg^{-1}) dry weight			
		Range	Mean	Uncertainty	Elsewhere*
Mean			300		
Marine and River Food					
Marine Fishes and Food)					
(i)	Oyster	1025-2740	1680	100	4712
(ii)	Shrimp	1640-4780	2460	170	4987
(iii)	Salmon	950-1880	1270	80	1030
(iv)	Herrings	650-1970	1120	70	1358
(v)	Tuna	880-1980	1280	90	
(vi)	Crab	1150-2540	1610	110	1292
(vii)	Kpana (Local name)	1470-2900	1995	150	
(viii)	Keta school boys (Local name)	1760-3220	2370	165	
Mean			1723		2676
Fresh River and Lake Food					
(i)	Tilapia	700 - 2080	1850	120	
(ii)	Mud Fish	32-155	87.5	3.7	
(iii)	One Man Thousand (Local name)	1000-1700	1245	103	
(iv)	Tupei (Local name)	210-1050	770	55	
(v)	Crab	900-1800	1230	95	
(vi)	Fresh water fishes	120-250	135	8.7	
(vii)	Bovi Lolo (Local Name)	1150-2890	1900	126	
Mean			1030		116

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Table 5.7: Continued

Local or English Name		Iodine concentration (μgkg^{-1}) dry weight			
		Range	Mean	Uncertainty	Elsewhere*
Milk and Milk product					
Fresh Milk		1750-3870	3418	750	
Evaporated					
(i)	Peak milk	350-1040	750	55	
(ii)	Ideal milk	345-1100	745	66	
Mean			748		
Powdered					
(i)	Peak milk	2000-3000	2350	150	
(ii)	Carnation Tea	455-1850	670	40	
	Creamer	650-2000	885	50	
(iii)	Cowbell Milk				
Mean			1300		
Milk Products					
(i)	Fan ice chocolate	550-1860	940	56	
(ii)	Fan ice vanila	870-2120	1360	175	
(iii)	Fanyogo	250-870	450	35	
(iv)	Fanchoco	346-1150	620	50	
(v)	Fan ice strawberry	765-1950	1035	125	
(vi)	Fangold ran raison	242-670	355	25	
(vii)	Fan ice sachet	264-765	395	28	
(viii)	Fanpop	123-456	250	18	
Mean			676		

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Table 5.7: Continued

Local or English Name	Iodine concentration (μgkg^{-1}) dry weight			
	Range	Mean	Uncertainty	Elsewhere*
Staple Foodstuff				
Plantain	<1.0-66	27.5	1.2	
Cassava	24-120	75.5	4.7	
Cocoyam	<1.0-98	55.0	3.5	
Yam	45-200	110	7.5	
Mean		67.0		
Beverages				
Cocoa	6.5-120	67.6	3.1	
Nescafe	950-1850	1050	75	
Tea (Lipton)	35-120	87.4	3.5	
Mean		402		
Fruits				
Banana	<1.0-85	33.7	1.7	
Pineapple	61-156	85.6	4.6	
Avocado Pear	65-340	195	13.8	62
Mean		105		159

* Fisher and Carr, 1974

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Uncertainty components associated with iodine determination in NIST 8415 Whole Egg Powder using the relative and the k_0 - standardization methods

The overall uncertainty associated with iodine determination in NIST 8415 Whole Egg Powder using the relative and the k_0 INAA standardization methods are presented in Table 5.8. The uncertainties were evaluated using the equations and conditions described under uncertainty calculations in Chapter 3. It is clear from the estimation that the overall uncertainty associated with determination of iodine using the k_0 method was higher by a factor of about 2.5% than the relative method due to uncertainties in the nuclear constants and detector full-energy photopeak efficiency determination in the k_0 quantification method which are absent in the relative method.

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Uncertainty components associated with iodine determination in NIST 8415 Whole Egg Powder using the relative and the k_0 - standardization methods

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Table 5.8: Uncertainty components associated with the determination of iodine in NIST 8415 Whole Egg Powder using the relative method

Uncertainty source	Symbol for variable, unit	Value of variable	Uncertainty	Conversion factor to standard uncertainty	Relative standard uncertainty	% Contribution to combined uncertainty
Mass of sample	W, mg	520.6	0.007	1	$1.34x10^{-5}$	*0.025*
Mass of I standard	w, mg	100	0.10	1	$5.78x10^{-5}$	*0.110*
Purity of standard	%	99.99	0.01	$1/\sqrt{3}$	$5.78x10^{-6}$	*0.011*
Isotopic abundance	Θ	Negligible	Negligible	-	Negligible	-
Irradiation geometry difference	Gi, cm	Negligible	Negligible	-	Negligible	-
Neutron self-shielding	N_s	99.75-100.05	0.30	1	$3.0x10^{-4}$	*0.574*
Timing	t, s	Negligible	Negligible	-	Negligible	-
Irradiation interference	I_T	Negligible	Negligible	-	Negligible	-
Counting statistics (sample)	$N_{p\,(sam)}$ Counts	1965	56	1	0.028	53.53
Counting statistics (standard)	$N_{p(std)}$	6650	52	1	$7.83x10^{-3}$	14.9

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Table 5.8: Continue

Uncertainty source	Symbol for variable, unit	Value of variable	Uncertainty	Conversion factor to standard uncertainty	Relative standard uncertainty	% Contribution to combined uncertainty
Counting geometry difference	G, cm	Negligible	Negligible	-	Negligible	-
Pulse pileup losses	P_p	Negligible	Negligible	-	Negligible	-
Dead-time effects	t_{dt}, s	Negligible	Negligible	-	Negligible	-
Decay-timing effects	t_d,s	Negligible	Negligible	-	Negligible	-
Gamma-ray self-shielding	Γ_s	Negligible	Negligible	-	Negligible	-
Gamma-ray interference	Γ_i	Negligible	Negligible	-	Negligible	-
Peak integration method	P_i	99.98-100.02	0.04	$1/\sqrt{6}$	0.016	30.6
Overall	-	-	-	-	0.0523	100

Value of Measurand $\rho_{cm} = 1.97 \pm 0.46$

Combined standard uncertainty $u_c(cl) = 0.11$, Expanded uncertainty = 1.85 ± 0.22 (Coverage factor $\kappa = 2$)

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Table 5.9: Uncertainty components associated with the determination of iodine in NIST 8415 Whole Egg Powder using the k_0 method

Uncertainty source	Symbol for variable, unit	Value of variable	Uncertainty	Conversion factor to standard uncertainty	Relative standard uncertainty	% Contribution to combined uncertainty
Mass of sample	W, mg	520.6	0.007	1	1.34×10^{-5}	0.006
Mass of Au standard	w, mg	0.200	0.01	1	5.78×10^{-5}	0.026
Purity of standard	%	99.999	0.001	$1/\sqrt{3}$	5.78×10^{-6}	0.0026
Isotopic abundance	Θ	100	Negligible	-	Negligible	Negligible
Irradiation geometry difference	Gi, cm	Negligible	Negligible	-	Negligible	Negligible
Neutron self-shielding	N_s	Negligible	Negligible	-	Negligible	Negligible
Timing	t, s	Negligible	Negligible	-	Negligible	Negligible
Irradiation interference	I_T	Negligible	Negligible	-	Negligible	Negligible
Counting statistics (sample)	$N_{p(sam)}$	97880	920	1	0.28	12.6
Counting statistics (standard)	$N_{p(std)}$	568580	1050	1	0.0059	2.67

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Table 5.9: Continued

Uncertainty source	Symbol for variable, unit	Value of variable	Uncertainty	Conversion factor to standard uncertainty	Relative standard uncertainty	% Contribution to combined uncertainty
Counting geometry difference	G, cm	Negligible	Negligible	-	Negligible	Negligible
Pulse pileup losses	P_p	Negligible	Negligible	-	Negligible	Negligible
Dead-time effects	t_{dt}, s	Negligible	Negligible	-	Negligible	Negligible
Decay-timing effects	t_d, s	Negligible	Negligible	-	Negligible	Negligible
Gamma-ray self-shielding	Γ_s	Negligible	Negligible	-	Negligible	Negligible
Gamma-ray interference	Γ_i	Negligible	Negligible	-	Negligible	Negligible
Peak integration method	P_i	99.98-100.02	0.04	$1/\sqrt{6}$	0.016	7.20
Resonance energy (iodine)	E_{rI}	57.6	0.40	1	6.94×10^{-3}	3.13
Resonance energy (gold)	E_{rAu}	5.65	0.03	1	5.31×10^{-3}	2.39
Resonance integral (gold)	Q_{0Au}	15.7	0.12	$1\sqrt{3}$	4.41×10^{-3}	1.99
Resonance integral (iodine)	Q_{0I}	24.8	0.21	$1/\sqrt{3}$	4.89×10^{-3}	2.20

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Table 5.9: Continued

Uncertainty source	Symbol for variable, unit	Value of variable	Uncertainty	Conversion factor to standard uncertainty	Relative standard uncertainty	% Contribution to combined uncertaint
k_0 factor	k_0	0.0112	$1{,}4\times10^{-4}$	$1/\sqrt{6}$	7.78×10^{-5}	0.035
Coincidence correction factors	COI	Negligible	Negligible	-	Negligible	Negligible
Au-wire standard	Au, mg	12.5	0.125	$1/\sqrt{3}$	0.0722	34.8
Thermal-to-epithermal neutron flux ratio	f	18.9	0.6	$1/\sqrt{6}$	0.0122	5.50
Epithermal neutron flux shape factor	α	0.0422	0.0051	$1/\sqrt{6}$	0.049	22.1
Efficiency (iodine)	E_I	0.01279	0.0031	$1/\sqrt{6}$	6.38×10^{-3}	2.87
Efficiency (gold)	E_{Au}	0.01367	0.0029	$1/\sqrt{6}$	5.97×10^{-3}	2.69
Overall	-	-	-	-	0.222	100

Value of Measurand $\rho_{cm} = 1.97 \pm 0.46$

Combined standard uncertainty $u_c(cI) = 0.24$, Expanded uncertainty $= 1.81 \pm 0.48$ (Coverage factor $\kappa = 2$)

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Determination of short-lived nuclides using PCINAA and Compton suppression gamma-ray spectrometry

A PCINAA method was developed for the simultaneous determination of Dy, Hf, Rb, Sc and Se in some Ghanaian foods through their short-lived nuclides. The precision and detection limits of these trace elements were significantly improved by increasing the number of cycles up to 4. However, as the long-lived nuclides such as ^{38}Cl and ^{24}Na built up and created significant undesirable Compton background activities, the number of cycles a sample could be irradiated becames limited. If a long delay period, *e.g.* several days, is allowed between the repetitions of cycles, the sensitivity of measurement can be further improved. Obviously, the total analysis time could then become undesirably long and if the element to be determined has long-lived nuclide, then this method is unnecessary. Alternatively, a Compton suppression counting coupled to PCINAA was used to achieve similar or better results within a shorter analysis time. In order to minimize errors caused by possibly high dead-time, the optimum distance between the detector and the sample was set at 1.0 cm to maintain dead-times less than 10%. The delay between the end of one cycle and the start of irradiation of the next cycle was 50 s.

The detection limit for each element varied from sample to sample depending on the background activity. The detection limits were also improved in the PCINAA up to 4 cycles. There were no significant reductions in detection limits for elements having gamma-ray energies less than 150 keV using the anticoincidence and conventional counting due to insignificant reduction in the Compton continuum. The range of the detection limits for each element is presented in Table 5.9.

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The precision and trueness of the method were checked by analyzing two NIST SRMs under the same experimental conditions as the samples. The results obtained in this work are comparable to the reported values as shown in Table 5.10.

Precision of the counting system was calculated as a percentage relative standard deviation (%RSD) of 6 or more replicate measurements of each sample and was found to be less than ±10%. At least 6 portions of selected Ghanaian cereals and vegetables were analyzed for Dy, Hf, Rb, Sc, and Se through their short-lived nuclides by PCINAA method and anticoincidence counting. Their average concentrations on dry-weight basis are presented in Table 5.11.

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Table 5.10: Range of detection limits for short-lived nuclides of various samples analyzed by PCINAA using both conventional and anticoincidence counting

Nuclide used	Gamma-ray energy used (keV)	Half-life (s)	Detection limits mg kg^{-1}			
			Anticoincidence		conventional	
			One-short irradiation	Four Cycles	One-short irradiation	Four Cycles
^{165}Dy	108.2	75.4	0.001-0.05	0.003-0.010	0.002-0.22	0.003-0.010
^{179}Hf	214.5	18.68	0.001-0.06	0.002-0.011	0.008-0.60	0.01-0.12
^{86m}Rb	555.4	61.0	1.5-2.0	1.0-1.5	2.5-10.0	5.0-15.0
^{46m}Sc	142.5	18.75	0.001-0.03	0.002-0.05	0.001-0.04	0.002-0.05
^{77m}Se	161.9	17.45	0.001-0.01	0.005-0.05	0.002-0.02	0.0058-0.06

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Table 5.11: Analysis of NIST 1547 Peach Leaves and NIST 1566b Oyster Tissue by PCINAA and Anticoincidence counting

Element	NIST 1547 Peach leaves (μgkg^{-1})		NIST 1566b Oyster Tissue (μgkg^{-1})	
	This work	Reported Values	This work	Reported values
Dy	650 ± 12	690 ± 20	42.2 ± 5.7	-
Hf	55.4 ± 3.1	50.0 ± 3.2	16.3 ± 1.1	-
Rb	21100 ± 1600	(19700)	3530 ± 210	3260 ± 145
Sc	38.4 ± 6.2	(40.0)	46.8 ± 5.5	-
Se	130 ± 7.3	120 ± 6.0	2020 ± 180	2060 ± 150

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Table 5.12: Concentrations of short-lived nuclides in cereals and vegetables using PCINAA and Compton suppression spectrometry (μgkg^{-1})

Element	Sorghum	Maize	Millet	Rice	Wheat	Okra	Cabbage
Dy	2.21 ± 0.22	<1.0	9.7 ± 0.8	9.81 ± 0.77	<1.0	66.5 ± 5.7	<1.0
Hf	11.5 ± 3.2	4.22 ± 0.31	3.00 ± 0.22	24.5 ± 2.12	63.6 ± 4.9	1750 ± 78	45.2 ± 3.9
Rb	6330 ± 448	3820 ± 230	6400 ± 340	13400 ± 1140	8270 ± 776	15600 ± 860	25600 ± 1200
Sc	4.17 ± 0.88	42.5 ± 2.2	11.4 ± 1.2	91.3 ± 8.6	52.1 ± 4.9	1260 ± 98	33.6 ± 2.8
Se	35.1 ± 5.6	48.7 ± 3.2	98.3 ± 6.7	75.1 ± 5.5	499 ± 58	25.3 ± 4.9	3810 ± 157

Element	Pepper	Garden eggs	Onion	Spring onion	Cocoyam leaves	carrots	Cassava leaves	Tomatoes
Dy	<1.0	<1.0	110 ± 10	<1.0	<1.0	52.4 ± 3.8	29.7 ± 1.8	<1.0
Hf	95.1 ± 7.7	51.4 ± 3.7	1150 ± 67	5.7 ± 0.31	120 ± 10	10.3 ± 0.87	10.9 ± 0.98	<1.3
Rb	79200 ± 1200	36500 ± 800	8610 ± 700	38600 ± 1200	41500 ± 2100	81200 ±1150	25400 ± 889	12700 ± 550
Sc	975 ± 88	150 ± 14	1050 ± 102	690 ± 66	110 ± 9.6	98.6 ± 7.9	182 ± 16	25.4 ± 1.7
Se	11.5 ± 1.1	15.2 ± 4.6	12.8 ± 1.1	55.2 ± 5.4	354 ± 26	45.2 ± 2.7	6340 ± 110	105 ± 9.8

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Simultaneous determination of short-to-medium lived nuclides by INAA and Compton suppression gamma-ray spectrometry

An INAA method with Compton suppression spectrometry has been used for determination of Al, Ba, Br, Ca, Cl, Co, Cu, Dy, K, Mg, Mn, Na, Rb, S, Sr, Th, Ti, U, V and Zn in Ghanaian food samples. Irradiation, decay and counting conditions were optimized for the simultaneous determination of these 20 elements with a short turn-around time. The detection limits L_D were calculated using the formula proposed by Currie, (1968). The detection limit for each element varied from sample to sample depending on the background activity as shown in Table 5.12. These limits ranged between 1.0 $ng.g^{-1}$ and 200 $mg.kg^{-1}$ for Dy and S, respectively, using the Compton suppression system in the anticoincidence mode as compared to 1.0 $mg.kg^{-1}$ and 400 $mg.kg^{-1}$ for the same elements using the conventional counting mode. The results of analysis of 5 SRMs for the validation of the analytical method are shown in Table 5.13. The results are in good agreement with the reported values.

The average concentrations of 6 portions of some Ghanaian food items on dry-weight basis are presented in Table 5.14. It is observed from the results that, there is a great variation in the concentration of the elements from sample to sample. Of the elements determined, Ca, Cl, K, Mg, Na and S are considered as nutritionally essential major elements while Br is being a nutritionally essential trace element (Trace elements in human nutrition and health; Prasad, 1993). Elements regarded having essential/toxic duality are Co, Cu, Mn, Rb, Sr, Ti, V and Zn depending upon their species as well as concentrations. The nutritional importance of the elements Ba, Dy, Th and U has not been well established.

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Table 5.13: Detection limits of short-to-medium lived nuclides in foods using INAA and anticoincidence counting. (t_i= 1min t_d=2 min t_c=10 min)

Element	Product isotope	Gamma-ray energy (keV)	Detection limit range ($mgkg^{-1}$)
Al	^{28}Al	1778.9	10-15
Ba	^{138}Ba	165.85	5.0-10
Br	^{79}Br	616.3	0.05-0.01
Ca	^{48}Ca	3084.54	50-150
Cl	^{37}Cl	1642.7, 2167.7	10-22
Co	^{59}Co	58.60,1332.5	0.01-0.02
Cu	^{65}Cu	1039.2	0.5-1.0
Dy	^{164}Dy	108.2,515.5	0.001-0.002
	^{164}Dy	94.70, 361.7	0.005-0.01
K	^{41}K	1524.58	50-100
Mg	^{27}Mg	1014.43	20-50
Mn	^{55}Mn	1810.72	0.5-1.0
Na	^{23}Na	1368.6, 2754	5-10
Rb	^{85}Rb	555.4	0.5-1.0
S	^{36}S	3103.98	200-400
Sr	^{86}Sr	388.4	1.0-1.2
Ti	^{50}Ti	320.08	1-5
Th	^{232}Th	86.53, 459.3	0.10-0.12
U	^{238}U	74.66	0.008-0.010
V	^{51}V	1434.08	0.001-0.005
Zn	^{68}Zn	438.63	10-50

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Table 5.14: Analysis of SRMs for short-to-medium lived nuclides using INAA and anticoincidence counting (t_i=1 min t_d=2 min t_c=10min)

Element	NIST 1547 Peach leaves Concentration		NIST 1515 Apple Leaves		NIST 1566 Oyster Tissue Concentration		NIST 1577 Bovine Liver Concentration		NIST 1549 Non Fat Milk Power Concentration	
	This work	Certified Value	This work	Certified Value	This work	Certified Value	This work	Certified Value	This work	Certified Value
Al	252±9.2	249±7.5	285±3.5	286±8.6	210±8.3	197±6.0	<10	(3.0)	<10	(2.0)
Ba	129± 6.1	124±3.7	46±1.7	49±2.0	8.15±0.5	8.6±0.3	<10	-	<10	-
Br	11.7±0.6	(11)	1.9±0.03	(1.8)	54.0±3.4	-	9.4±0.86	(9.7)	11.5±0.7	(12)
Ca(%)	1.53±0.05	(1.56)	1.57±0.11	(1.526)	0.086±0.003	0.0838±0.002	<0.0150	(0.0116)	1.48±0.06	1.3±0.05
Cl	371±13	360±18	555±55	579±79	5165±89	5140±100	2900±64	2780±56	10900±350	10900±200
Co(µgkg-1)	63±4.3	(70)	86±9	(90)	357±50	371±20	220±30	(250)	<10	(4.1)
Cu	3.21±0.2	3.7±0.2	5.54±0.33	5.64±0.23	72.2±2.1	71.6±1.6	165±7.8	160±8.0	0.66±0.04	0.7±0.1
Dy(µgkg-1)	600±12	690±20	2210±103	2320±140	42.0±2.5	-	2800±100	2100±100	<2.0	-

*All values are in $mg.kg^{-1}$ unless otherwise stated

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Table 5.14: Continued

Element	NIST 1547 Peach leaves Concentration		NIST 1515 Apple Leaves		NIST 1566 Oyster Tissue Concentration		NIST 1577 Bovine Liver Concentration		NIST 1549 Non Fat Milk Power Concentration	
	This work	Certified Value	This work	Certified Value	This work	Certified Value	This work	Certified Value	This work	Certified Value
K(%)	2.59±0.11	2.43±0.00243	1.59±0.021	(1.61)	0.64±0.01	0.652±0.14	0.96±0.015	(0.994)	1.87.±0.36	1.69±0.03
Mg	4800±200	4320±86	2780±100	2710±81	1190±55	1085±23	583±48	601± 30	1300± 66	1200± 30
Mn	107±9.5	98± 2.9	60 ± 2.6	54 ± 3.2	20.3 ± 1.2	18.5	9.7±0.77	10.5 ± 1.7	<0.5	0.26±0.06
Na	22.5±1.2	24± 1.9	23.6 ± 1.4	24.4 ± 1.5	2900±44	3297±53	2300±38	2420±48	4970 ± 110	4970±100
Rb	21.1±1.6	(19.7)	10.5 ± 1.1	10.2 ± 1.5	3.53 ±0.02	3.26±0.145	13.3±1.4	13.7 ± 1.1	12.3 ± 1.1	(11)
S	2100±300	(2000)	1950±240	(1800)	7040±89	(6887)	7160±100	7850±79	3400 ± 46	3510±50
Sr	51.8 ±3.9	53±4.2	26.7 ±2.7	25 ± 2.0	6.52±0.32	6.8 ± 0.2	<1.0	0.136±0.02	<1.0	-
Ti	52.4 ±4.8	-	15.0 ±1.9	-	11.3 ±1.0	-	<5	-	<5	-

*All values are in $mg.kg^{-1}$ unless otherwise stated

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Table 5.14: Continued

Element	NIST 1547 Peach leaves Concentration		NIST 1515 Apple Leaves		NIST 1566 Oyster Tissue Concentration		NIST 1577 Bovine Liver Concentration		NIST 1549 Non Fat Milk Power Concentration	
	This work	Certified Value	This work	Certified Value	This work	Certified Value	This work	Certified Value	This work	Certified Value
Th(μgkg^{-1})	<100	(50)	<100	(30)	<100	36.7±4.0	<100	-	<100	-
U(μgkg^{-1})	20.2±3.1	(15)	<8	(6)	294±20	.255±14	<8	-	<10	-
V	0.37±0.02	0.37±0.03	0.27 ±0.02	0.26±0.03	0.600±0.04	0.577±0.03	0.129±0.01	(0.123)	<0.005	-
Zn	21.9±0.56	17.9± 0.36	<20	12.5 ± 0.25	1225±78	1424±46	121±15.9	127±16.5	<50	46.1±2.2

*All values are in $mg.kg^{-1}$ unless otherwise stated

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Table 5.15: Concentration of short-to-medium lived nuclides in Ghanaian foods using INAA and Compton suppression gamma-ray spectrometry (t_i=1 min, t_d=2 min, t_c=10 min)

Elements	Ideal Milk	Fanice chocolate	Fanice Vanilla	Fanyogo	Fanchoco	Fanice strawberry	Fangold Ran Raisin	Fan Ice Sachet	Fanpop
Al	<10	<10	<10	<10	<10	<10	<10	<10	<10
Ba	<5.0	7.89±0.2	<5.0	<5.0	6.70±0.35	<5.0	2.17±0.18	4.89±0.28	<5.0
Br	35.0±0.21	5.29±0.15	7.37±0.45	3.92±0.35	3.73±0.22	4.51±0.29	10.0±0.44	3.40±0.17	1.37±0.09
Ca (%)	0.690±0.02	0.340±0.038	0.15±0.01	0.34±0.022	6.95±0.33	0.348±0.017	0.324±0.018	2440±110	220±25
Cl	13680±2500	3400±170	6230±350	3100±120	3180±190	3880±185	3000±200	4670±300	867±40
Co(μgkg^{-1})	<10	0.340±0.015	0.162±0.01	<10	0.067±0.002	<10	<10	<10	<10
Cu	<0.5	1.67±0.16	<0.5	0.59±0.001	4.62±0.27	<0.5	1.15±0.07	0.923±0.07	<0.5
Dy(μgkg^{-1})	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	2.8±0.1	<1.0
K(%)	0.808±0.060	0.55±0.042	0.128±0.015	0.276±0.016	0.0556±0.05	0.567±0.25	0.40±0.020	0.423±0.022	0.077±0.005

*All values are in $mg.kg^{-1}$ unless otherwise stated

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Table 5.15: Continued

Elements	Ideal Milk	Fanice chocolate	Fanice Vanilla	Fanyogo	Fanchoco	Fanice strawberry	Fangold Ran Raisin	Fan Ice Sachet	Fanpop
Mg	542±75	640±42	421±22	306±10	950±55	500±20	285±10	230±9	103±5
Mn	<0.5	6.68±0.22	0.554±0.025	<0.5	6.02±0.25	<0.5	<0.5	<0.5	<0.5
Na	4030±40	1530±85	2760±110	2800±200	1570±80	1830±100	1050±85	1170±90	1500±100
Rb	14.17±1.26	15.30±1.18	14.65±1.31	12.66±0.94	15.63±1.42	13.76±0.75	12.84±0.85	15.80±1.38	<5.0
S	<200	1680±120	2000±160	1000±76	3550±200	1800±140	1600±90	<200	<200
Sr	<1.0	<1.0	<1.0	7.75±0.43	8.85±0.55	<1.0	<1.0	<1.0	<1.0
Ti	<1.0	1.87±0.15	<2	1.41±1.010	<1.0	<1.0	<1.0	<1.0	<1.0
Th(μgkg^{-1})	<100	<100	<100	<100	<100	<100	<100	<100	<100
U(μgkg^{-1})	<10	<8	<8	<8	<8	<8	<8	<8	<8
V	0.0130±0.02	0.015±0.007	0.014±0.005	0.051±0.001	0.054±0.001	0.014±0.005	0.005±0.001	<0.001	<0.001
Zn	31±1.7	30.0±2.1	32.5± 2.2	41.0± 3.2	25.2± 1.8	34.7± 3.1	28.0±2.2	36.5±2.6	<10

*All values are in $mg.kg^{-1}$ unless otherwise stated

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Table 5.15: Continued

Element	Fresh Milk	Cocoa	Yam	Plantain	Cocoyam	Cassava	Cassava Leaves	Cocoyam Leaves	Goat Meat
Al	33.1±2.3	<10	14.5	<10	170±42	<10	340±20	66.9±5.8	35.6±3.4
Ba	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
Br	28.1±2.1	<0.5	2.89±0.15	1.07±0.45	5.2±0.35	13.59±1.22	22.9±2.17	11.6±1.09	40.5±0.17
Ca (%)	0.842±0.06	0.018±0.001	<100	<100	0.34±0.022	0.037±0.002	0.783±0.022	0.491±0.025	<100
Cl	17800±950	<20	3400±170	3280±350	1670±120	1080±190	565±44	2690±640	6050±300
Co(μgkg^{-1})	<10	<10	<10	<10	<10	<10	<10	<10	<10
Cu	<0.5	2.94±0.18	3.67±0.16	1.03±0.02	12.0±1.0	4.62±0.27	7.14±0.33	11.3±0.96	<0.5
Dy(μgkg^{-1})	27±1.5	<0.5	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
K(%)	1.14±0.11	0.115±0.06	1.09±0.42	0.828±0.068	1.7±0.16	0.171±0.023	0.1.51±0.32	1.97±0.011	1.25±0.21
Mg	1040±75	615±45	443±42	1075±212	976±88	570±55	3500±200	1960±650	920±49
Mn	<0.5	2,60±0.14	6.68±0.22	2.35±0.025	2.1±0.42	<	47.5±0.56	23.6±1.9	<0.5

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Table 5.15: Continued

Element	Fresh Milk	Cocoa	Yam	Plantain	Cocoyam	Cassava	Cassava Leaves	Cocoyam Leaves	Goat Meat
Na	3800±40	11.0± 0.72	15.3±0.85	27.6±1.10	110±20	159.5±12.80	1170±90	17.0±1.3	2980±790
Rb	4.17±0.26	<5.0	5.30±0.18	4.65±0.31	2.66±0.14	5.63±0.42	5.80±0.38	<5.0	5.80±0.38
S	560±150	<200	1680±120	<200	1000±76	<200	2300±440	<200	<200
Sr	<1.0	<1.0	<1.0	<1.0	7.75±0.43	<1.0	<1.0	<1.0	<1.0
Ti	38.8±2.6	<1.0	<1.0	<1.0	3.41±0.10	<1.0	1.78±0.12	<1.0	<1.0
Th(μgkg^{-1})	<100	<100	<100	<100	<100	<100	<100	<100	<100
U(μgkg^{-1})	<5.0	<5.0	<8	<8	<8	<8	<8	<8	<8
V	0.0100±0.002	0.0178±0.003	<0.005	<0.005	0.031±0.001	<0.005	0.65±0.034	0.128±0.02	<0.001
Zn	35.2±17	<20	30.0±2.1	<20	41.0± 3.2	<20	41.2± 2.2	36.1± 2.5	20.4± 1.2

*All values are in $mg.kg^{-1}$ unless otherwise stated

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Table 5.15: Continued

Element	Crab	Shrimp	Oyster	Tuna	Tilapia	Salmon	Beef	Mutton	Chicken
Al	298±40	188±16	560±30	20.0±2.2	15.9±1.3	210±15	<10	59.2±3.5	<10
Ba	<0.5	495±28	30.8±2.5	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0
Br	135±66	135±8.3	9.32±0.21	56.0±3.45	77.3±3.35	45.0±3.2	8.51±0.69	35.8±2.44	3.50±0.22
Ca (%)	5.41±0.25	7.23±0.28	0.275±0.02	0.218±0.01	<100	0.075±0.33	<100	<100	<10
Cl	30700±1290	6080±290	620±25	17900±4350	13670±120	1650±190	6450±185	11650±9200	6830±590
Co(μgkg^{-1})	<15	<10	<10	<10	<10	<10	<10	<10	<10
Cu	10.8±0.66	73.7±4.6	10.8±0.66	6.86±0.22	<0.5	<0.5	3.41±0.27	1.15±0.07	4.02±0.27
Dy(μgkg^{-1})	27±1.5	24±1.4	27±1.5	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
K(%)	1.12±0.10	0.97±0.048	0.151±0.010	0.924±0.08	1.02±0.016	1.60±0.02	1.22±0.25	1.18±0.120	0.674±0.025
Mg	17100±775	2340±120	1200±75	1258±722	665±10	715±55	710±40	770±90	1065±55
Mn	45.9±3.2	380±12	570±20	0.55±0.025	<0.5	6.02±0.25	<0.5	<0.5	<0.5

*All values are in $mg.kg^{-1}$ unless otherwise stated

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Table 5.15 Continued

Element	Crab	Shrimp	Oyster	Tuna	Tilapia	Salmon	Beef	Mutton	Chicken
Na	15000±940	9470±200	730±40	16800±2110	4580±340	3150±780	3510±100	3660±815	3860±780
Rb	14.17±0.26	3.8± 0.22	4.17±0.26	<5.0	12.66±0.84	3.63±0.42	37.6±2.25	12.84±0.85	15.63±1.42
S	<200	4800±150	2100±150	8700±560	<400	1950±200	<200	<200	<200
Sr	12400±0.51	380±10	8.14±0.51	<1.0	<1.0	8.85±0.55	<1.0	<1.0	<1.0
Ti	7600±2.6	40.8±2.2	38.8±2.6	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Th(μgkg^{-1})	<100	<120	<100	<100	<100	<100	<100	<100	<100
U(μgkg^{-1})	<10	<10	57±4.4	<8	<8	<8	<8	<8	<8
V	0.685±0.02	0.25±0.08	0.700±0.02	0.014±0.005	<0.001	0.26±0.01	<0.001	0.164±0.015	<0.001
Zn	<20	68.0± 2.9	255±17	<20	41.0± 3.2	85.3±4.5	36.4±2.1	71.5±4.6	61.2±4.1

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Table 5.15: Continued

Element	Maize	Millet	Beans	Groundnut	Sorghum	Agushie	Werewere	Dawadawa	Preksese
Al	45.0± 3.0	76.7±7.1	2230±107	13.2±1.7	27.6±3.3	36.0±2.3	83.7±5.6	44.2±3.1	447±23
Ba	1.53± 0.11	3.09±0.32	45.5±2.6	5.35±0.42	0.83±0.03	<0.5	<0.5	4.74±0.25	42.2±2.2
Br	1.41± 0.08	1.59±0.12	0.745±0.03	0.15±0.02	0.87±0.05	0.368±0.01	0.26±0.01	2.71±0.12	1.39±0.08
Ca(%)	<0.0150	<0.0100	0.037±0.002	0.036±0.005	0.020±0.001	0.081±0.005	0.060±0.005	0.25±0.018	0.68±0.048
Cl	1000 ± 78	405±32	99.5±5.6	58.5±2.4	470±45	26.2±1.5	41.2±3.1	65.0±5.2	108±3.1
Co(μgkg^{-1})	38.6±2.9	232±40	14800±970	221±13	38±3.1	450±25	97±7	170±10	900±30
Cu	1.69 ±0.12	2.09±0.11	1.64±0.08	7.95±0.42	3.65±0.08	13.7±2.1	10.3±0.8	4.00±0.26	10.7±0.33
Dy(μgkg^{-1})	<1.0	<1.0	60±5.2	3.1±0.1	2.2±0.21	2.7±0.15	8.3±0.1	<1.0	39.2±1.9
K(%)	0.38±0.024	0.233±0.012	1.4±0.03	0.69±0.01	0.40±0.012	0.76±0.03	0.43±0.015	0.87±0.035	0.202±0.014

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Table 5.15: Continued

Element	Maize	Millet	Beans	Groundnut	Sorghum	Agushie	Werewere	Dawadawa	Preksese
Mg	1200±80	980±54	1470±40	2400±55	870±22	5200±120	3100±100	880±40	14300±720
Mn	6.94±0.45	14.4±1.1	626±15	18.0±1.2	10.9±1.0	76.3±2.9	23.3±2.1	22.0±1.7	44.7±2.1
Na	24.5±1.9	9.00±0.62	77.8±3.7	20.1±0.53	<10	11.7±0.78	11.0±0.8	52.6±3.5	106±6.1
Rb	0.72±0.03	6.40±0.34	11.5±0.71	5.51±.0.07	6.33±0.48	8.40±0.32	9.30±0.6	8.00±0.45	17.3±1.2
S	<200	<200	<300	845±28	<200	1500±60	1050±55	<250	700±40
Sr	1.40±0.25	<1.0	<1.2	4.80±0.22	<1.0	<1.0	5.73±0.18	6.57±0.42	22.3±1.3
Ti	4.19±0.76	1.85±0.07	35.2±1.2	5.03±0.27	2.34±.0.05	3.82±0.11	20.0±1.2	10.0±0.58	44.6±2.5
Th(μgkg^{-1})	<100	<100	<102	<100	220±60	930±50	720±35	630±27	465±27
U(μgkg^{-1})	9.2±0.7	<8	95±6.1	8.6±0.1	<8	15±0.1	<8	10±0.7	<8
V	0.12±0.05	0.05±0.001	13.6±0.7	0.013±.0001	0.021±.004	0.023±.003	0.128±0.07	0.036±.0017	1.03±.0.07
Zn	37.0±2.0	49.1±2.6	26±1.9	54.4±3.5	17.9±1.3	70.9±5.2	<20	<20	<20

*All values are in $mg.kg^{-1}$ unless otherwise stated

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INAA of Long lived nuclides in Ghanaian foods using Compton suppression gamma-ray spectrometry

Fourteen long lived nuclides in samples of Ghanaian foods and 4 SRMs were determined using Compton suppression gamma-ray spectrometry operating in both conventional and anticoincidence counting modes. The samples were irradiated for 3h in the inner irradiation sites of the DUSR facility at thermal neutron flux of 2.5×10^{11} $cm^{-2}s^{-1}$. The samples were allowed to decay between 3-10 days and counted for 10 h. Of the 14 elements determined using long-lived nuclides, Br, Rb, Sc, Th and Zn were also quantified using their relatively short-lived nuclides in the previous schemes. It was observed that the detection limits for these elements were significantly reduced using their long-lived nuclides due to long irradiation, decay and counting times. Even though Na and K have only one half-life each their determination by long irradiation, decay and counting yielded lower detection limits than short irradiation decay and counting conditions. The detection limits calculated using the equation proposed by Curie (1968) is shown in table 5.15. Table 5.16 shows the results of analysis of the SRMs which are in good agreement with the certified values. The anticoincidence counting worked best for the determination of As, Au, Cr, Mo, K, Rb, Sb, Th and Zn while the conventional counting was good for Br, Fe La, Na and Sc. This was due to the fact that, the full-energy photopeaks of Br, Fe, La, Na and Sc were suppressed in the anticoincidence counting because they emit coincident gamma-rays in cascade. The concentrations of these elements are given in Table 5.17. The superiority of the anticoincidence counting over the conventional counting system is illustrated in Fig. 5.4. The Compton background is drastically reduced resulting in low detection limits allowing photopeaks with relatively low activity to be determined.

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It was observed from the results that, contrary to the general view that plantain contains high levels of Fe, it was rather one of the foodstuffs with the lowest concentration of Fe. Of the foodstuffs analyzed, the highest Fe concentration was found in beans. The concentration of As was found to be higher in sea and river foods than all other foods. It was also observed that foodstuffs from the mining areas had higher levels of toxic elements like As, Cr and Sb than the same foodstuffs obtained elsewhere. The detailed discussion of the nutritional and toxicological effects of the elements determined is beyond the scope of this work.

Table 5.16 Range of detection limits of long-lived nuclides in various food items using INAA and Compton suppression spectrometry (t_i = 3 h, t_d =3-10 d, t_c =10 h)

Element	Product nuclide	γ- energy (keV)	Range of detection limit ($\mu g kg^{-1}$)
As	^{76}As	559	0.01-1.0
Au	^{198}Au	411.87	0.001-0.1
Br	^{82}Br	776. 554	1.0-5.0
Cr	^{51}Cr	320.1	20-100
Fe	^{59}Fe	1099, 1291	20-50
K	^{42}K	1524.8	1000-10000
La	^{141}La	486, 1596	10-100
Mo	^{98}Mo	140.2	100-500
Na	^{24}Na	1369, 2754	500-1000
Rb	^{76}Rb	1076	10-50
Sb	^{121}Sb	564	0.001-0.01
Sc	^{46}Sc	889	0.001-0.01
Th	^{233}Th	312	100-500
Zn	^{65}Zn	1115.6	500-10000

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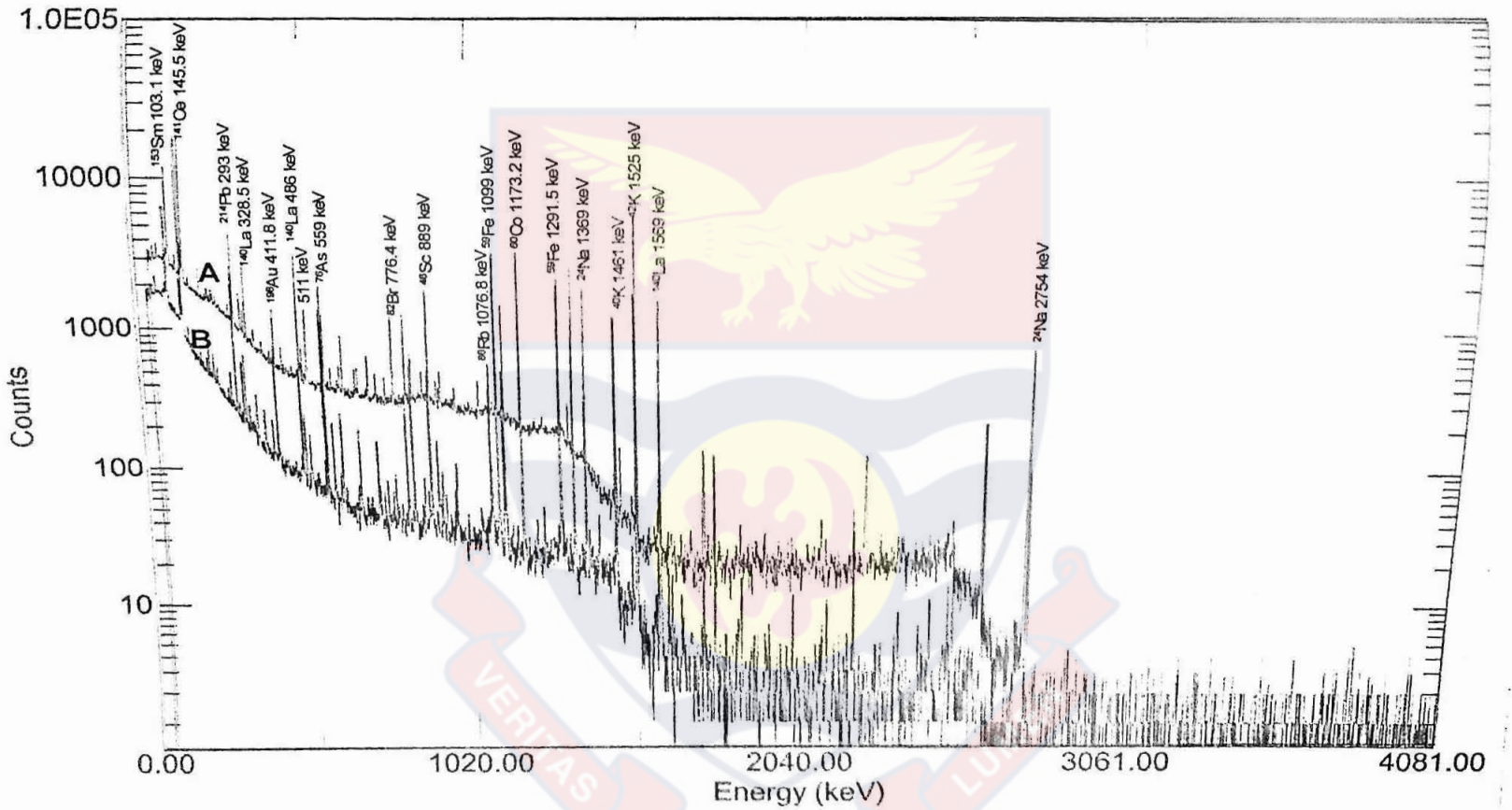

Fig.5.4. Spectra of beans using Compton suppression gamma-ray spectrometry (t_i=3 h, t_d=5 d, t_c=10 h)

A=Conventional counting. B=Anticoincidence counting

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Table 5.17: Analysis of long-lived nuclides in SRMs by INAA and Compton suppression spectrometry (t_i=3 h t_d=3-10 d t_c=10 h)

Element	NIST 1547 Peach leaves Concentration		NIST 1515 Apple Leaves Concentration		NIST 1566b Oyster Tissue Concentration		NIST 1577b Bovine Liver Concentration	
	This work	Certified Value	This work	Certified Value	This work	Certified Value	This Work	Certified Value
As(μgkg^{-1})	58.6± 9.9	60.0±18	41.3±4.5	38.0±7.1	7580±450	7605±605	54.3 ±5.3	(50)
Au(μgkg^{-1})	<0.01	-	1.12±0.09	(1.0)	<0.1	-	<0.01	-
Br	11.2± 0.6	(11)	1.9± 0.03	(1.8)	55.0± 3.4	-	9.6 ± 0.86	(9.7)
Cr	1.12±0.05	(1)	3.2± 0.11	(0.3)	<0.04	-	<0.03	-
Fe	225± 13	218± 14	85± 5.0	(83)	200± 13	205.8±6.8	190± 10	184±16
K (%)	±4.3	2.43±0.30	86±9	1.61 ± 0.016	357±50	0.652±0.009	220 ±30	0.994±0.003
La	8.7±0.2	(9)	22±2.3	(20)	<0.04	-	<0.04	-

*All values are in mg.kg^{-1} unless otherwise stated

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Table 5.17: Continued

Element	NIST 1547 Peach leaves Concentration		NIST 1515 Apple Leaves Concentration		NIST 1566b Oyster Tissue Concentration		NIST 1577b Bovine Liver Concentration	
	This work	Certified Value	This work	Certified Value	This work	Certified Value	This Work	Certified Value
Mo	0.058±0.003	0.060±0.008	0.098±0.009	0.094 ±0.013	<0.05	-	3.8±0.42	3.5±0.33
Na	25±2.5	24±2	23.5±1.6	23.4±1.2	3310±97	3297±53	2495±88	2420±48
Rb	19.5±1.1	19.7±1.2	11.2±0.44	10.2 ± 0.51	3.15±0.41	3.262±0.145	13.5±1.5	13.7±1.2
Sb(μgkg^{-1})	20.1±15	(20)	12.8±1.1	(13)	11.4±1.0	11±2	2.8±0.12	(3.0)
Sc(μgkg^{-1})	38.5±2.1	(40)	32.2 ±1.5	(30)	<0.01	-	<0.01	-
Th(μgkg^{-1})	<100	(50)	<0100	(30)	<150	36.7±4.3	<100	-
Zn	16.1±1.6	17.9±0.4	11.5 ± 0.25	10.2 ± 1.5	1430±97	1424±46	125±14	127 ± 15

*All values are in $mg.kg^{-1}$ unless otherwise stated

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Table 5.18: Concentration of long-lived nuclides in Ghanaian foods using INAA and Compton suppression spectrometry (t_i=3h, t_d=3-10d, t_c=10h)

Element	Beans	Cocoa	Maize	Cassava	Cassava Mining 1	Cassava Mining 2	Cocoyam	Cocoyam Mining Area
As (μgkg^{-1})	707±13	<0.1	10.6±0.92	<0.5	322±20	144±3.8	2.0±0.10	58.4±5.1
Au (μgkg^{-1})	10.6±0.9	<0.02	4.51±0.23	16.3±1.4	9.7±0.21	15.2±0.41	0.44±0.1	6.78±0.4
Br	0.55±0.11	<1.0	1.85±0.07	12.5±1.1	20.5±1.5	23.1±1.7	55.0±3.5	32.2±2.6
Cr	2.56±0.018	<0.02	<0.02	<0.03	9.28±0.22	12.2±0.96	<0.020	9.97±0.57
Fe	9620±750	<40	96.5±8.5	<50	<45	255±70	<50	<50
K	15070±985	1200±170	3790±250	16800±1750	8350±190	11420±185	32210±200	2250±300
La	0.78±0.03	<0.003	0.0346±0.007	0.033±0.002	<0.002	0.094±0.006	<0.002	<0.002
Mo	2.00±0.17	<0.02	0.227±0.043	<0.02	<0.02	<0.02	0.439±0.07	9.28 ±0.87
Na	78.3±2.4	12.7±1.0	30.6±2.8	133±12	107±12	80.5±9.5	21.4±.0001	255± 14

*All values are in $mg.kg^{-1}$ unless otherwise stated

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Table 5.18: Continued

Element	Beans	Cocoa	Maize	Cassava	Cassava Mining 1	Cassava Mining 2	Cocoyam	Cocoyam Mining Area
Rb	11.69±0.79	5.12±0.21	2.05±0.14	6.35±0.02	4.28±.0.22	15.84±1.2	1.75±0.08	37.8± 14
Sb (μgkg^{-1})	39.5±2.7	<0.05	4.71±0.21	6.4±0.21	96.9±3.8	451±62	0.90±0.02	45.8±3.8
Sc(μgkg^{-1})	240±15	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Th(μgkg^{-1})	200±11	<0.5	284±54	<0.2	12.9±1.1	8.3±0.1	6.38±0.17	124±10
Zn	29.5±2.7	7.22±0.22	31.7±2.5	<1.0	10.4±0.85	13.16±1.22	36.4±1.8	25.3± 1.7

*All values are in mg.kg-1 unless otherwise stated

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Table5.18: Continued

Element	Sorghum	Mushroom (Wild)	Mushroom (Cultivated)	Millet	Cocoyam Leaves	Cassava Leaves	Plantain	Yam
As(μgkg^{-1})	32.4±2.7	540±85	508±78	9.32±0.67	405±33	84.9±5.1	12.8±0.32	7.3±0.18
Au(μgkg^{-1})	<0.001	13.4±0.87	46.5±2.6	1.94±0.08	66.4±3.5	7.93±0.44	4.71±0.18	1.11±0.1
Br	8.71±0.32	2.15±0.086	35±2.1	2.00±0.17	10.5±0.83	18.8±1.3	3.6±0.21	3.15±0.7
Cr	<0.02	<0.02	6.77±0.45	<0.02	0.614±0.02	0.303±0.009	0.406±0.044	0.264±0.09
Fe	120±10	1350±380	220±134	123±9.5	<45	265±47	<45	<50
K	3900±380	41100±1170	10990±950	3200±400	17825±700	3880±185	9900±200	11170±1000
La	0.085±0.005	0.137±0.015	<0.05	<0.03	<0.02	0.490±0.042	<0.05	<0.002
Mo	0.231±0.014	1.67±0.16	<0.02	5.89±0.32	0.766±0.02	2.38±0.11	<0.2	<0.02
Na	96.5±1.7	135±48	145±13	11.8±1.4	46.3±2.5	20.4±1.5	11.2±0.99	22.6±1.88

*All values are in mg.kg-1 unless otherwise stated

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Table5.18: Continued

	Sorghum	Mushroom (Wild)	Mushroom (Cultivated)	Millet	Cocoyam Leaves	Cassava Leaves	Plantain	Yam
Rb	9.02±0.85	7.28±0.44	14.3±1.5	5.43±0.34	50.6±.3.7	12.87±1.0	22.9±1.8	11.2±.0.89
Sb(μgkg^{-1})	7.98±0.65	26.0±2.2	109±10	15.64±1.2	34.1±2.6	14.61±1.3	0.48±0.02	0.82±0.01
Sc(μgkg^{-1})	13.7±1.0	220±20	<0.02	<0.01	<0.01	20.6±1.9	<0.02	<0.001
Th(μgkg^{-1})	18.5±1.3	149±42	12.7±0.11	<0.01	<0.01	16.1±1.2	12.9±1.0	169±25
Zn	25.7±2.1	134±22	61.7±4.2	33.1±2.5	71.8±3.9	255±40	4.4±0.67	26.74±0.86

*All values are in $mg.kg^{-1}$ unless otherwise stated

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Table 5.18: Continued

Element	Chicken	Goat Meat	Beef	Mutton	Shrimps	Herrings	Tuna	Salmon
As(μgkg^{-1})	<0.01	<0.01	<0.01	<0.01	4760±120	8.15±0.51	2050±320	3380±760
Au(μgkg^{-1})	2.17±0.33	19.6±0.2	2.23±0.15	2.24±0.20	<0.01	<0.01	1.7±0.18	<0.02
Br	3.8±0.02	37.1±6.7	7.60±0.87	16.6±2.8	163±15	24.6±4.2	45±7	47.2±3.8
Cr	<0.01	<0.02	<0.02	<0.02	<0.05	<0.03	<0.03	<0.04
Fe	<50	88.6±3.8	198±15	<55	435±58	230±70	218±180	<60
K	5950±400	13537±970	13670±850	9151±410	9700±480	388±85	8680±200	1670±530
La	<0.03	<0.03	<0.04	<0.03	0.91±0.002	<0.04	<0.05	<0.04
Mo	<0.02	<0.04	<0.02	<0.02	<0.04	<0.03	<0.03	<0.03
Na	3020±100	2855±230	3620±450	3030±340	9470±600	5061±200	17600±700	2981±250

*All values are in $mg.kg^{-1}$ unless otherwise stated

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Table 5.18: Continued

Element	Chicken	Goat Meat	Beef	Mutton	Shrimps	Herrings	Tuna	Salmon
Rb	15.65±1.8	13.3±.1.1	35.3±5.6	11.4±0.91	3.55±0.23	2.19±0.77	0.854±0.02	3.59±0.27
Sb(μgkg^{-1})	8.59±0.42	10.6±0.3	15.4±0.55	7.9±0.51	<0.02	8.10±0.12	0.96±0.01	5.14±0.22
Sc(μgkg^{-1})	<0.001	<0.001	<0.001	<0.001	<0.01	<0.001	<0.01	<0.01
Th(μgkg^{-1})	<0.02	<0.02	<0.02	<0.02	<0.05	<0.03	<0.04	<0.03
Zn	61.1±3.5	212±18	399±25	132±21	63.1±0.25	88.7±5.8	25.1±2.2	87.7±5.2

*All values are in $mg.kg^{-1}$ unless otherwise stated

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Table5.18: Continued

Element	Keta School boys	Crab	Tilapia	Groundnut	Konkonte	Whole Egg	Fresh Milk	Ideal
As(μgkg^{-1})	12080±850	171±13	129±10	17.84±1.2	14.65±1.00	32.1±1.9	<0.1	<(
Au(μgkg^{-1})	<0.01	<0.01	<0.02	0.574±0.023	<0.01	<0.01	<0.01	<0.
Br	68.5±5.9	129±18	71.5±3.8	0.088±0.002	0.49±0.01	7.25±0.11	28.7±1.2	31.2±
Cr	<0.05	0.083±0.003	<0.03	<0.02	0.157±0.009	<0.02	<0.02	<0.0
Fe	348±55	368±29	<60	57±3.7	<45	123±10	36.2±2.1	<35
K	8165±300	1165±767	11100±760	7750±140	10510±970	5250±320	1050±150	8050±1.
La	<0.06	0.265±0.01	<0.02	<0.02	0.045±0002	<0.03	<0.02	<0.02
Mo	<0.3	<0.5	<0.4	0.824±0.022	<0.3	<0.2	<0.1	<0.1
Na	10270±900	36100±5000	4020±500	19.6±1.4	31.9±2.2	5600±350	1560±110	4100±250

*All values are in mg.kg^{-1} unless otherwise stated

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Table5.18: Continued

Element	Keta School boys	Crab	Tilapia	Groundnut	Konkonte	Whole Egg	Fresh Milk	Ideal Milk
Rb	2.32±0.12	16.65±1.2	12.6±1.1	6.13±0.17	10.1±0.55	11.0±0.87	14.4±1.2	18.6±1.3
Sb(μgkg^{-1})	24.4±1.5	1.2±0.009	73.0±4.4	17.0±1.2	16.2±1.3	10.1±0.66	4.46±0.21	5.84±0.22
Sc(μgkg^{-1})	81.8±5.2	28±5	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Th(μgkg^{-1})	<0.02	8.9±0.7	<0.04	<0.03	62.4±4.6	<0.03	<0.02	<0.02
Zn	87.6±4.7	<1.0	17.5±1.4	53.4±2.5	10.45±0.77	39.9±2,6	38.6±2.4	30.5±1.9

*All values are in $mg.kg^{-1}$ unless otherwise stated

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CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

Various types of INAA methods have been developed and used for the analysis of different food samples from Ghana using the Dalhousie University SLOWPOKE-2 Reactor (DUSR), in Halifax, Canada and Ghana Research Reactor-1 (GHARR-1), in Accra, Ghana. The results have shown that INAA is a useful tool for analysis of food samples. The methods developed improved the sensitivities of the elements of interest and reduced the detection limits an gave better precision and accuracy. About 31 elements were determined in various food items. The methods included pseudo-cyclic INAA in conjunction with both conventional and Compton suppression gamma-ray spectrometry for simultaneous determination of 5 short-lived nuclides with half-lives less than 80s.

The determination of iodine was carried out *via* 4 INAA methods, namely conventional flux INAA, EINAA, PCINAA and PCEINAA in both conventional and anticoincidence counting modes. The two major standardization methods, namely the relative and k_0 were critically evaluated and compared in the quantification of iodine. The lowest detection limit of 0.5 $\mu g.kg^{-1}$ as well as the highest sensitivity of iodine was obtained by the use of PCEINAA with anticoincidence counting. Since the methods used for the determination of iodine in this work does not involve chemical treatments and the use of blanks, the problem of loss of iodine and contamination were

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avoided resulting in better accuracy of the results. Iodine levels in the Ghanaian foods were found to be comparable to those determined elsewhere (Fisher and Carr, 1974). Even though the iodine content of most Ghanaian foods is comparable to that in some non-goitrous areas in the world, iodine deficiency disorders is still prevalent in Ghana. This may be attributed to the way foods are prepared in Ghana. It has been reported that between 20-70% of iodine is lost during food preparation (Dodd and Digbe, 1993). Cereals lose 30-60% iodine during boiling; frying of vegetable results in 25-52% loss; other procedures such as steaming result in a loss of 30%. In fish 20% is lost by frying or grilling and as much as 58% by boiling. The iodine loss in mixed diet ranges between 37 and 70%. The other factor for the low intake of iodine in Ghana may be due to the proportions of individual foods in the diets. Ghanaian diets usually consist of staple foodstuffs which are generally low in iodine. It is also possible that the iodine in these food items may not be bioavailable. From these results it can be said that the IDD problem in Ghana can be reduced if our diets are well structured and the use of excessive heat avoided or reduced. It is also necessary for nutritionists and scientists to study the bioavailability of iodine in Ghanaian foods and diets and to make appropriate recommendations on which food items are more suitable for reducing the IDD problem in Ghana. The results showed that foods obtained from southern Ghana had iodine content far above same foodstuffs obtained from the north. The reason being that iodine is taken up by plants from the atmosphere rather than from the soil and since the southern sector of Ghana is surrounded by the sea which is the greatest source of iodine, plants grown in this area are likely to accumulate more iodine.

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Twenty short- to medium-lived nuclides were simultaneously determined using conventional reactor neutron flux together in conjunction with anticoincidence counting. This method offered short turn-around time for the analysis of the food samples. The method allowed the determination of most elements in the $mg.kg^{-1}$ range and lowered the detection limits to $\mu g.kg^{-1}$ in some cases.

The Nisle unified formulation for characterization of neutron spectra in reactor irradiation sites was proposed over 40 years ago. Since then it has not been investigated and applied to real situations have been found to be suitable for the k_0 -NAA standardization method in this work. With this, the Nisle unified formulation can now be added to the well known Hogdhal convention and Westcott formalism for characterization of neutron spectra for the k_0 -NAA standardization method. The generalized k_0-NAA standardization method holds for all three conventions. The advantage of the Nisle unified formulation is that, the effective cross section for the calculation of the elemental concentration can be determined without the Cd ratio method.

There were no significant differences between the accuracy of results using the k_0-NAA and the relative standardizations methods. The only difference in using the two methods is in the uncertainty associated with the measured values. Since there are more parameters (*i.e.*, nuclear data) in using the k_0 method, the uncertainty is usually higher than the relative method because the nuclear data are measured with a certain degree of uncertainty.

The interpretation of the results in relation to their nutritional and toxicological significance is beyond the scope of this work. But a glance through the results reveals that the concentrations of essential macro-and

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micro-nutrients in Ghanaian staple foodstuffs are generally low compared to sea foods, cereals and vegetables. For example, the general view held in Ghana that plantain contains high concentration of Fe is incorrect since Fe content of plantain was found to be less than 50 $mg.kg^{-1}$ compared to 9,620 $mg.kg^{-1}$ in beans. The same or similar values of Fe content in plantain have been reported by Danso *et al*, (2006). Even though, Ghanaian seafoods were found to contain high levels of essential nutrients, they also contained the highest concentration of As which is toxic even at low concentrations. This may not pose any threat since it has been reported that about 70% of arsenic in fish is in the form of nontoxic arsenobetaine (Ebisuda *et al.*, 2002). The task is therefore to determine which species of As is in the foods since toxicity depends both on the concentration levels and the species present. Foods obtained from mining areas were found to contain higher amount of toxic elements such as As, Cr, Sb and V than the same food obtained from non-mining areas. Though some food samples obtained from the markets showed high levels of these elements, it may be possible that these foods were also grown or cultivated in the mining areas.

This work has demonstrated the capabilities of nuclear analytical methods in the determination of major, minor and trace elements in food at the $\mu g.kg^{-1}$ levels for most elements. Since there is no database for elemental composition of Ghanaian foods and diets, it is recommended that nutritionists; toxicologists and scientists of different background get together and carry out a more extensive research on elemental composition of what Ghanaians eat and drink. This will go a long way in helping the country to solve its nutritional and health-related problems.

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Some of the publications arising from this work and conference presentations are listed under appendix A.

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APENDIX A

PUBLICATIONS AND PRESENTATIONS

Publications

1. Simultaneous determination of short-to-medium lived nuclides in Ghanaian foods using Compton suppression gamma-ray spectrometry, Journal of Radioanalytical & Nuclear Chemistry, 270,1 (2006) 243-248

2. Neutron Activation analysis of Dy, Hf, Rb, Sc and Se in Ghanaian cereals and vegetables using Compton suppression gamma-ray spectrometry, Applied Radiation and Isotopes (2006) Accepted.

Conference Presentations

3. Simultaneous determination of major, minor and trace elements in Ghanaian foods using INAA and anticoincidence counting, Poster presentation at the 8th International Conference on Nuclear Analytical Methods in the Life Sciences, Rio de Janeiro, Brazil, 17-22 April, 2005

4. Activation analysis of Dy, Hf, Rb, Sc and Se in Ghanaian cereals and vegetables using Compton suppression gamma-ray spectrometry, Poster presentation at MARC-VII conference, Hawaii, USA, Feb. 8-12, 2006

5. Iodine, Bromine, and Strontium Content of some Staple Foodstuffs from Southern Ghana Using EINAA and Compton Suppression Gamma Ray Spectrometry, Oral presentation at the 89th Canadian chemistry Conference, May 27-31, 2006, Halifax, Nova Scotia, Canada

Manuscripts under preparation

1. EINAA and Compton suppression counting for determination of trace elements in Ghanaian sea foods (in preparation), B.J.B. Nyarko, E.H.K. Akaho, J.J. Fletcher, A. Chatt

2. The Application of Nisle unified formulation for k_0-NAA standardization Method, B.J.B. Nyarko, E.H.K. Akaho, J.J. Fletcher, A. Chatt

3. Nuclear activation analysis methods for determination of Iodine contents in foods, B.J.B. Nyarko, E.H.K. Akaho, J.J. Fletcher, A. Chatt

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4. Toxic and potentially toxic elements in cassava and cocoyam from some communities around a gold mining town in Ghana, B.J.B. Nyarko, E.H.K. Akaho, J.J. Fletcher, A. Chatt

5. INAA of long-lived nuclides in Ghanaian staple foodstuffs using conventional and Compton suppression counting, B.J.B. Nyarko, E.H.K. Akaho, J.J. Fletcher, A. Chatt

6. Trace elements in Ghanaian foods by nuclear methods, B.J.B. Nyarko, E.H.K. Akaho, J.J. Fletcher, A. Chatt

7. Estimation of combined uncertainties associated with the determination of iodine in foods by the relative and k_0 INAA standardization methods, B.J.B. Nyarko, J.J. Fletcher, E.H.K. Akaho, A. Chatt

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