EVALUATION OF IODINE CONTENT OF SOME COMMONLY CONSUMED FOODS IN ZARIA METROPOLIS, NIGERIA.

By

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Declaration

I declare that the work in this Project Report entitled Evaluation of the Iodine Content of Some Commonly Consumed Foods in Zaria Metropolis, Nigeria, has been carried out by me in the Department of Chemistry. The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this project report was previously presented for another degree or diploma at this or any other Institution.

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Certification

This project report entitled EVALUATION OF THE IODINE CONTENT OF SOME COMMONLY CONSUMED FOODS IN ZARIA METROPOLIS, NIGERIA;By TUKUR MUHAMMAD meets the regulations governing the award of the degree of Masters of Science (Analytical Chemistry) of the Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation

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Dedication

To my family.

Abstract

This thesis investigates the level of iodine consumption in Zariametropolisusing individual duplicate diet assessment, total diet assessment and food frequency questionnaire. The project also includes the study of transfer of iodine from soil to crop in some selected plants, and the determination of iodine contents of table salt. Standard spectroscopic and radiochemical neutron activation techniques were used for iodine determination in the samples and the results obtained were validated by the use of standard reference materials. The concentrations range from 0.14 ± 0.04 to 2.04 ± 0.11mg/ kg, with meanconcentration of1.30mg/kg in duplicate diet sample, while that of total diet range from 0.30 ± 0.05 to 2.09 ± 0.08 mg/ kg, with meanconcentration of 1.43mg/kg.The iodine consumption per day range from 224 to1165 mg/day and 464 to 2237 mg/day for duplicate and total diet respectively.Iodized table salt was found to contain iodine from 14.87 ± 0.51mg/ kg to 74.78 ± 0.59 mg/ kg with a mean of 41.89mg/ kg.The results indicates that the studied population'siodine dietary intake meets the recommended daily allowance(150µg/day) of the WHO and ICCIDD. However, the iodine intake level is also too close to the upper tolerable intake level(1100µg/day) advised by the WHO.

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LIST OF ABBREVIATIONS

FAO	Food Agricultural Organization
ICCIDD	International Council for Control of Iodine Deficiency Disorder
IDD	Iodine Deficiency Disorder
INAA	Instrumental Neutron Activation Analysis
INFOODS	International Food Database
IMVA	International Medical Veritas Association
NIST	National Institute of Standards and Testing
PCNAA	Pre-Concentration Neutron Activation Analysis
RDA	Recommended Daily Allowance
RDI	Required Dietary Intake
S-K Spectroscopy	Sandell- Kolthoff Spectroscopy
SRM	Standard Reference Material
TUI	Tolerable Upper Intake Limit
UNICEF	United Nations Children Fund
USI	Universal Salt Iodization
WHO `	World Health Organization

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background

Micronutrients are required by humans throughout life in small quantities to coordinate a whole range of physiological functions which the body cannot do without (World Health Organization (WHO), 1996). These micro nutrients include iron, cobalt, chromium, copper, iodine, manganese, selenium, zinc, molybdenum and vitamins. They are generally required in less than 100 milligrams per day, and as such are termed dietary trace minerals (WHO, 1996; WHO / FAO, 1998). The human body only needs 150 micrograms (or 20,000th of a teaspoon) of iodine to meet daily requirement and not more than a teaspoon full in an entire lifetime (WHO, 1996).

Iodine is an essential component in the synthesis of L-monoiodotyrosine, which is a precursor of the thyroid hormones, thyroxine (T4) and triiodothyronine (T3), comprising 65 and 59 percent of their respective weights (Ahmed *et al.* 2008). The thyroid hormone T4 and T3 regulate many key biochemical reactions, particularly protein synthesis and enzymatic activity. Major target organs are the developing brain, muscle, heart, pituitary gland, and kidney (Ahmed *et al*., 2008).

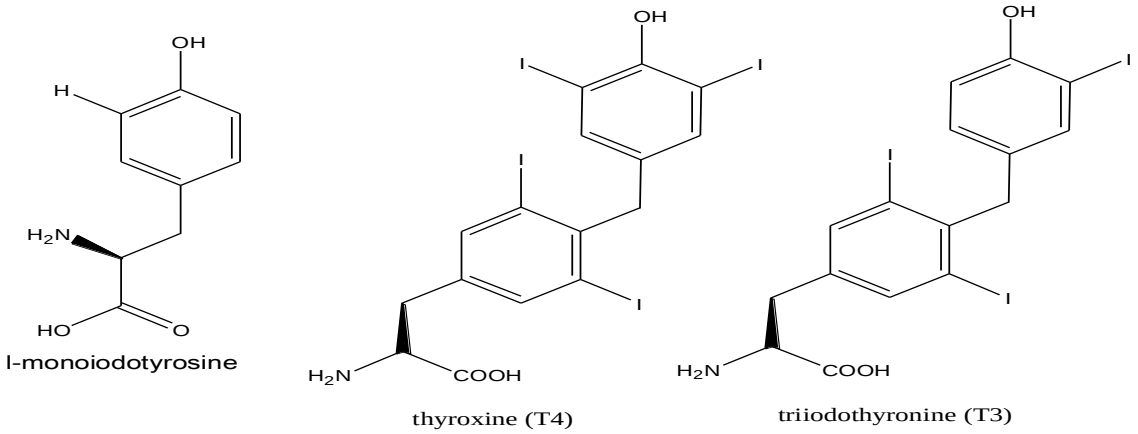


Figure 1: Thyroid hormones

Thyroid hormones, and therefore iodine, is essential for mammalian life, as without sufficient iodine, the body would be unable to synthesize these hormones, and since the thyroid hormones regulate metabolism in every cell of the body and play a role in virtually all physiological functions, iodine deficiency can have a devastating impact on health and well-being which is summarily termed Iodine Deficiency Disorders (IDD) (International Council for Control of Iodine Deficiency Disorders, 2011).Iodine deficiency is the single most common cause of preventable mental retardation and brain damage in the world (De Maeyer*et al*., 1979, ICCIDD, 2011).

Several other physiological functions of iodine have been suggested (ICCIDD, 2011).Iodine plays a role in the prevention of fibrocystic breast disease, a condition characterized by painful swelling in the breasts. Research work by Dijkstra*et al.* (2010) indicates that iodine is involved in controlling the effects of estrogen on breast tissue. It is used in fibrocystic breast disease for mitigating and even overcoming the impact of the medical condition (Dijkstra*et al.*, 2010).

Diets deficient in iodine increase risk of retarded brain development in children (cretinism), goitre (Hetzel and Mark, 1989), mental retardation (Hetzel and Mark, 1989; Hetzel, 2000), high cholesterol (Takeo *et al.* 1963), fatigue and depression (Verse and George, 1935; Takeo *et al.*, 1963), lethargy (Hetzel, 2000), and weight gain (Syed *et al.*, 2001; Loireau*et al.*, 1987). Cretinism is a severe iodine deficiency disorder that causes mental retardation while goitre or swelling of the thyroid gland results in inhibition of immune system function, dry skin, excessive estrogen production and hypothyroidism (Hetzel, 2000).

Iodine deficiency in pregnant women can lead to neurocognitive deficits in the children. Stunted growth, apathy, impaired speech and hearing problems may also develop due to this deficiency (ICCIDD 2011). Iodine is also an important element that determines the health of

connective tissues in fetuses (Perker*et al.,* 1951).Parkinson's disease, multiple sclerosis and Alzheimer's disease have also been linked with iodine deficiency (International Medical Veritas Association, 2011). Consumption of minerals such as magnesium, manganese, calcium, and fluoride can restrict the absorption of iodine uptake by the thyroid gland. Like under-consumption, too much iodine prevents proper production of thyroid hormones leading to iodine induced hyperthyroidism with similar manifestation to IDD (Kennedy, 1942).

The toxicity from iodine depends on its concentration level and the amount of food taken (Brittmarie, 1998). In a balanced diet, the recommended daily intakes of iodine for a normal adult is 100–150 μg per day. In the presence of goitrogens in the diet, the intake should be increased to the range of 200–300 μg per day (WHO, 1996). Information about the proper amount of iodine according to age is given below.

Table 1.1: Dietary reference intake for iodine

Age (years)	Recommended Daily Allowance, RDA.(μg)	Tolerable Upper Intake Levels (μg)
0-6 months	110	ND
7-12 months	130	ND
1 - 3	90	200
4 - 8	90	300
9 - 13	120	600
14 - 18	150	900
19+	150	1100
Pregnant	220	1100
Lactating	290	1100

Institute of Medicine, Food and Nutrition Board, 2001.
ND = Not determinable due to lack of data of adverse effects in this age group and concern with regard to lack of ability to handle excess amounts.

1.2 History

Iodine (Greek; iodes, meaning violet) was discovered by Barnard Curtois in 1811. He was destroying seaweed waste after extraction of nitrate for gun powder production when he

accidentally added too much sulfuric acid. Curtois observed a cloud of purple vapor and he noted that the vapour crystallized on cold surfaces making dark crystals (Moehlmann, 2005).

Endemic cretinism, a condition that was well known in Alpine Europe in the Middle-Ages was mentioned in Diderot's Encyclopedia in 1754 and was of such importance as to warrant a special commission to be set up by the King of Sardinia in 1848 (Hetzel, 1989).

The demonstration for the prevention of cretinism in a double-blind controlled trail with iodized oil in Papua New Guinea established the causal role of iodine deficiency in cretinism by an effect on developing fetus (Pharoah*et al.*, 1971).

Federal Ministry of Health Nigeria, in 1974 formed a Ministerial Committee on iodization of salt. This committee wound up after two years due to poor funding. Efforts from Non-Governmental Organizations (NGO) and individuals led to a meeting of Ministerial Expert Committee on IDD in 1988 to consider the iodization problem again. However, the committee went into abeyance, due to administrative and funding problems again (Hetzel 1989).

Early studies showed that IDD was a public health problem in Nigeria (Ekpechi, 1967; Ekpechi, 1973; Ekpechi*et al.*, 1996). The prevalence of IDD in Nigeria was established by the measurement of total goitre rate which was put at 20% acroos the nation (Egbuta, 2002).

1.3 Policies

The UNICEF (2008) reported that Standard Organization of Nigeria (SON), in 1992, mandated that all table salt should be iodized with 50ppm potassium iodide at packaging stage. This standard was reviewed based on evidences from Universal Salt Iodization (USI) programs worldwide on loss of potassium iodide, especially when sold in bulk in open air markets. The new standard for food grade salt, required potassium iodate to replace

potassium iodide so as to improve retention. The revised SON standard also established packaging to be in 50g retail bags. The new standard defines properly iodized salt as:

i. Greater than 50 ppm iodine at port of entry and salt factory level
ii. Greater than 30 ppm iodine at distributor and retail levels and
iii. Greater than 15 ppm iodine at household level

Codex standard for food grade salt as revised in 1997 and amended in 1999, 2001 and 2006 requires that in iodine deficient areas, food grade salt should be iodized to prevent iodine-deficiency disorders for public health reasons. It also suggested that, the use of sodium and potassium iodides or iodates for the fortification of salt, as well required that maximum and minimum levels used for the iodization of food grade salt are to be calculated as iodine expressed in mg/kg and be established by the National Health Authorities in the light of the local iodine deficiency situation (CX STAN 150:1985).

1.4 Research Problem

Iodine in its natural form is available in food. However, the quantity of iodine in the food is directly proportional to those obtainable in the soil and water of the region (Aston and Brazier, 1979: Pennington, 1990). Mountainous regions and regions with heavy rainfall tend to have very little iodine contents in the soils (Pennington, 1990).

As a reference, the level of iodine in foods such as fish, shellfish, meat, milk, cereal grains, fruits, and vegetables were in the range of 30–4990 mg/kg, with sea foods having the highest concentration (Koutras, 1986).

Table 1.2: Iodine content of some food.

Food	Serving Size	Amount (µg)	% RDA Value
Sea Vegetables	0.25 cup	415.00	276.60
Yogurt	1 cup	87.22	58.10
Milk	1 cup	58.56	39.00
Eggs	1 each	27.00	18.00
Strawberries	1 cup	12.96	8.60
Cheese	1 oz.	10.09	6.70
Whole bread	1 slice	1.95	1.30
Chicken/ Turkey	1 oz.	175.00	116.67
Orange (navel)	1 each	0.60	0.40

Retrieved from Kautras, 1986.

Considering the reported success and ongoing efforts of Universal Salt Iodization (USI) program in Nigeria, some global stakeholders assert that it is difficult to concur that household access to iodized salt is at the elevated levels (United Nations Children and Education Fund (UNICEF), 2008; ICCIDD, 2005).

Although salt iodization is an important strategy in the management of iodine deficiency disorders, dietary habit is a major problem of salt iodization. High dietary salt is considered to cause about 30% of cases of hypertension (Committee on public Health, 2011).

IDD result, primarily from insufficient amounts of iodine in soil (Aston and Brazier, 1979), food and animal feed (Kennedy, 1942) as well as consuming foods rich in goitrogenic substances such as cassava, soybeans, peanuts, millet, spinach, sweet potatoes, rapeseed (Sharpless*et al.*,1943; Moudgal*et al.*,1958).

It is important to note that excessive consumption of iodine can be toxic and just as damaging as a deficiency since the intake of as little as 1000 micrograms of iodine in a day causes irritations like burning of the mouth and throat, nausea, vomiting, stomach ache, and even coma (Salocks*et al.*, 2003).

It is now clear that the effects of iodine deficiency on human health are much greater than previously thought because of the extensive occurrence of hypothyroidism and its effects on

physical and mental development. All these effects can be prevented by the correction of iodine deficiency (Hetzel, 1983).

1.5 Justification

Human micro-nutrition deficiency appears to be much more widely spread than previously thought and it should be noted that human nutrition is analogous to plant nutrition. The World Health Organization, estimates that over three quarter of a billion people are suffering from iodine deficiency disorders (ICCIDD, 2011).

Iodine deficiency in pregnant women causes miscarriages, stillbirths, and other complications. Children with IDD can grow up stunted, apathetic, mentally retarded, and incapable of normal movements, speech, or hearing. Globally, 2.2 billion people (38% of the world's population) live in areas with iodine deficiency and risk its complications (ICCIDD, 2011). Fifty percent of the population in Nigeria lives in rural areas. Malnutrition in children is high, 29 percent are underweight, and 38 percent suffer from stunting (Food Agricultural Organization (FAO), 2011). Nigeria has the largest index of childhood mortality in Africa and the 6th largest in the world (FAO, 2011).

The entire landscape of Nigeria predisposes the country to iodine deficiency disorders because of its proximity to the Equator and the long months of rainfall spreading from April to November (Nwokolo and Ekpechi, 1996: Egbuta*et al.*, 2002). The risk of IDD is quite high in Nigeria, it has a well demarcated goiter belt, where almost all the inhabitants within the belt live on cassava based food staple. At least 60 million Nigerians (from a total population of 140 million) are at risk of IDD. (Egbuta and Hettiaratchy, 1995).

Iodine and other trace elements naturally occur in the soil but erosion leaches off these elements from the soil. Soils with less vegetative cover and locations susceptible to erosion loses their iodine concentrations through the wash up and leaching (Pennington, 1990). A

study on reported goitrous area in Jos plateau shows that the water used for drinking and cooking is low in iodine, other halides and their compounds, but high in mineral contents, including calcium, magnesium, nitrate, chloride and total hardness (Ubom, 1991). Similarly, comparative studies at a community prone to soil erosion, Nanka with nearby Oba (both in Anambra State) to serve as control, shows that Nanka has 14% less iodine in drinking water and 8% less in mean urinary iodine concentration of school children. However, the results indicated that both communities could be at risk of IDD (Orisakwe*et al.,* 2007).

Mapping of livelihoods and nutrition in Nigeria reported that some state such as Abia, Adamawa, Bayelsa, Borno, Delta, Niger have no risk of IDDs, while some states indicate risk at only a few local government areas (L.G.A.) such as Ebonyi with only 4% possible iodine deficiency at Abakaliki L.G.A. and Sokoto with Kware L.G.A. showing 18.50% possible iodine deficiency in its population. AkwaIbom and Kaduna States show possible iodine deficiency in quite a number of L.G.A.s ranging from 10.5 to 29.5 % and 3 to 55.5% with a mean of 9.23% and 12.66% respectively. Bauchi and Oyo states show greater risk of IDDs in the population with an average of possible iodine deficiency at 34.15% and 32.23% respectively. Jigawa state is at most risk with all L.G.A.s (except Gwaram) showing possible iodine deficiencies in their population ranging from 5 up to 100% with a mean of 57.5% (Legg et *al.,* 2002).

Population suffering from iodine deficiency should be supported by more iodine, especially in the form of iodized salt, iodized oil and iodized flour. Iodine supplements are required for the synthesis of thyroid hormones and for correcting neurological, gastrointestinal and skin abnormalities in human beings (Hetzel, 2000).

However, an acute increase in iodine intake when there is chronic iodine deficiency is associated with risk of developing iodine induce hyperthyroidism. That is why the iodine

concentration range (130 to 1100 µg/day) in the diet is very important for public health (WHO, 1996).

1.6 Aim and Objectives

The aim of this research is to study the iodine contents of some commonly consumed foods in Zaria metropolis.

The aim of the study was achieved through the following set objectives:

- Determining the iodine contents in foods consumed by the people of different economic status (income) in Zaria metropolis.
- Determining the concentrations of iodine in agricultural soil, crops, vegetables and foods.
- Comparing the iodine contents in local diets with the Required Dietary Intake (RUI).
- Identifying the foods that are rich in iodine.
- Compare the results obtained with Recommended Daily Allowance (RDA) and Upper Tolerable Intake Level (UTI).

1.7 Research Hypothesis

The Study was guided by the following Null Hypothesis

i. There is no significant difference in the iodine concentrations of local and continental diets.
ii. There is no significant difference in the iodine intake among people with different incomes.
iii. Iodine in food is solely supplied by iodized salt.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 IODINE DETERMINATION.

Iodine is an essential element of human nutrition (Moehlmann, 2005). Even though it is very important for mammalian well-being, it is not an essential element of plant nutrition (Welch and Graham, 1999).

The concentration of iodine in most natural foods is quite low and coupled with its physiochemical property, it is quite difficult to analyse accurately (Dasgupta and Shelor, 2011). Numerous analytical methods have been applied to determine iodine mainly in the form of iodide, such as Spectrophotometry (Van Staden*et al.,* 2004; Gamallo-lorenzo*et al.,* 2005), Electro-potentiometry (Masadome*et al.,* 2000; El-Hallag, 2010), Atomic Absorption Spectroscopy (Adams *et al.*, 1974; Bermejo-Barrera*et al.,* 1999; Yebra and Cespon, 2000) and Chromatography (Rendl*et al*., 1994; Below and Kahlert 2001; Hu *et al.,* 2009).

However, only a few methods are currently used routinely for analysis. These methods are often at the end of sophistication, requiring expensive instrumentation with qualified personnel (inductively coupled plasma-mass spectrometry, instrumental neutron activation analysis) or by kinetic colorimetric method requiring oxidative sample digestion (Dasgupta and Shelor, 2011).The kinetic method has many merits, for example, high sensitivity, good selectivity and simple experimental procedures as well as good reproducibility and low budget apparatus. The kinetic method has been widely applied for determination of iodide, (Van Staden*et al.*, 2004; Gamallo-lorenzo*et al.*, 2005). Most of the kinetic methods are based on the catalytic effect of iodide on oxidation (Gamallo-lorenzo*et al.,* 2005).

The Sandell-Kolthoff reaction is based on the Ce^{4+} -As $^{3+}$ iodine catalyses redox reaction, with rate constant that is stable over the first 3 minutes. The iodide-catalysed reaction is mainly free from significant interference. Ru(IV) and Os(VIII) can catalyse the reduction of

Ce(IV), while Ag(I) and Hg(II) ions significantly inhibit the reaction at low concentrations (Sandell and Kolthoff, 1937; Rodriguez and Pardue, 1969). Other more common ions, such as PO_4^{3-}, SO_4^{2-}, ClO^-, Cl^-, Br^- and compounds such as Fe_3O_2, Al_3O_2, MgO, Cu_2O, Zn_2O have no effect on the assay although any species that absorbs in the region of 350 nm will interfere (Rodriguez and Pardue, 1969).

Travnicek*et al*., (2006) used Sandell-Kolthoff method after alkaline ashing to analyse milk samples collected at dairy farms, from 14 areas of South-western Bohemia. Their result shows that the average iodine content in milk samples was 442.5 ± 185.6 μg/ L, with minimum and maximum value of 68.6 and 1000.6 μg/L respectively. About 82% of the samples contained more than 250 μg/ L. The average iodine content in milk from the collecting areas ranged from 230.2 ± 133.0 μg/ L to 702.7 ± 166.2 μg/ L, with about 36% of collecting areas being higher than 500 μg/L. Higher mean values were recorded in the period of winter feed rations (April 495.9 ± 50.8 and October 494.3 ± 176.4 μg/ L), while lower values were measured during the period of summer feed rations (September 350.9 ± 178.4 μg/ L). If converted per dry matter, 1 kg of dry milk matter contained, on average, 3.428 ± 1.497 mg iodine, maximum and minimum values were 0.543 and 7.995 mg respectively (Travnicek*et al*., 2006).

Gamallo-Lorenzoet*al.*, (2005), employed microwave assisted alkaline digestion with tetramethylammonium hydroxide (TMAH) to digest seaweed samples. The determination of iodide was performed directly in the alkaline digest by Sandell–Kolthoff reaction. The linear calibration range was 0.30–20.0 μg/ kg and the limit of detection was 9.2 μg/ g while the relative standard deviation for 11 determinations was 2.6% for 196.3 mg/kg of iodide.

Xiashi and Yiaqin(2008), satisfactorily applied inhibition kinetic method, which was based on iodine inhibition effects on the reaction between rhodamine and $KBrO_3$ in H_2SO_4 medium

at 45±0.5 ^{O}C for 15 min to determine iodide concentration in kelp. The linear range was 2.0–6.0 ng/ L and the detection limit was 0.06 ng/ L.

Van Staden*et al.,* (2004), proposed a kinetic method for the determination of iodide based on its catalytic effect on the 4, 4- methylenebis (N,N-dimethylaniline)-chloramine-T reaction in acidic medium. This method involves sequential aspiration of tetra base and then chloramine-T solutions into a carrier stream to be stacked inside a holding coil and flow reversed through a reaction coil towards a detector. The resulting coloured compound is measured at 600 nm using an UV/Vis-spectrophotometer, with a linear calibration curve over a range of 0.1–6.0 μg/ L of iodide concentration with detection limit of 0.05 μg/ L (Van Staden*et al.*, 2004).

A catalytic kinetic method for the determination of trace amount of iodine was developed by Liu and Zhang, (1998). The method is based on the inhibitory action of iodide ion on the reduction of toluidine blue with $N_2H_4 \cdot 2HCl$ in hydrochloric acid medium. It has high sensitivity and selectivity for determination of trace iodine. The linear range of determination in the method was 0-8 μg/ L with a detection limit of 0.41μg/ L.

Instrumental Neutron Activation Analysis, INAA, has been used extensibly for the determination of iodine. However, due to Compton interference from ^{28}Al, ^{24}Na, ^{56}Mn, ^{38}Cl, and ^{82}Br, Epithermal NAA along with Radiochemical NAA are often employed for lower detection (Dasgupta and Shelor, 2011).

Table 2.1: Limit of detection of iodine by NAA methods (mg/kg)

Food Item	**INAA**		**ENAA**		**PNAA**		**RNAA**	
	Conc.	**L_D**	**Conc.**	**L_D**	**Conc.**	**L_D**	**Conc.**	**L_D**
Milk Powder (NIST 1549)	3300±200	500						
Mixed Diet (IAEA H9)			**370± 45**	**200**				
Animal Muscle (IAEA H4)					17±2	0.2		
Potato Powder (RM 127)							**3.8±0.2**	**0.02**

Retrieved from Chatt and Zhang, 2012.

Conventionally, iodine is determined through Epithermal Neutron Activation Analysis, ENAA (Dasgupta and Shelor, 2011; IAEA, 2008). Lutz *et al.,* (1987) reported the use of ENAA for the analysis of iodine in foods. The procedure involves irradiation of wet foods in a boron nitride vessel, followed by direct counting of the 442.9 keV gamma ray of 128-I without any processing of the sample. The University of Virginia reactor at Charlottesville with a nominal power of 2.0 MW was employed. Iodine concentrations ranging from <0.003 to 0.74ppm are reported for 17 different food samples. Employment of epithermal instrumental neutron activation analysis in conjunction with anti-coincidence (EINAA-AC) gamma-ray spectrometry method successfully lead to the development of reliable procedure for determination of iodine in biological materials. Analysis of SRM shows 92 to 108% accuracy (Chatt and Zhang, 2012).

Table 2.2: Summary of NAA regimes for determination of Iodine

Flux ($ncm^{-2}s^{-2}$)	Irradiation Time (sec)	Decay Time (min)	Counting Time (min)	Shield	Reference
2MW	120- 180	15	15-25	4mm BN	Lutz *et al.,* 1987
9MW	30	15-20	20-25	1mm Cd	Chatt and Rao, 1991
$8x10^{13}$	10	7.5	10	5mm BN	Nicholas *et al.,* 1998
$2x\ 10^{12}$	600	$\geq$ 10	10	1mm Cd	Chien-Yi *et al.,* 2003
$4x10^{12}$	30	20	20	1mm Cd	Maihara*et al.,* 2008
$1x10^{11}$(epi)	120	5	5	2mm Cd	IAEA-TECDOC-1592,2008

Lansberger and Wu, (1993) determined iodine in airborne particulate matter by epithermal neutron activation analysis using Compton suppression method. Chatt*et al.,* (2012) used the TRR1/M1, in the Thailand Institute of Nuclear Technology, with a maximum in-core thermal neutron flux of 3.9×10^{13} n cm^{-2} s^{-1} to determine iodine and other elements in Thailand diet. The result shows 5 % or better deviation from the Standard Reference Material (SRM) samples analysed.

Pre-concentration Neutron Activation Analysis, (PNAA), methods for iodine determination are well established, and the reported methods have solved the problem of personnel

exposure during post irradiation processing as well as possessing similar or better precision and accuracy (Chatt and Zhang, 2012). The most common procedure reported by Chatt and Rao, (1991) and is currently being employed by National Institute of Standard and Testing (NIST) for SRM certification (NIST SRM 1548a, 2009). The procedure involves co-precipitation of iodine with bismuth and sulfide. The method has a reported absolute detection limit of 5ng with relative standard deviation of 5% at 100 ppb and 10% at 20 ppb (Chatt*et al.,* 2012).

2.2 Theory of Analytical Method (NAA)

Neutron activation analysis (NAA) with its inherent sensitivity, selectivity and freedom from analytical blank is the technique of choice for determining iodine even at ultra-trace levels in biological materials (Dasgupta and Shelor, 2011; IAEA TECDOC-564, 1990).

Neutrons of energy ranging from about 0.025eV (thermal) to greater that 10keV (fast) are employed in non- elastic collisions to excite target nuclei into compound nuclei with the release of prompt gamma ray. The compound nuclei returns to ground state by emitting the absorbed energy in form of particles (beta, alpha etc.) and energy (delayed gamma). The delayed gamma is captured by a suitable detector and forms the basis for Neutron Activation Analysis (NAA). Unknown sample and comparator standard are both measured on the same detector and the decay and irradiation times between the two are corrected, the mass of the element in the unknown will be calculated by using the half-life of the measured isotope (IAEA-TECDOC-564, 1990).

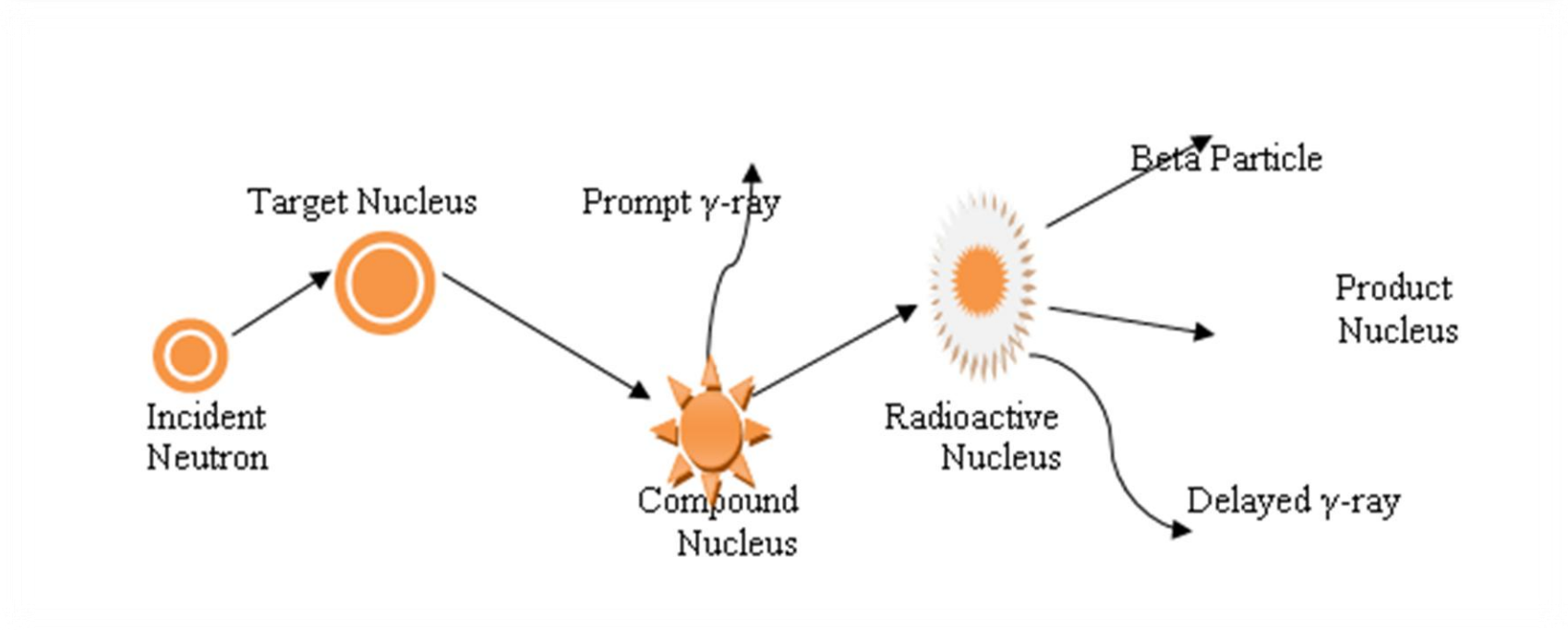


Figure 2.1: Scheme of Neutron Activation Analysis (NAA).

Retrieved from Radiation Center, Oregon State University, 2003.

The equation 1.0 below is used to calculate the mass of an element in the unknown sample relative to the comparator standard.

$$\frac{A_{sam}}{A_{std}} = \frac{M_{sam}(e^{-\lambda T_d})_{sam}}{M_{std}(e^{-\lambda T_d})_{std}} \text{ ---------- } (1.0)$$

Where A = activity of the sample (*sam*) and standard (*std*)

M= mass of the element

λ= decay constant for the isotope and

T_d = decay time.

For a stable reactor flux, the irradiation, decay and counting times are normally fixed for all samples and standards such that the time dependent factors cancel. Thus equation 1.0 simplifies equation 2.0.

$$C_{sam} = C_{std}\frac{W_{std}A_{std}}{W_{sam}A_{sam}} \text{ ---------- } (2.0)$$

Where C= concentration of the element and

W= weight of the sample and standard.

Table 2.3: Iodine Nuclear Data.

Target nuclide	127I
Nuclear Reaction	^{127}I (n,γ) ^{128}I
Natural abundance (%)	100
Thermal cross-section, barns	4.04
Resonance integral, barns	100
Gamma-ray energy, keV	442.9
Half-life of Product	24.99 m

Retrieved from IAEA-TECDOC-564, (1990)

2.3 Detection and Counting

The basic premise of detection and counting is that the decay rate of the source or activity of the sample (R) is proportional to the number of atoms of radioactive nuclide present (N), the proportionality constant being the decay constant λ (IAEA-TECDOC-564, 1990), Thus:

$$R = \frac{\delta N}{\delta t} = \lambda N \text{----------} \quad (3.0)$$

Given the count for the number of events C, detected by the detector in a fixed period of time δt, it can be estimated that the decay rate is as follows

$$R = \frac{C}{\varepsilon \delta t} \text{----------} \quad (4.0)$$

ε is the effective efficiency of counting taking into account the source, detector geometry, the intrinsic detection efficiency of the particular radiation and the probability of emission of the detected radiation. Considering that each atom in the sample will have a certain probability P that the atom will decay during the period of measurement

$$P = (1 - e^{-\lambda \delta t}) \text{----------} \quad (5.0)$$

Then, the overall probability of detection as opposed to decay is Pε and the expected count is

$$E(C) = P\varepsilon N \text{----------} \quad (6.0)$$

If the measured count C is an estimate of the expected count, then equation (6.0) above can be employed to calculate the rate of decay as

$$R = \lambda N = \frac{\lambda C}{(1 - e^{-\lambda \delta t})\varepsilon} \text{----------} \quad (7.0)$$

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

3.1.1 Description of the study area

Zaria is the capital city of the ancient Zazzau Kingdom as well as the headquarters of Zaria Local Government Area. However, the name Zaria now applies to a large metropolitan area that includes the ancient city as well as neighboring towns/ villages such as Sabongari, Samaru, Shika and Soba. Zaria metropolis has a population of 1.5 million, situated in Kaduna State, Nigeria, and located on longitude N11 and latitude E7 with an approximate land mass of 300 sq. Km. Zaria is about 950 Km from the coast on an altitude of 653.6 Km. (*Ahmadu Bello University website*, 2013).

A significant percentage of the population comes from all over Nigeria due to the presence of many Federal Institutions. As such, Zaria can be divided into four broad regions, the ancient city and Tudunwada which hosts mostly the indigenous population, the TudunJukun-Sabongari Area which hosts quite a diverse set of people from all over Nigeria, the Government Residential Area (GRA), which accommodates both the natives and non-natives, mostly of higher income earners and the Samaru Area which have both the indigenous population from villages around as well as a significant population that are mostly students and civil servants from the surrounding Federal Institutions (Abbas and Arigbede, 2011).

The people of the ancient city of Zaria are mostly farmers, traders and skilled artisan, with some civil servants. The people of Sabongari are mostly traders, with a significant portion that were/ are staff of agro-allied industries and the railway, with some more civil servants. However, the population of Samaru is predominantly civil servants, with some of the indigenous population being farmers and artisans (Abbas and Arigbede, 2011).

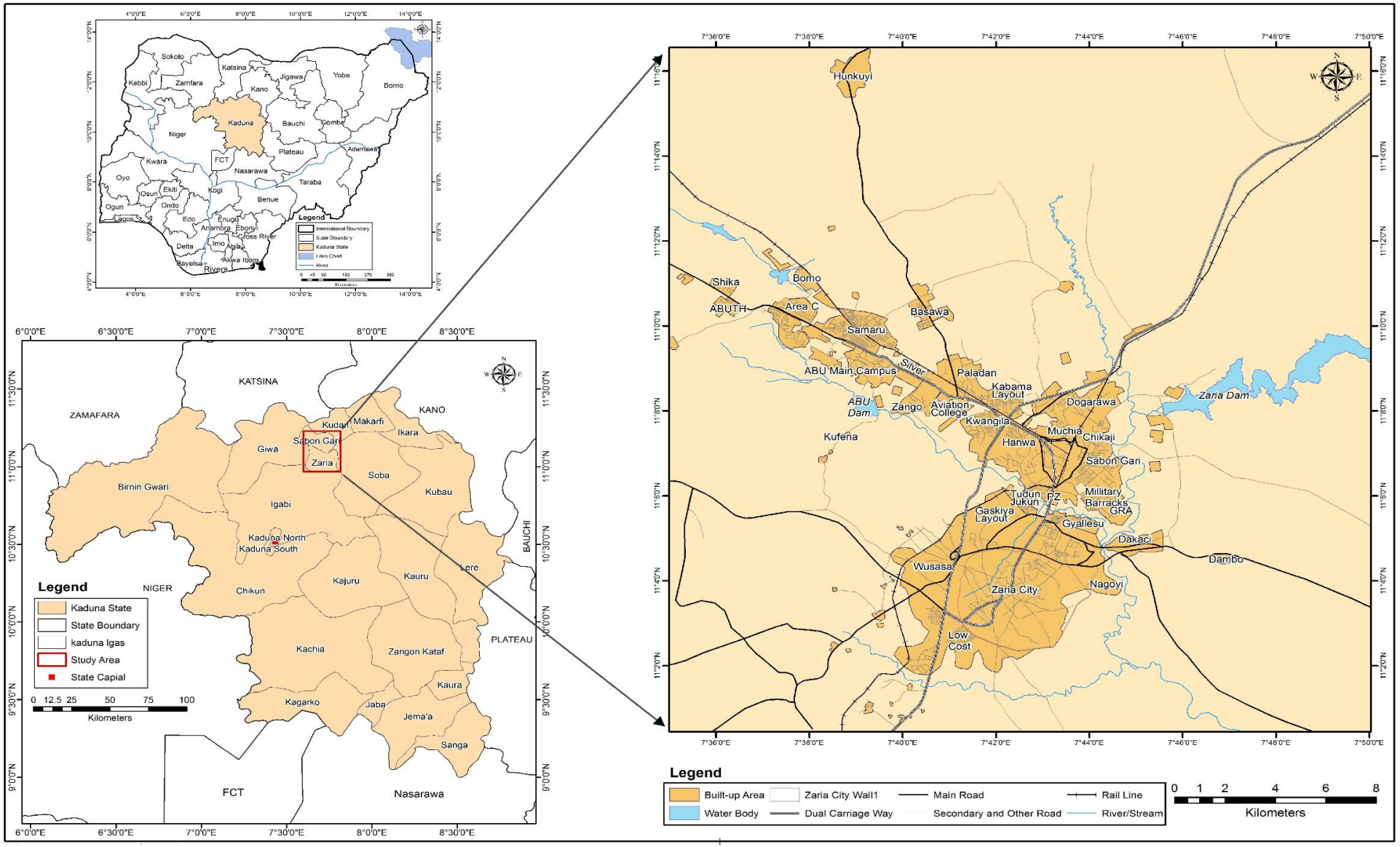


Figure 3.1: Map of Zaria

Adapted and modified from the administrative map of Kaduna state, 2013.

3.1.2 List of apparatus and equipment

The apparatus, equipment and reagents used are listed in Appendix I. All reagents were of analytical grade and were obtained from Sigma- Aldrich through Zayo sigma except sodium hydroxide (NaOH), sodium thiosulphate ($Na_2S_2O_{3)}$ and potassium iodate (KIO_3) which were produced by BDH analar grade were obtained through local distributors.

3.1.3 Preparation of reagents

- 5% w/v Hydrazine Sulphate ($H_4N_2.H_2SO_4$, molecular weight (mw)130.12 g/mol)

  50 g of hydrazine sulphate was dissolved in about 500 cm^3 of deionized water in a 1000 cm^3 volumetric flask, then the flask was filled to the mark with deionised water, caped and invert several times to ensure homogeneity.

- 10% w/v Ammonium hydroxide (density= 0.88, 32.0% NH_3 basis, in $H_2O \geq 99.99\%$)

  11.36 cm^3 of ammonium hydroxide was dissolved in about 50 cm^3 of deionized water in a 100 cm3 volumetric flask, then the flask was filled to the mark with deionized water and then the flask was invert several times to ensure homogeneity.

- 10% w/v Potassium Iodide (KI, mw= 166.0 g/mol)

  50 g of potassium iodide was dissolved in about 250 cm^3 of deionized water in a 500 cm^3 volumetric flask, then the flask was filled to the mark with deionised water.

- 0.1M Bismuth nitrate ($BiN_3O_9.5H_2O$, mw- 485.07 g/mol)

  In a 250cm^3 volumetric flask, 12.14g of bismuth nitrate was dissolved in 60 cm^3 of 5% nitric acid, and then the flask was filled to the mark with deionised water, and invert several times to ensure homogeneity.

- 0.25M Thioacetamide ((C_2H_5NS, mw- 75.13 g/mol)

  4.70g of thioacetamide was dissolved in about 100 cm^3 of deionized water in a 250 cm^3 volumetric flask, then the flask was filled to the mark with deionized water and invert several times to ensure homogeneity.

- 0.1M Potassium Nitrate (KNO_3, mw- 101.1 g/mol)

  10.11g of potassium nitrate was dissolved in about 500 cm^3 deionized water in a 1000 cm3 volumetric flask, then the flask was filled to the mark with deionized water and invert several times to ensure homogeneity.

- 1mM Sodium Hydroxide (NaOH, mw- 40.0 g/mol)

  0.04g of sodium hydroxide was dissolved in about 100 cm^3 of deionized water in a 1000 cm^3 volumetric flask, then the flask was filled to the mark with deionized water and invert several times to ensure homogeneity.

- 0.1M Sulphuric acid (98% H_2SO_4, mw-98.08 g/mol , density- 1.84 g/mL at 25 °C)

  5.5 cm^3 of 98% sulphuric acid was dissolved in about 600 cm^3 of deionized water in a 1000 cm^3 volumetric flask, then the flask was filled to the mark with deionized water and invert several times to ensure homogeneity.

- 0.1M Cerium Ammonium Sulphate ($2(NH_4)_2SO_4.Ce(SO_4)_2.2H_2O$, mw- 633.56 g/mol)

  In a 500cm^3 volumetric flask, 33.43g of cerium sulphate was dissolved in 100 cm^3 of 0.1M sulphuric acid, then the flask was filled to the mark with deionized water and invert several times to ensure homogeneity.

- 0.1M Arsenious acid (H_3AsO_3) from arsenic trioxide (As_2O_3, mw- 197.84 g/mol)

  In a 500cm^3 volumetric flask, 4.95g of arsenic trioxide was dissolved with gentle heating in 20 cm^3 of 10% sodium hydroxide, then the flask was filled to the mark with 5% sulphuric acid and invert several times to ensure homogeneity.

- 0.005M Sodium thiosuphahte ($Na_2S_2O_3 \cdot 5H_2O$, mw- 248.18 g/mol)

0.124g of sodium thiosulphate was dissolved in about 60 cm^3 of deionized water in a 100 cm^3 volumetric flask, then the flask was filled to the mark with deionized water and invert several times to ensure homogeneity.

- 1000 ppm iodine solution (KIO_3, mw- 214.0 g/mol)

  4.038g of potassium iodate was dissolved in about 500 cm^3 of deionized water in a 1000 cm^3 volumetric flask, then the flask was filled to the mark with deionized water and invert several times to ensure homogeneity. Subsequent iodine standards were prepared by dilution of the stock using the dilution formula.

$$C1V1 = C2V2$$

3.1.4 Sampling.

The following sampling methods were adopted in order to achieve a true representation reflecting the population;

i. Dietary assessment method

The dietary assessment method is based on taking a dietary history, usually 24 hour recall. A questionnaire that takes into account the type of food a population normally consumed as well as the frequency and amount (Thompson and Suber, 2008; National Obesity Observatory, 2010). Intake of iodine from the data obtained is calculated using international food composition databases. The 24 hour recall questionnaire is shown in Appendix II (a) and (b).

ii. Total diet studies

This involves purchasing foods commonly consumed, then processing and analyzing them for analyte(s) of interest (iodine). The dietary intake of the food components was estimated from the analytical results and food intake information for different

population groups (Thompson and Suber, 2008; National Obesity Observatory, 2010).

iii. Duplicate portion method

This entails accurate assessment of the nutrient intake of an individual. An exact duplicate of intake (fluids and solid foods eaten) over the survey period were collected and analyzed. Margetts and Nelson (1991) and Thompson and Suber, (2008), establish the accuracy of this method and suggested its use as standard against other methods.

3.1.4.1 Sample Collection

i. Questionnaire: three hundred and fifty (350) questionnaires were distributed and a total of two hundred and eighty two were recalled and processed. The location and frequency are given in results.

ii. Total diet studies; a total of 23 food samples were collected from restaurants, while 21 salts samples were collected from markets and shops across Zaria. The result for the dietary assessment method was employed as guide for collecting samples. All samples were collected in triplicate portions on alternate days. The samples were collected in polyethylene container and were immediately processed. Table 3.1a shows the locations where food samples were collected, weight and cost of the samples. Salt sampling was carried out by random sampling of the available brands of retail packs (CX STAN 150-1985).

iii. Duplicate portion method: An exact duplicate of foods intake by volunteers were collected over a 24 hour period. Salt sample from volunteers were collected along with the food samples. Five volunteers also supplied farm soil and produce for studies of iodine in farm soil and crops grown on it. Samples were collected from twelve (12) volunteers across Zaria metropolis.

Table 3.1:Total diet samples and cost

S/N	Location	Code	Item*	FM (Kg)	DM (Kg)	FM (Kg)	DM (Kg)	FM (Kg)	DM (Kg)	Average FM (Kg)	Average DM (kg)	Cost
1	Smrmkt	RL 1-1	Koko da Kosai	1.010	0.2124	0.9734	0.2084	1.0234	0.2103	1.0026	0.2104	N90
2	Smrmkt	RL 1-2	Rice and beans meat, stew	0.3953	0.0637	0.3665	0.0617	0.3939	0.0626	0.3852	0.0627	N150
3	Smrmkt	RL 1-3	TuwonMasar, dry okra sauce	0.8821	0.2226	0.9289	0.2051	0.7935	0.2095	0.8682	0.2124	N100
4	Twdmrt	RL 2-1	Fried Sweet potato and Gruel	0.6233	0.2110	0.6678	0.2152	0.7051	0.2054	0.6654	0.2106	N70
5	Twdmrt	RL 2-2	Rice, Spagetti and vegetable	1.0201	0.2361	1.1619	0.2384	1.0970	0.2176	1.0930	0.2307	N70
6	Twdmrt	RL 2-3	TuwomMasaramiyartaushe	1.2173	0.2411	1.2214	0.2351	1.2323	0.2386	1.2237	0.2383	N150
7	Zcmrt	RL 3-1	Moi-Moi	0.6389	0.1578	0.7031	0.1553	0.5747	0.1551	0.6389	0.1560	N70
8	Zcmrt	RL 3-2	Jellop Rice and Beans	0.5763	0.2290	0.5529	0.2202	0.5458	0.2256	0.5593	0.2249	N100
9	Zcmrt	RL 3-3	TuwonMasar, dry okra sauce	1.2426	0.2210	1.0597	0.2008	1.4552	0.2400	1.2525	0.2206	N100
10	Hanwa GRA	ML 1-1	Potato chip, Eggs, Liver suace	0.5107	0.2507	0.5208	0.2399	0.5777	0.2615	0.5364	0.2507	N350
11	Hanwa GRA	ML 1-2	Fried rice, pea, carrot, carbage	0.4801	0.1685	0.4797	0.1671	0.5248	0.1672	0.4949	0.1676	N450
12	Hanwa GRA	ML 1-3	Pounded Yam,Vegetable soup	0.9841	0.3327	0.9806	0.3298	0.8756	0.3402	0.9468	0.3342	N400
13	Zaria City	ML 2-1	Waina da Taushe	0.7351	0.2687	0.8104	0.2637	0.6404	0.2391	0.7287	0.2571	N350
14	Zaria City	ML 2-2	White rice ,vegetable salad	0.5303	0.1899	0.5330	0.1798	0.6128	0.1960	0.5587	0.1886	N500
15	Zaria City	ML 2-3	Tuwonshinkafa, egusi soup	0.7132	0.1381	0.7065	0.1366	0.7430	0.1398	0.7209	0.1382	N400
16	Samaru	ML 3-1	Potato chip, Eggs, Beef suace	0.6303	0.2510	0.7445	0.2699	0.6433	0.2463	0.6727	0.2556	N400
17	Samaru	ML 3-2	Fried rice, pea, onion, beef	0.3375	0.1275	0.3370	0.1253	0.3519	0.1291	0.3421	0.1273	N500
18	Samaru	ML 3-3	Tuwonshinkafa, egusi soup	0.8713	0.1403	0.9931	0.1579	0.9464	0.1328	0.9369	0.1437	N500
19	Hanwa	HL 1-1	French Fries, Eggs, liver sauce	0.5127	0.1636	0.5159	0.1622	0.5160	0.1635	0.5149	0.1631	N1035
20	Hanwa	HL 1-2	Savory Rice, vegetable chicken	0.9559	0.2618	0.9260	0.2572	1.0071	0.2769	0.9630	0.2653	N1100
21	Hanwa	HL 1-3	Semovita,Vegetable Soup	0.9366	0.2578	0.9943	0.2920	0.8966	0.2567	0.9425	0.2688	N1050
22	Pz	HL 2-1	Not available									
23	Pz	HL 2-2	Fried Rice and salad, chicken	0.4687	0.1841	0.4605	0.1775	0.4917	0.1956	0.4736	0.1857	N900
24	Pz	HL 2-3	Pounded Yam,Vegetable soup	0.8871	0.3018	0.8794	0.2968	0.8393	0.2792	0.8686	0.2926	N950

Smrmkt = Samaru market; Twdmrt= Tudunwada market; Zcmrt= Zaria city (AbubakarLadan) Market

FM= Fresh mass; DM= Dry mass

* Approximation of names in English is presented in Appendix III.

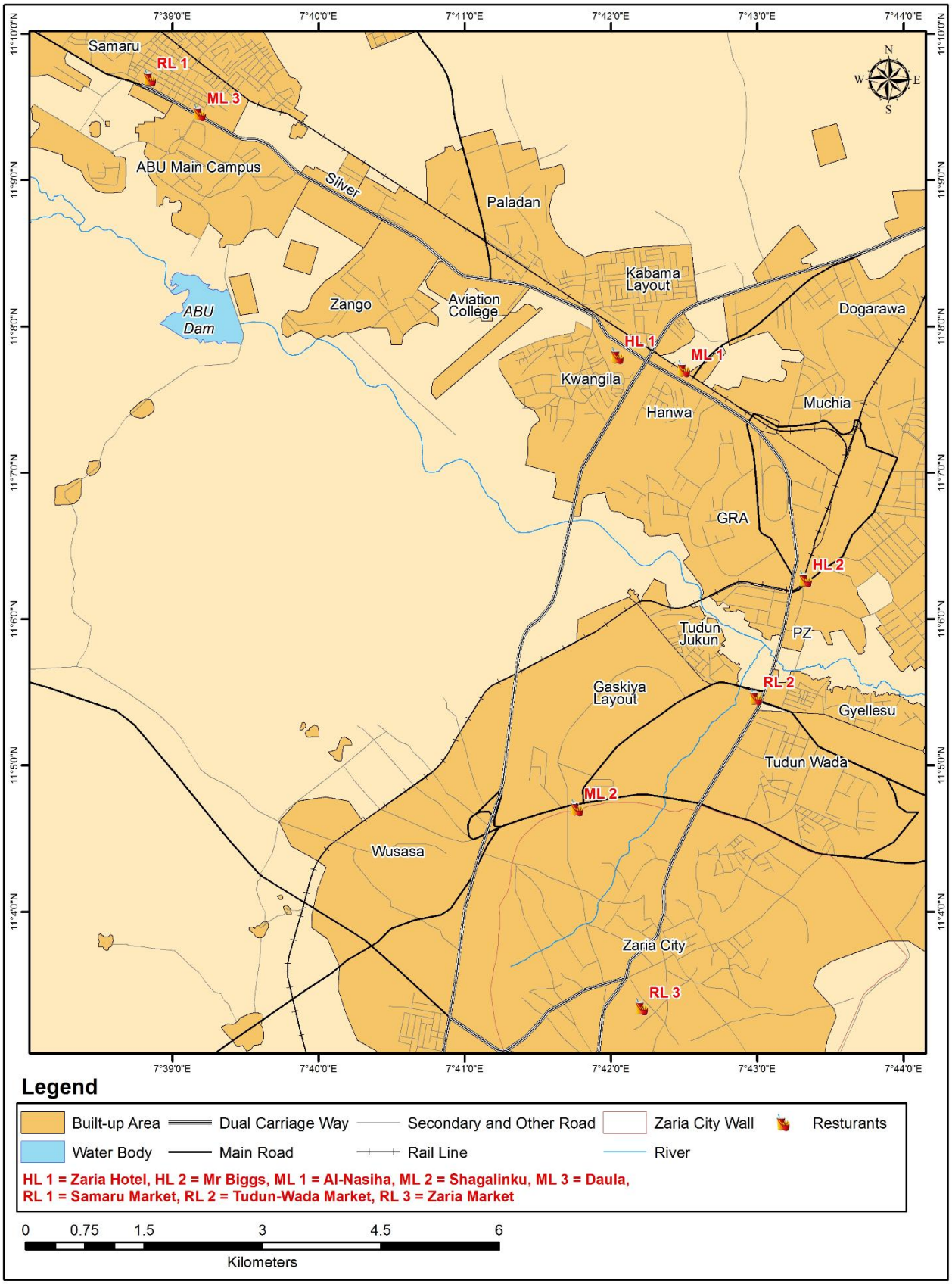


Figure 3.2: Map of Zaria showing Total diet Sampling Points

Adapted and modified from the administrative map of Kaduna state, 2013

Table 3.2: Duplicate diet samples

S/N	Code	Item	Fresh Mass (kg)	Dry Mass (kg)
1	HDM01-1a	Yam with chicken sauce, Tea and lemon	0.3614	0.1461
2	HDM01-2a	Jellop rice and beef	0.4838	0.1709
3	HDM01-3a	Dashishi beef stew	0.2243	0.0850
4	HDM01-1b	Fried Sweet Potato, tea and milk	0.5078	0.2063
5	HDM01-2b	TuwonShinkafamiyar wake	0.7444	0.2394
6	HDM01-3b	AlkubusmiyarTaushe, beef, chicken	0.1814	0.0934
7	HDM02-1	Buttered Breas, Egg omlette, tea and milk	0.3275	0.1002
8	HDM02-2	TuwonShinkafa, dried okoro soup	0.8187	0.1821
9	HDM02-3	White rice, beef stew and lettuce	0.4927	0.2093
10	HDM03-1a	Tea, milk, 4 slice of bread and butter	0.2823	0.0706
11	HDM03-2a	Jellop rice and beef	0.3979	0.1158
12	HDM03-3a	Tuwonshinkafamiyarkuka	0.6315	0.1413
13	HDM03-1b	Wainarshinkafa da romonkai	0.6128	0.2142
14	HDM03-2b	Tuwonshikafamiyarkuka	0.6154	0.1118
15	HDM03-3b	Jelloproce	0.3931	0.1115
16	HDM04-1	French fries, lettuse, baked beans	0.5826	0.2164
17	HDM04-2	Rice and beans	0.7376	0.2761
18	HDM04-3	Jellop rice	0.6592	0.2634
19	HDM05-1	Koko, Kosai, Namanrago	0.1448	0.0930
20	HDM05-2	Jellop rice, fish and lettise, carbage	0.8856	0.5958
21	HDM05-3	Tuwonshinkafa vegetable soup	0.8340	0.2157
22	HDM06-1	Fried yam, kununtsamiya	1.2078	0.3182
23	HDM06-2	White rice and beef stew	0.5543	0.2394
24	HDM06-3	Jellop rice, carrot, pea, cabbage	0.5328	0.1649
25	HDM07-1	Wainarshinkafa da bindinshanu	0.5386	0.1681
26	HDM07-2	Semovita and dried okoro soup and beef	1.2484	0.2672
27	HDM07-3	Hura	0.6238	0.1012
28	HDL08-1	Moi- Moi and Pap	0.7126	0.2072
29	HDL08-2	Taliya da mai	0.7329	0.1540
30	HDL08-3	Tuwonmasaramiyarkuka	0.7033	0.1568
31	HDL09-1	Kosai da Kununtsamiya	0.9060	0.1644
32	HDL09-2	Tea and Biscute	0.4128	0.0217
33	HDL09-3	Rice and stew	1.0325	0.2395
34	HDL10-1	Tuwonmasaramiyartaushe	1.6224	0.5161
35	HDL10-2	Skipped	0.0000	0.0000
36	HDL10-3	Tuwonmasaramiyartaushe	1.4412	0.4947
37	HDL11-1	Kosai da Kununtsamiya	0.8868	0.1408
38	HDL11-2	Fried yam	0.2835	0.1499
39	HDL11-3	TuwonShinkafamiyartaushe	0.7375	0.2655
40	HDL12-1	Tuwondawamiyarkubewadanya	1.0185	0.2954
41	HDL12-2	Jellop rice	0.9559	0.2581
42	HDL12-3	Skipped	0.0000	0.0000

* Sample description is presented in Appendix III

Foods samples were collected in polyethylene containers and were immediately processed in the laboratory. Water samples were also collected in polyethylene bottles and processed immediately. Salt samples are collected directly from storage vessel and were tightly packaged in polythene bags. The crop and vegetables samples were collected randomly from the storage prior to processing for cooking. Soil samples were collected in multiples to form a bulk sample then reduced to laboratory by coning and quartering (IUPAC, 1990). Samples were collected in doubled acid pre-cleaned polyethylene bags. Figure 3 shows map of food sampling areas and figure 4 shows map of farm sampling areas. The volunteers were group in low (less than ₦ 79500), average (₦ 79500 to ₦ 124000), and above average (more than ₦ 124000) monthly incomes (World Bank Country Survey: Nigeria).

1. Volunteer number one (HDM1a and HDM1b) is an adult female of a family of four (three adults, one adolescent), of average income residing at Collage of Aviation Staff Quarters. Two Soil samples from farmlands located at Zabi(11^{o} 08′ 57″ N, 07^{o} 44′ 41″ E) and Hunkuyi (11^{o} 15′ 39″ N, 07^{o} 38′ 57″ E) villages, along with the farm produce, Wheat and Beans respectively, were collected. Six meals, two salt and drinking water samples were collected from her.
2. Volunteer number two (HDM2 is of above average income, an adult male of a family of four (two adults, a toddler and an infant), residing at G. R. A. Three food samples, salt and drinking water were collected from him.
3. Volunteer number three (HDM3a and HDM3b) is an adult female, from a family of six (two adults, two adolescents, a child and a toddler), residing at Military barrack, Sabongari. Six food samples, two salt and drinking water samples were collected from her. The volunteer is of above average income.

4. Volunteer number four (HDM4) is an adult male of a family of four (three adults and an infant), residing at area C, A.B.U. staff quarters. The volunteer is of average income. One soil sample from farmland located at Shika Village (11^{o} 19′ 35″ N, 07^{o} 59′ 23″ E), one farm produce (rice), three food sample and salt were collected from him.
5. Volunteer number five (HDM5) is an adult male of a family of eight (three adults, three adolescents, a child and an infant), residing at Limanci, Zaria city. One soil sample from farmland located at Gwargwaje (11^{o} 02′ 41″ N, 07^{o} 40′ 40″ E), one farm produce (spinach), three food samples, salt and drinking water were collected from him. The volunteer is of average income.
6. Volunteer number six (HDM6) is an adult male of a family of three (two adults, a toddler), residing at Dogarawa, Sabongari. Three food samples, salt and drinking water were collected from him. The volunteer is of average income.
7. Volunteer number seven (HDM7) is an adult female of a family of five (two adults, two children and an infant), residing at G. R. A. Three food sample, salt and drinking water were collected from her. The volunteer is of average income.
8. Volunteer number eight (HDL8) is an adult male of a family of eight (two adults, three adolescents, two children and an infant), residing at Hayin-dogo village, Samaru. One soil sample from farmland located at Jama'a village (11^{o} 08′ 29″ N, 07^{o} 39′ 18″ E), one farm produce (maize), three food samples, salt and drinking water were collected from him. The volunteer is of low income.
9. Volunteer number nine (HDL9) is an adult male of a family of six (two adults, three children and an infant), residing at Bomo, Samaru Three food samples, salt and drinking water were collected from him. The volunteer is of low income.

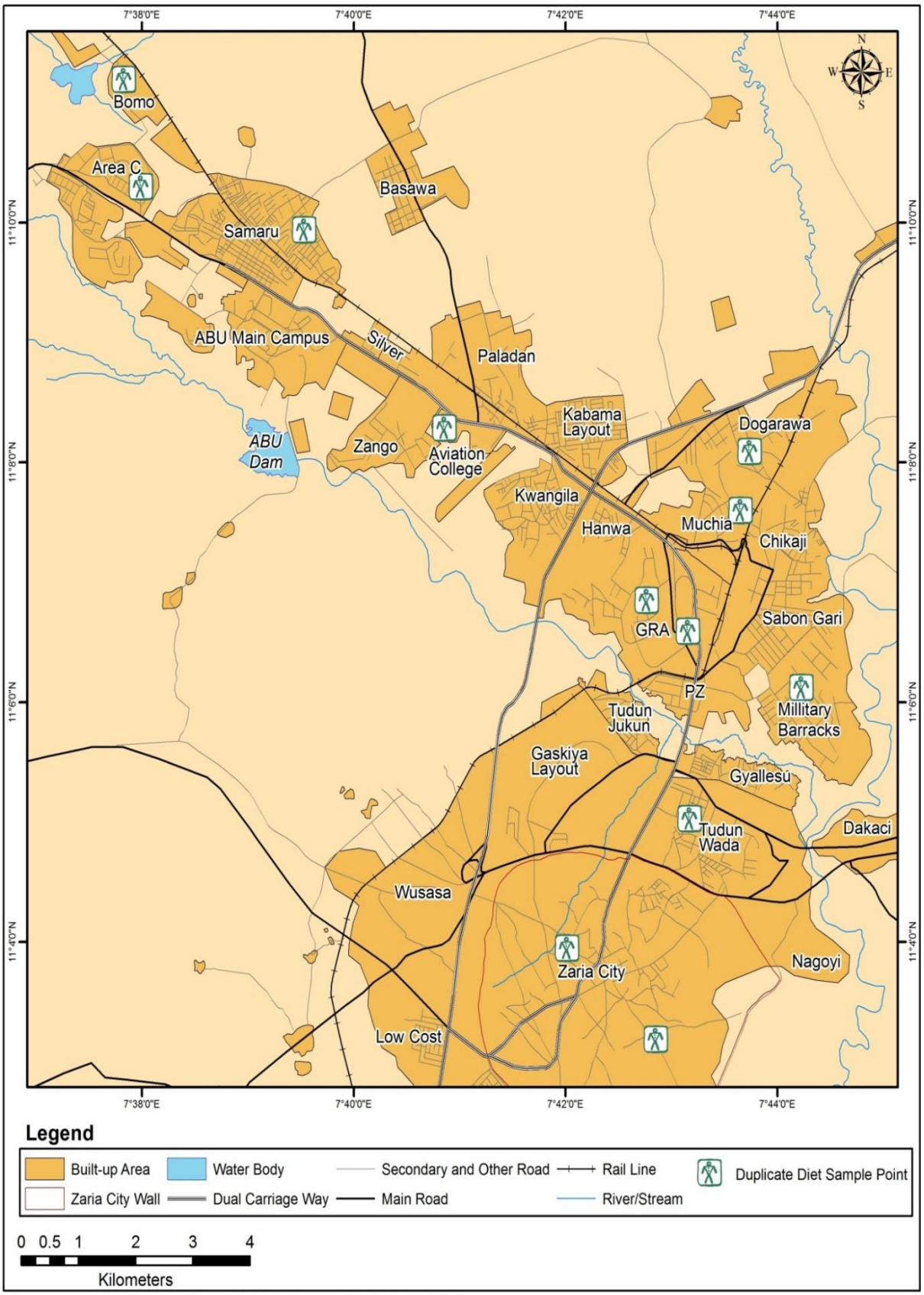


Figure 3.3: Map of Zaria showing Duplicate Diet Sampling Points

Adapted and modified from the administrative map of Kaduna state, 2013

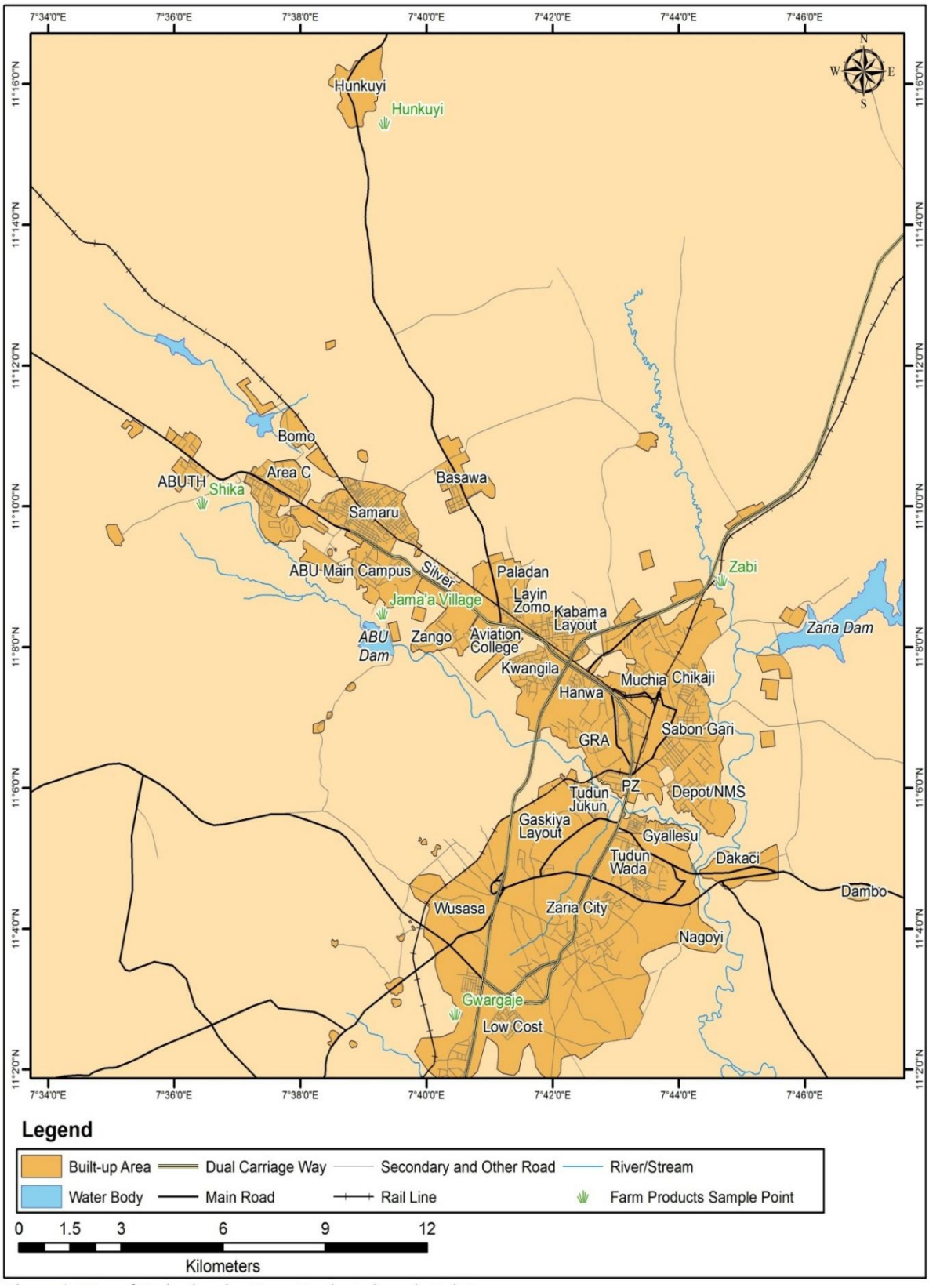


Figure 3.4: Map of Zaria showing Farm Produce Sampling Points

Adapted and modified from the administrative map of Kaduna state, 2013

10. Volunteer number ten (HDL10) is an adult male of a family of twelve (three adults, one adolescent, six children and two infants), residing at Alfadarai, Zaria city. Three food samples, salt and drinking water were collected from him. The volunteer is of low income.
11. Volunteer number eleven (HDL11) is an adult male of a family of six (two adults, four children), residing at Tudun Wada. Three food samples, salt and drinking water were collected from him. The volunteer is of low income.
12. Volunteer number twelve (HDL12) is an adult male of a family of five (two adults, two children and an infant), residing at Muchia, Sabongari. Three food samples, salt and drinking water were collected from him. The volunteer is of low income.

3.1.4.2 Sample Processing

Soil: samples were dried in the oven at a temperature of 50^{o} C. The sample was cooled and weighed. This process was repeated until when a constant weight was obtained. Then, the soil was then grounded using vibratory ball mill and homogenized by through mixing and seal in an air tight polyethylene bag (IAEA-TECDOC-564, 1990).

Farm produce: (wheat, beans, rice, spinach and maize)samples were oven dried at a temperature of 50^{o} C. The samples were cooled and weighed. This process was repeated until when a constant weight was obtained. The samples were then ground to powder and homogenized using an agate mortar and stored below -4^{o} C (IAEA-TECDOC-1592, 2008).

Food and water: samples were mixed in to slurry by warring laboratory blender, equipped with stainless steel titanium coated blade, then oven dried at 45 ± 2^{o} C. The samples were cooled and weighed. The cooling and weighing process was repeated until when a constant weight was obtained. The resulting dried material was pulverized and homogenized using an agate mortal and stored below -4^{o} C (IAEA-TECDOC-1592, 2008).

3.2 METHODS

The soil, produce and food samples were analysed for Na, K and Br by INAA while I in the samples was analysed by PCNAA and Sandell- Kolthoff spectroscopy. Salt samples were analysed by iodometric titration.

3.2.1 Instrumental Neutron Activation Analysis.

Samples were thoroughly homogenised to ensure consistency. About 0.15g of soil sample was measured into polyethylene bag and repacked in another bag with the identification tag in the outer bag. The bag was then heat-sealed. 0.15g of IAEA standard reference material (SRM), SL-3 (Lake Sediment), was also packaged identical to the samples. Then, samples and SRM were packaged into the same irradiation capsule (IAEA-TECDOC-564, 1990: Jonah *et al*., 2006).

Dried farm produce and food samples were retrieved from the refrigerator and immediately about 0.25g of each sample was weighed singly into polyethylene bag and repacked in another bag with the identification tag in the outer bag. The bags were then heat-sealed. Standard reference materials, NIST 1573a (Tomato Leaves), NIST 1515 (Apple Leaves), NIST 1570a (Spinach leaves) and IAEA 359 (Cabbage) were packaged identical to the samples. Not more than six samples and one SRM were then packaged into one irradiation capsule (Jonah *et al.*, 2006: Chatt*et al*., 2012).

The samples were irradiated at the inner irradiation channels (A1, B2 and B3) of the Nigerian Research Reactor 1 (NIRR-1) for six (6) hours at a nominal flux of $5e^{11}ncm^{-1}s^{-1}$. The samples were allowed a decay period of three to four days for medium-lived radioisotopes and ten to twelve days for long-lived radioisotopes, after which the nuclide spectrum was obtained by counting the samples for 1800 and 3600 seconds respectively with a high-purity Germanium (HPGe) detector with a relative efficiency of 35% and a resolution (FWHM) of 1.93keV at 1332.5 keV gamma ray line, the MAESTRO emulation software coupled to multi-channel

analyzer and associated electronic modules all made by EG&G ORTEC and a personal computer (Jonah *et al.*, 2006). The spectra acquired were processed with the gamma ray spectrum analysis software WINSPAN 2004, and the results obtained in mg/kg (Jonah *et al.*, 2006).

3.2.2 Pre-concentration Neutron Activation Analysis

0.5g of dried and homogenized food sample were accurately weighed into polytetrafluoroethylene (PTFE) pressure digestion cup containing 7mL ultra-pure 65% nitric acid. The digestion cup was securely fasten and the sample digested under pressure in a closed system microwave oven at 1000W for two minutes. After digestion, the system was cooled to room temperature and further cooled in ice-bath for one hour. The clear solution of digested sample was transferred into a beaker containing 1g of hydrazine sulphate, the digestion cup and its lid were washed with 3 portions of 5mL aliquots of 5% hydrazine sulphate solution and deionized water. The digested sample and the washings were combined and diluted to 100mL. The pH of the solution was adjusted between 2 to 3 with 10% ammonia solution. 1 mL each, of freshly prepared 0.05M bismuth sulphate and 0.25M thioacetamide were used to co-precipitated iodine (Chatt and Rao, 1991). The precipitate was filtered by suction with 0.45µm membrane filter, dried, weighed, and then packaged for irradiation. The samples were then irradiated individually at the outer irradiation channel B4 of NIRR-1 for 600 seconds and counted after a decay period of about 2 minutes and then the nuclide spectrum was obtained by counting the samples for 600 seconds using the 442 keV gamma ray of ^{128}I at a height of 1 cm from the detector (Chattand Rao, 1991; IAEA, 2008). The spectra acquisition and analysis were done with same as INAA (Jonah *et al.*, 2006).



Plate 3.1: Packaged sample with Irradiation Capsule.



Plate 3.2: HPGe detector with spectrum acquisition and analysis computers.

3.2.3 Spectroscopy

3.2.3.1 Alkaline dry ashing

In a nickel crucible, 0.5g of the SRM was added and then 1cm^3 each of 0.5M sodium hydroxide and 0.1M potassium nitrate. The crucible was placed in a Nabertherm furnace, and pre-heated at 250^{o}C for 15 minutes, then at 450^{o}C for another 15 minutes and finally at 580^{o}C for 180 minutes. The samples were cooled overnight, and then extracted four times with 2cm^3 portions of 1 millimolar sodium hydroxide. The extract was made up to 10 cm^3 with deionized water then centrifuged at 2500 rpm for 30 minutes, and then the iodate converted to iodine by the addition of 1g hydrazine sulphate (Fisher *et al*., 1986).

3.2.3.2 Microwave assisted acid digestion

About 1g of sample was accurately weighed and digested in a closed system microwave with 7cm^3 ultra-pure 65% nitric acid. The sample was air cooled in the microwave cavity to about 30^o C, and then further cooled in an ice bath to about 0^o C. The digested sample was diluted to 10 cm^3 with deionized water, and then the iodate formed after digestion was reduced to iodide via iodine by the addition of 1g hydrazine sulphate (Milestone, 2011; Chatt and Rao, 1991).

$$2IO_3^- + 12H^+ + 10e^- \longrightarrow I_2 + 6H_2O$$

$$2I_2 + N_2H_4 \longrightarrow N_2 + 4H^+ + 4I^-$$

1 cm^3 of the digested samples was pippeted into a 10 cm^3 test tube and mixed with 1 cm^{3}of 0.1M Arsenous acid and 1 cm^3 0.1 M sulphuric acid. The test tube was then immersed in a water bath thermostated at 40^{o}C for about 15 minutes or until the temperature of the mixture is 30 ^{o}C. 1 cm^3 of 0.1M ceric ammonium sulfate (which was also prior immersed in the water bath) was then added to the test tube.

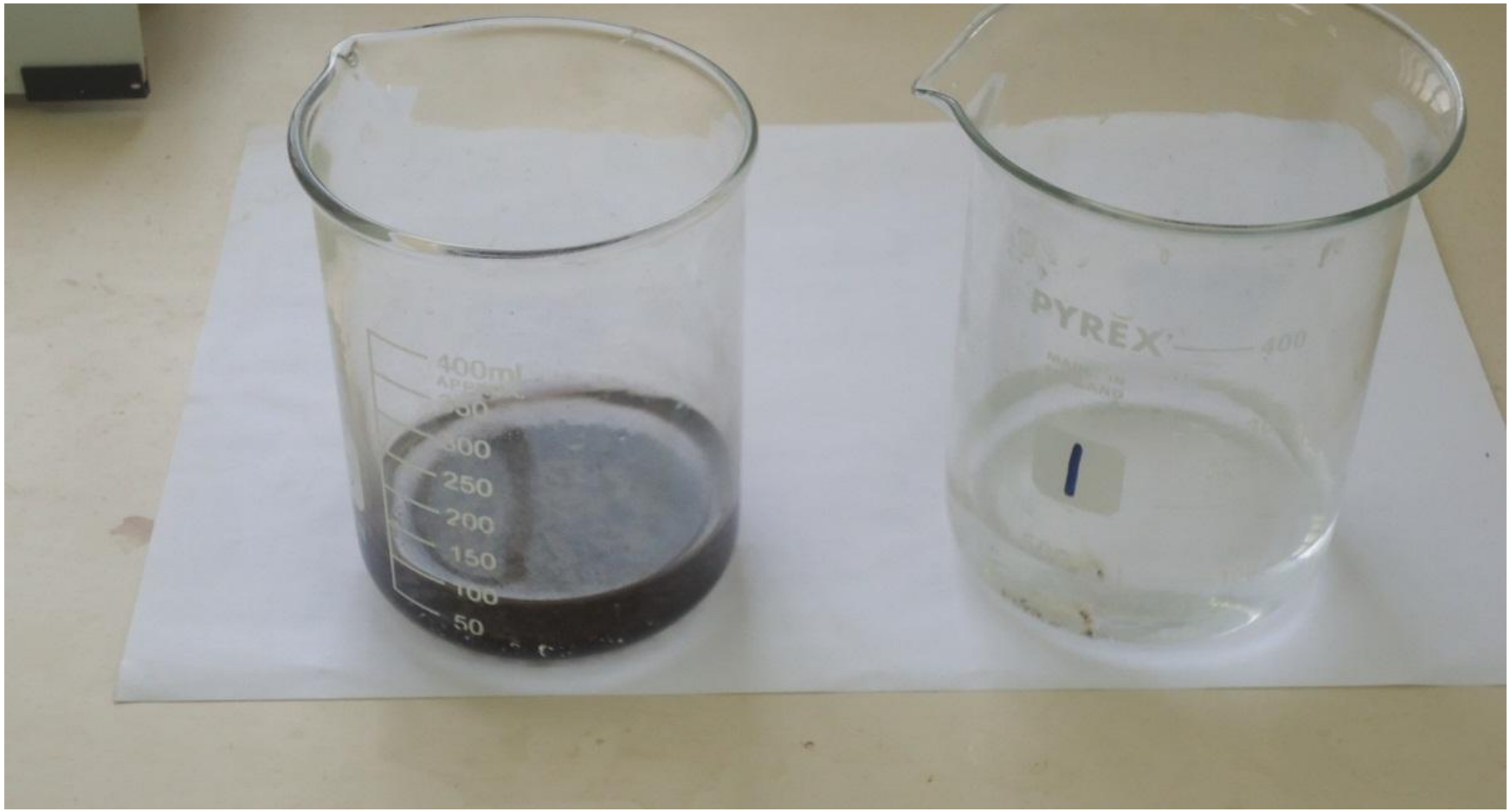


Plate 3.3: BiI_3 precipitate (left beaker) been formed from digested food sample (right beaker).

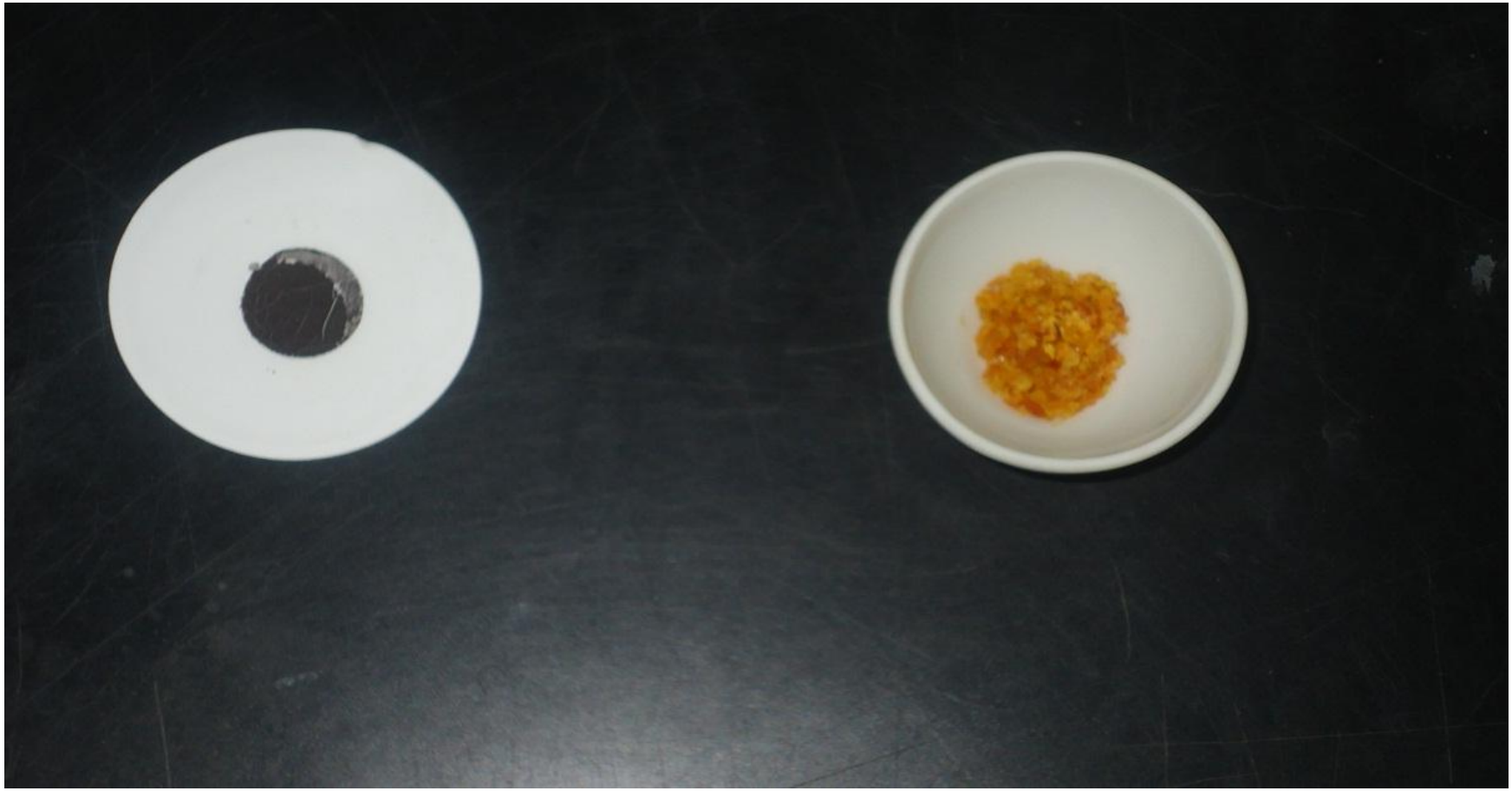


Plate 3.4: BiI_3 on membrane filter after processing from food sample (in crucible)

A timer was started and in exactly two (2) minutes, the content was transferred to a cuvette and after 15 seconds the absorbance was recorded using a UV/ visible GBT Cintra 6 spectrophotometer at 420 nm (Fischer *et al.*, 1986).

A standard curve using potassium iodate solution containing 0.5 to 10 mg/kg (0.5 to 10 ppm) iodine was prepared. The intensity of the developed color was measured at 420 nm. The iodine concentration in the samples was calculated from the standard curve.

Quality control for iodine was maintained by including three reagent blanks to monitor contamination and estimate detection limits. Validation of the analytical techniques with each batch of samples analyzed was tested by the analysis of certified reference materials.

$$2Ce^{4+} + 2I^{-} \longrightarrow 2Ce^{3+} + I_2$$

$$AS^{3+} + I_2 \longrightarrow As^{5+} + 2I^{-}$$

3.2.4 Iodometric Titration

In a conical flask, 10 grams of the salt sample was dissolved in 50 cm^3 of deionized water and then 1g of hydrazine sulphate was added to release the free iodine from the iodate. Exactly 5.0 cm^3 of 10% potassium iodide was then added and the flask stoppered and kept in the dark for ten minutes. After the period, the salt solution was directly titrated against 0.005M sodium thiosulphate until the yellow colour diminished to pale yellow, then, 2 cm^3 of starch indicator solution was added, turning the solution to blue. Titration was continued until the solution becomes colorless (ESPA/CN 109, 1984; CODEX STAN 150, 1985). The titre value was recorded and used to calculate the iodine concentration.

$$2Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2NaI$$

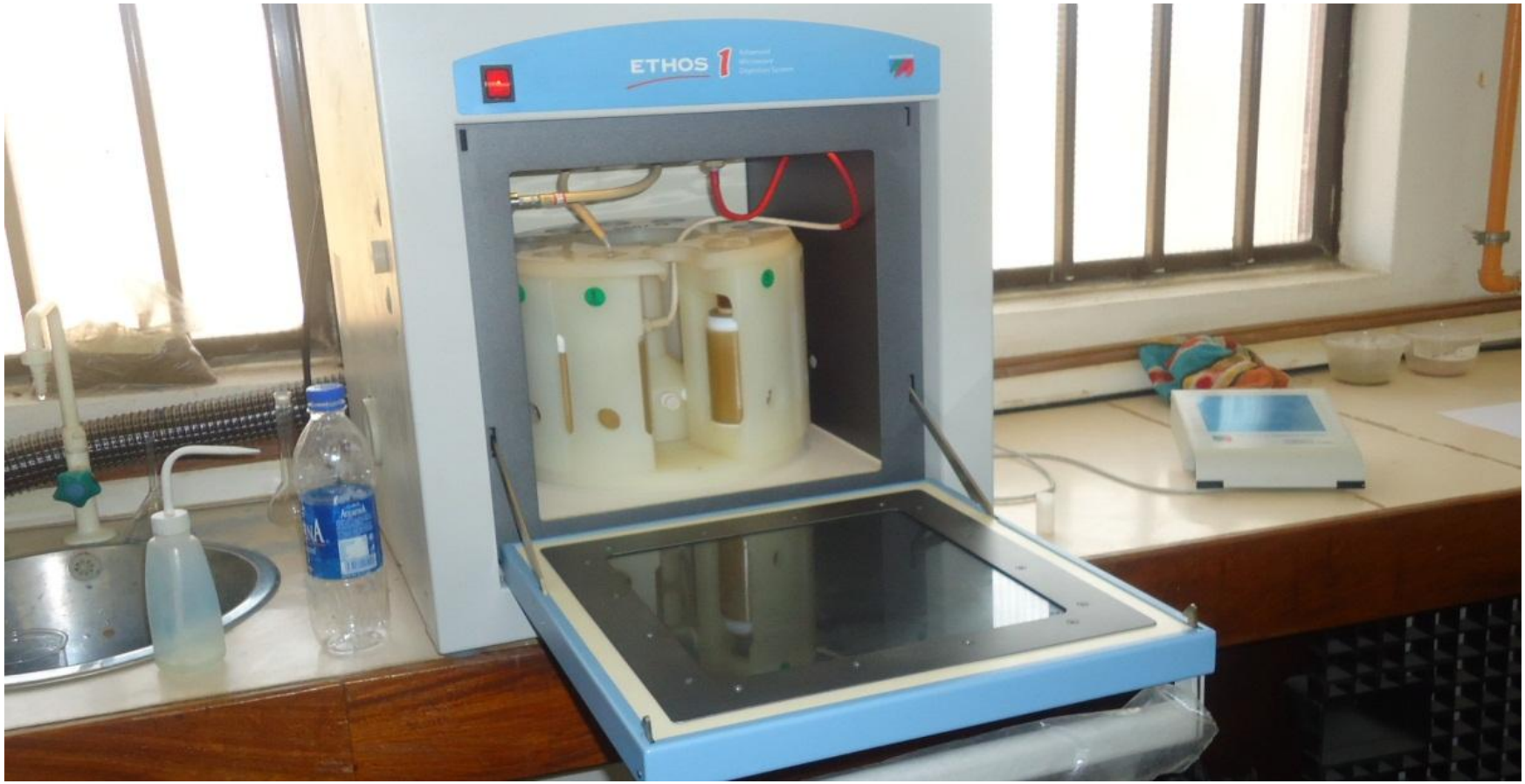


Plate 3.5: Milestone microwave oven packed with the rotor body.



Plate 3.6: Microwave pressure vessel components.



Plate 3.7: GBT Cintra 6 spectrophotometer.

CHAPTER FOUR

4.0 RESULTS

4.1 Recovery Experiments and Calibration

Recovery experiments for both Pre-concentration Neutron Activation Analysis (PCNAA) and Sandell- Kolthof Spectroscopy (S-K spectroscopy) were carried out using standard reference materials (SRM). Table 4.1 compares the obtained iodine concentrations of the SRMs analysed in this work by PCNAA with the certificate values. NIST 1548a (Typical diet) is the only SRM that have certified iodine concentration. Table 4.2 present the comparison of iodine concentration of SRMs obtained in this work by S-K spectroscopy with certificate values. Table 4.3 however, compares the obtained sodium, potassium and bromine concentrations of the SRMs analysed in this work by INAA with the certificate values. The SRM analysed in Table 4.3 are NIST 1573a (Tomato leaves), NIST 1570a (Trace element in spinach leaves), NIST 1515 (Apple leaves), IAEA 359 (Cabbage), and IAEA SL 3 (Lake sediment). Bromine was not reported in NIST 1570a and IAEA 359, and it was not certified in all the reported SRMs except IAEA SL 3.

Table 4.4 present the absorbance at 420nm against prepared standard iodine concentrations of 0, 5, 10, 15 and 20 mg/kg. The data obtained in Table 4.4 was used to plot the calibration curve (Figure 4.1) for S-K spectroscopy. Figure 4.1 have a linear equation of y= -0.0301x + 0.7827 and linear regression of 0.9875. The residuals from the plot reported a standard deviation of residuals of 10.2%

Table 4.5 present the average titre values from iodine titrimetry against prepared standard iodine concentrations of 10, 20, 40, 50 and 100 mg/kg. Figure 4.2 present the calibration curve for iodine titrimetry with a linear equation of y= 0.0971x + 0.0061, a regression of 0.9996 and residuals standard deviation of residuals of 1.8 %

Table 4.1: Quality Assurance result for PCNAA

SRM Duplicate Diet	This work (mg/kg)	Certificate (mg/kg)	% Dev.	t test
NIST 1548a	0.758 ± 0.076	0.759 ± 0.103	0.02	1
NIST 1515	0.298 ± 0.018	0.3	0.33	
NIST 1515	0.298 ± 0.009	0.3	0.33	
NIST 1515	0.300 ± 0.070	0.3	0.00	
Total Diet				
NIST 1515	0.300 ± 0.072	0.3	0.00	
NIST 1548a	0.759 ± 0.055	0.759 ± 0.103	0.00	1
Farm Produce				
NIST 1515	0.330 ± 0.030	0.3	10.0	

Table 4.2: Quality assurance result for SandellKolthof spectroscopy

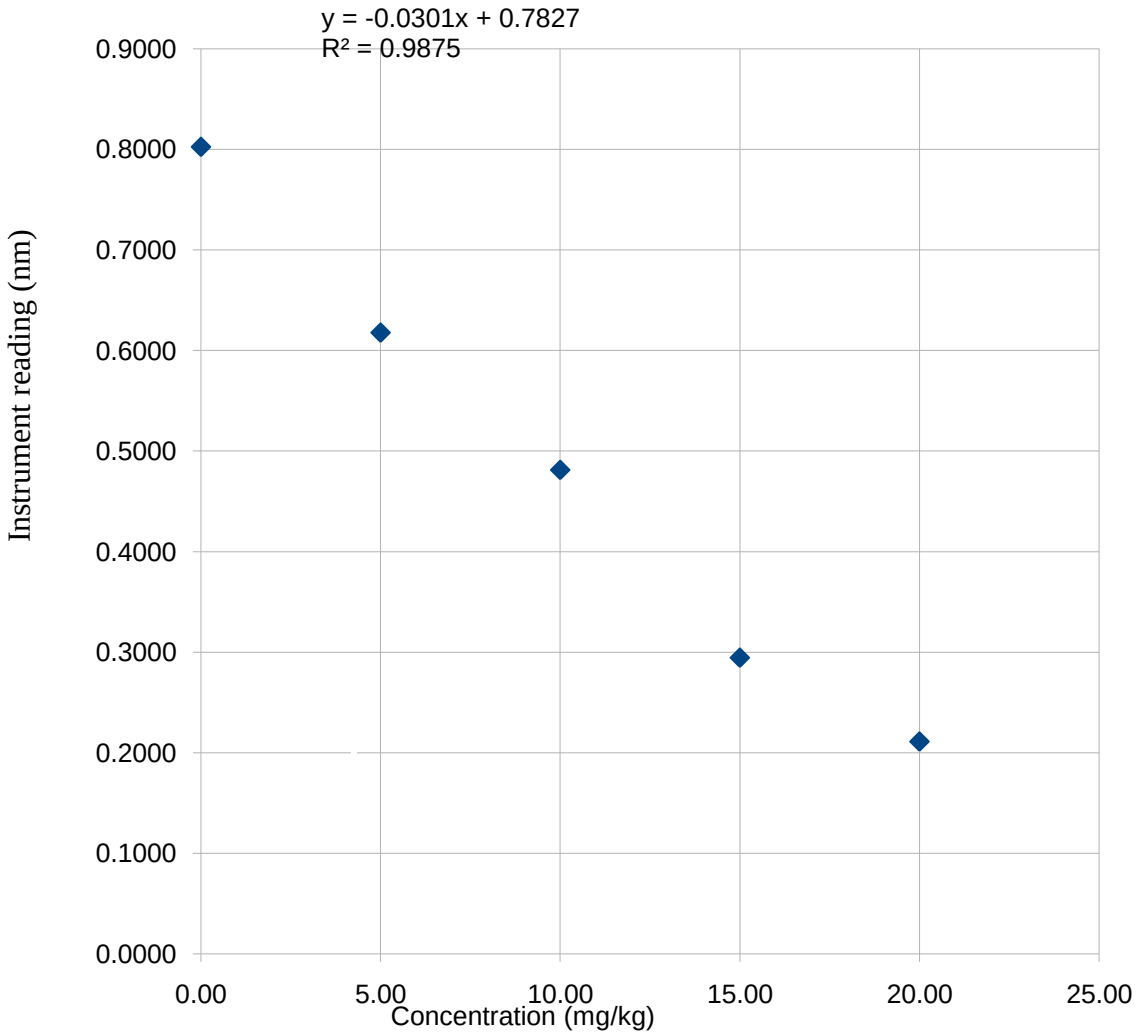
SRM Duplicate Diet	This work (mg/kg)	Certificate (mg/kg)	Percentage deviation (%).	t test
NIST 1548a	0.713 ± 0.044	0.759 ± 0.103	6.06	0.72
NIST 1573a	0.796 ± 0.131	0.85	6.35	
NISTT 1515	0.249 ± 0.056	0.30	17.00	
Total Diet				
NIST 1548a	0.751 ± 0.050	0.759 ± 0.103	1.05	0.95
NIST 1573a	0.851 ± 0.017	0.85	0.12	
NIST 1515	0.286 ± 0.011	0.30	4.67	

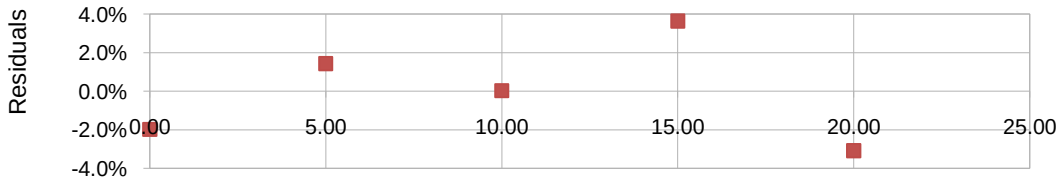
Table 4.3: Quality assurance result for INAA.

	Sodium				Potassium				Bromine			
Duplicate Diet	This Work (mg/kg)	Certified Value (mg/kg)	% Dev.	t test	This Work (mg/kg)	Certified Value (mg/kg)	% Dev.	t test	This Work (mg/kg)	Certified Value (mg/kg)	% Dev.	t test
NIST1573a	132 ± 4	136 ± 4	2.11	0.75	27120 ± 730	27000 ± 5	0.31	0.88	1356 ± 5.78	1300	3.05	
NIST 1573a	94.4 ± 2.6	136 ± 4	25.57	0.01	23530 ± 776	27000 ± 5	9.09	0.05	1368 ± 1.17	1300	3.70	
NIST 1570a	18070 ± 361	18180 ± 4	0.43	0.79	28890 ± 186	29030 ± 5	0.34	0.53	34.01 ± 1.09			
NIST 1515	24.4 ± 0.3	24.4 ± 1.2	0.00	1.00	16100 ± 80	16100 ± 2	0.00	1.00	1.80 ± 0.05	1.8	0.00	
IAEA 359	565 ± 14	584 ± 17	2.33	0.48	31050 ± 214	32500 ± 690	3.15	0.18	5.73 ± 0.12			
Total Diet												
NIST 1515	24.4 ± 0.6	24.4 ± 1.2	0.00	1.00	16100 ± 177	16100 ± 2	0.00	1.00	1.8 ± 0.06	1.8	0.00	
NIST 1573a	148 ± 2	136 ± 4	5.98	0.13	27240 ± 872	27000 ± 5	0.63	0.81	850.1 ± 1.7	1300	24.47	
Farm Produce												
IAEA 359	607 ± 16	584 ± 17	2.78	0.90	32370 ± 206	32500 ± 690	0.28	0.87	6.98 ± 0.14			
NIST 1515	24.1 ± 0.5	24.4 ± 1.2	1.17	0.79	16120 ± 161	16100 ± 2	0.09	0.91	1.850 ± 0.08	1.8	1.96	
Farm Soil												
IAEA SL 3	6696 ± 9	6690 ± 330	0.06	0.99	8565 ± 78	8740 ± 830	1.42	0.86	6.15 ± 1.02	5.6 ± 0.8	7.00	0.71

Table 4.4: Calibration data for Sandell- Kolthoff Spectroscopy

Concentration of standards (mg/kg)	Instrument readings (nm)
00.00	0.8026
05.00	0.6178
10.00	0.4812
15.00	0.2944
20.00	0.2110



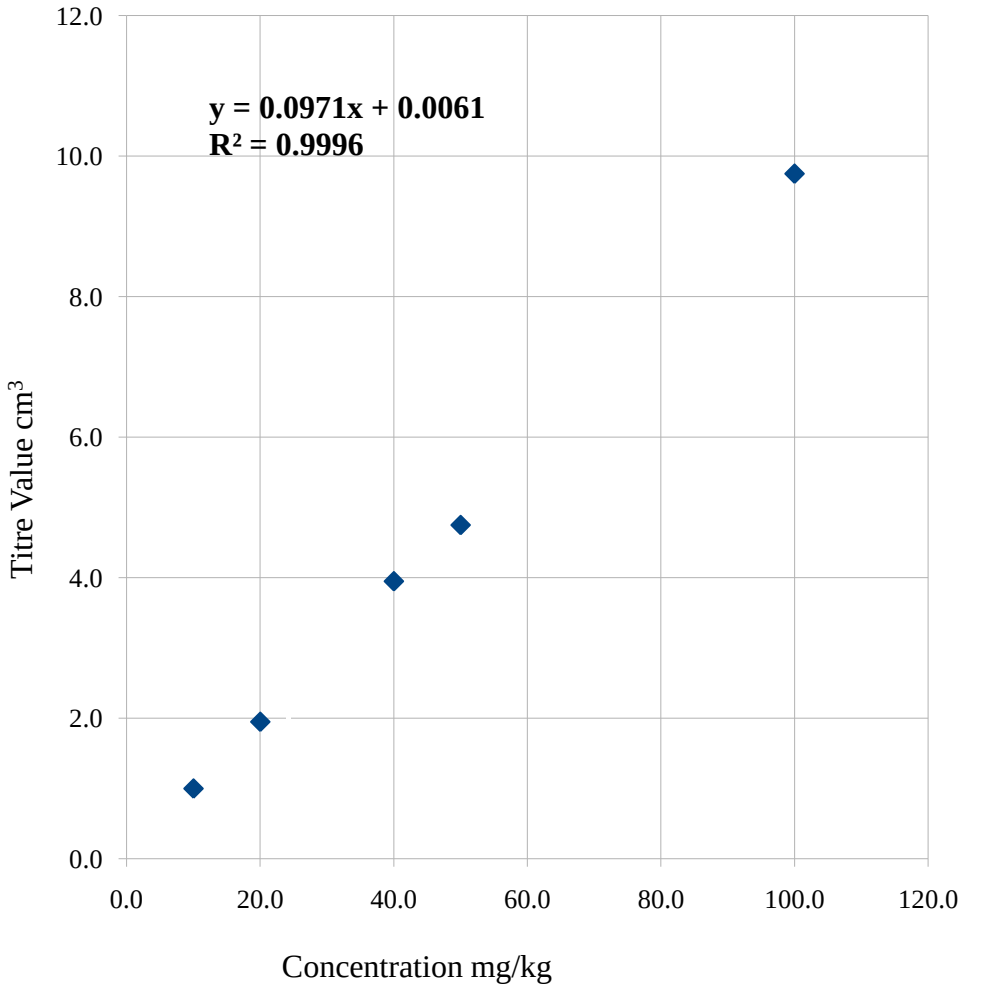


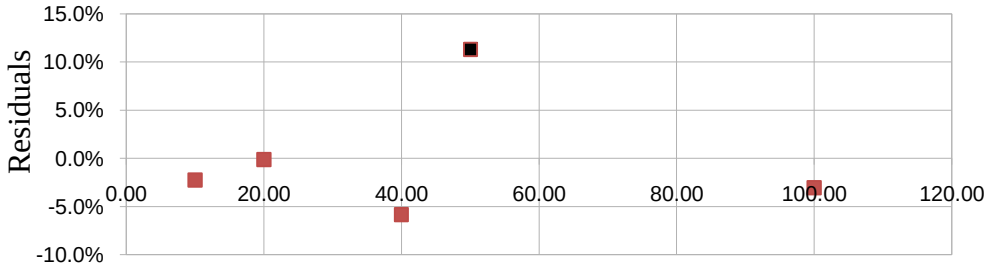
Standard deviation of the residuals = 10.2%

Figure 4.1: The calibration curve and residual values for Sandell-Kolthoff Spectroscopy.

Table 4.5: Calibration curve data for iodine titrimetry

Iodine Concentration (mg /kg)	Titre Value (cm^3)
10	1.00
20	1.95
40	3.95
50	4.75
100	9.75





Standard deviation of the residuals = 1.8%

Figure 4.2: The calibration curve, and residual values for iodine titrimetry.

4.2 Samples Analysis Results

The comparison of the results from PCNAA and S-K spectroscopy methods, in the analysis of duplicate diet samples is presented in table 4.6, while spectroscopy data and PCNAA certificate are presented in Appendix IV and VI respectively. The iodine concentration ranges from 0.03 to 5.51mg/kg with deviation ranging from 1.04 to 92.92%.Table 4.7 compares the total diet samples results from PCNAA and S-K spectroscopy methods, the absorbance recorded for S-K spectroscopy and the certificate from PCNAA analysis are presented in Appendix V and VII respectively. The concentration range is wider but the deviations range is narrower in total diet than duplicate diet at 0.26 to 9.7 mg/kg and 0.04 to 36.83 % respectively. Table 4.8 compares the concentration of sodium, potassium, bromine and iodine in farm soil and the produce from the soil, while the INAA certificate is at Appendix VIII. Potassium have the most concentration in both the soils and the farm produce while iodine is below detection limit in all the soil. Bromine have the highest soil to farm produce ratio ranging from 0.14 to 9.97. INAA result of sodium, potassium and bromine for duplicated diet and total diet samples are presented in tables 4.9 and 4.10 respectively. The concentration ranges in the duplicate and total diet samples are: sodium (2548 to 11040 mg/kg, 1646 to 14280 mg/kg), potassium (1355 to 12510 mg/kg, 3015 to 12150 mg/kg), bromine (2.60 to 24.63 mg/kg, 3.13 to 11.31 mg/kg) respectively. Calculated concentration of sodium, potassium, and iodine using INFOODS data base is tabulated at Table 4.11. Table 4.12 presents the salt iodine concentration, while the titre values are at Appendix IX. The iodine concentration in the salt samples ranges from 14.87 to 73.55 mg/kg with standard deviation between titre runs ranging from 0.0 to 1.03.Finally, the summary of dietary assessment questionnaire is presented at Table 4.13.

Table 4.6: S-K Spectroscopy and PCNAA result, duplicate diet

		SANDEL-KOLTOFF			PCNAA	
S/N	Code	Iodine (mg/Kg)	I in food (µg)	Percentage Deviation	Iodine (mg/Kg)	I in food (µg)
1	HDM01-1a	0.16 ± 0.01	23.96	2.5	0.16 ± 0.02	23.37
2	HDM01-2a	0.88 ± 0.05	149.69	1.6	0.89 ± 0.20	152.08
3	HDM01-3a	0.04 ± 0.01	3.73	68.7	0.14 ± 0.04	11.9
4	HDM01-1b	1.05 ± 0.05	217.01	4.4	1.10 ± 0.09	226.91
5	HDM01-2b	0.03 ± 0.02	6.70	-	BDL	BDL
6	HDM01-3b	2.28 ± 0.08	212.54	18.5	1.92 ± 0.08	179.29
7	HDM02-1	1.09 ± 0.07	109.45	20.9	1.38 ± 0.34	138.31
8	HDM02-2	0.04 ± 0.01	6.56	-	BDL	BDL
9	HDM02-3	0.38 ± 0.05	78.92	8.0	0.41 ± 0.04	85.83
10	HDM03-1a	0.22 ± 0 .03	15.38	77.8	0.98 ± 0.07	69.15
11	HDM03-2a	0.50 ± 0.09	57.42	0.8	0.50 ± 0.03	57.89
12	HDM03-3a	0.84 ± 0.05	119.11	5.3	0.89 ± 0.01	125.75
13	HDM03-1b	0.33 ± 0.05	71.12	2.4	0.34 ± 0.08	72.83
14	HDM03-2b	0.20 ± 0.02	22.47	75.5	0.82 ± 0.01	91.67
15	HDM03-3b	0.96 ± 0.06	107.47	4.6	1.01 ± 0.13	112.6
16	HDM04-1	0.53 ± 0.23	114.02	10.7	0.59 ± 0.06	127.65
17	HDM04-2	0.11 ± 0.02	31.19	-	BDL	BDL
18	HDM04-3	0.51 ± 0.04	134.09	74.4	1.99 ± 0.09	524.24
19	HDM05-1	2.59 ± 0.02	240.75	38.5	1.87 ± 0.25	173.82
20	HDM05-2	0.42 ± 0.01	544.56	32.8	1.36 ± 0.13	810.29
21	HDM05-3	0.80 ± 0.00	171.69	5.2	0.84 ± 0.23	181.18
22	HDM06-1	1.05 ± 0.02	334.09	1.9	1.03 ± 0.08	327.72
23	HDM06-2	0.18 ± 0.08	42.14	88.7	1.56 ± 0.02	373.53
24	HDM06-3	0.38 ± 0.01	62.82	72.4	1.38 ± 0.05	227.54
25	HDM07-1	0.32 ± 0.06	53.27	84.5	2.04 ± 0.11	342.83
26	HDM07-2	0.08 ± 0.03	21.37	92.9	1.13 ± 0.08	301.92
27	HDM07-3	1.52 ± 0.03	153.34	4.6	1.45 ± 0.10	146.67
28	HDL08-1	0.76 ± 0.01	157.91	1.0	0.77 ± 0.05	159.57
29	HDL08-2	0.83 ± 0.04	127.52	17.2	1.00 ± 0.10	154.01
30	HDL08-3	0.05 ± 0.02	7.37	-	BDL	BDL
31	HDL09-1	0.66 ± 0.02	108.00	1.1	0.65 ± 0.18	106.85
32	HDL09-2	1.34 ± 0.08	28.92	4.3	1.28 ± 0.06	27.72
33	HDL09-3	1.06 ± 0.03	254.07	24.2	1.4 ± 0.19	335.25
34	HDL10-1	5.25 ± 0.09	266.28	53.9	1.12 ± 0.03	577.98
36	HDL10-3	5.51 ± 0.22	273.10	49.4	1.09 ± 0.02	539.27
37	HDL11-1	0.34 ± 0.03	47.88	6.3	0.32 ± 0.09	45.07
38	HDL11-2	0.93± 0.05	138.62	3.6	0.96 ± 0.08	143.87
39	HDL11-3	0.03 ± 0.02	6.64	-	BDL	BDL
40	HDL12-1	1.40 ± 0.05	414.68	26.5	1.44 ± 0.18	327.85
41	HDL12-2	0.46 ± 0.03	117.95	10.4	0.51 ± 0.05	131.63
42	HDL12-3	Meal Skipped				

BDL= Below Detection Limit

Table 4.7: S-K Spectroscopy and PCNAA result, total diet

		S-K METHOD			PCNAA	
S/N	Sample	Iodine (mg/Kg)	Iodine in food (µg)	Percentage deviation	Iodine (mg/Kg)	Iodine in food (µg)
1	L 1-1	0.26 ± 0.02	55.54	10.51	0.30 ± 0.05	62.06
2	L 1-2	1.19 ± 0.03	74.80	36.83	1.89 ± 0.08	118.41
3	L 1-3	7.94 ± 0.29	1685.98	18.01	9.60 ± 0.16	2056.23
4	L 2-1	0.63 ± 0.11	133.49	15.01	0.75 ± 0.06	157.07
5	L 2-2	1.25 ± 0.06	288.11	6.35	1.34 ± 0.08	307.64
6	L 2-3	BDL	BDL		BDL	BDL
7	L 3-1	0.78 ± 0.02	120.92	3.85	0.81 ± 0.10	125.75
8	L 3-2	1.12 ± 0.02	251.92	3.78	1.16 ± 0.08	261.82
9	L 3-3	1.09 ± 0.02	233.38	36.16	0.78 ± 0.06	171.40
10	M 1-1	1.16 ± 0.03	290.03	15.11	1.36 ± 0.07	341.66
11	M 1-2	1.55 ± 0.02	260.04	3.78	1.61 ± 0.08	270.26
12	M 1-3	1.22 ± 0.02	408.73	11.76	1.40 ± 0.07	463.20
13	M 2-1	1.24 ± 0.02	317.83	4.41	1.29 ± 0.07	332.48
14	M 2-2	0.94 ± 0.03	177.44	5.38	0.89 ± 0.09	168.39
15	M 2-3	1.16 ± 0.02	159.57	2.58	1.13 ± 0.22	155.57
16	M 3-1	0.45 ± 0.03	114.58	18.84	0.55 ± 0.07	141.17
17	M 3-2	1.28 ± 0.01	163.05	2.44	1.31 ± 0.06	167.12
18	M 3-3	2.73 ± 0.15	391.50	7.94	2.96 ± 0.09	425.26
19	H 1-1	1.14 ± 0.09	186.41	2.89	1.18 ± 0.07	191.96
20	H 1-2	0.27 ± 0.02	72.42	34.53	0.42 ± 0.04	110.63
21	H 1-3	1.93 ± 0.04	517.52	7.89	2.09 ± 0.08	561.88
22	H 2-2	2.44 ± 0.03	452.95	0.04	2.44 ± 0.08	453.13
23	H 2-3	1.26 ± 0.02	367.52	1.41	1.27 ± 0.04	372.79

BDL= Below Detection Limit

Table 4.8: Elemental analysis of farm soil and produce

Sample	Element	Farm Soil (mg/kg)	Farm produce (mg/kg)	Soil- Produce ratio
Beans 1-2a	Na	1438 ± 2.9	26.61 ± 0.40	0.02
	K	18090 ± 127	11780 ± 118	0.65
	Br	1.21 ± 0.13	1.12± 0.04	0.93
	I	BDL	0.32± 0.07	0.00
Wheat 1-3b	Na	2292 ± 4.58	33.53 ± 0.40	0.01
	K	31330 ± 282	4443 ± 75.5	0.14
	Br	6.19 ± 0.88	0.84± 0.03	0.14
	I	BDL	0.31± 0.08	0.00
Rice 4-1;2	Na	515.7 ± 1.5	31.30 ± 0.30	0.06
	K	8732± 78.6	1928 ± 30.9	0.22
	Br	3.52 ± 0.77	35.09 ± 2.28	9.97
	I	BDL	0.29 ± 0.05	0.00
Spinach 5-3	Na	2066± 4.1	435.9 ± 1.3	0.21
	K	21330 ± 235	38540 ± 231	1.81
	Br	2.57 ± 0.19	4.39± 0.11	1.71
	I	BDL	1.27± 0.10	0.00
Maize 8-3	Na	1797± 3.6	15 ± 0.20	0.01
	K	25610 ± 153	1334 ± 26	0.05
	Br	0.39 ± 0.10	0.42 ± 0.02	1.08
	I	BDL	0.22 ± 0.08	0.00

BDL= Below Detection Limit

Table 4.9: INAA Result, Duplicate Diet.

S/N	Code	Na (mg/kg)	K (mg/kg)	Br (mg/kg)
1	HDM01-1a	7139 ± 7.1	12510 ± 512	5.04 ± 0.27
2	HDM01-2a	7472 ± 7.5	1500 ± 321	4.39 ± 0.18
3	HDM01-3a	8110 ± 8.1	5241 ± 446	4.49 ± 0.26
4	HDM01-1b	4109 ± 8.2	10840 ± 357	2.88 ± 0.19
5	HDM01-2b	3943 ± 7.9	2904 ± 342	2.60 ± 0.18
6	HDM01-3b	4378 ± 8.8	5075 ± 345	9.72 ± 0.26
7	HDM02-1	5790 ± 11.6	2202 ± 244	8.37 ± 0.26
8	HDM02-2	6393 ± 6.4	2628 ± 315	4.41 ± 0.23
9	HDM02-3	4863 ± 9.7	3041 ± 225	3.84 ± 0.23
10	HDM03-1a	3354 ± 6.7	2935 ± 264	24.63 ± 0.30
11	HDM03-2a	6590 ± 13.2	3037 ± 340	4.06 ± 0.22
12	HDM03-3a	6770 ± 13.5	1821 ± 304	8.04 ± 0.19
13	HDM03-1b	5085 ± 10.2	1355 ± 262	4.87 ± 0.16
14	HDM03-2b	7351 ± 7.6	2598 ± 351	9.01 ± 0.39
15	HDM03-3b	5999 ± 12	2871 ± 376	3.95 ± 0.23
16	HDM04-1	8112 ± 16.2	11370 ± 532	7.40 ± 0.29
17	HDM04-2	5254 ± 10.5	2558 ± 378	3.90 ± 0.27
18	HDM04-3	11040 ± 11	4431 ± 516	10.03 ± 0.03
19	HDM05-1	4923 ± 9.8	7659 ± 413	3.76 ± 0.22
20	HDM05-2	6363 ± 12.7	3556 ± 380	6.60 ± 0.24
21	HDM05-3	2633 ± 7.9	3112 ± 410	10.60 ± 0.21
22	HDM06-1	2643 ± 2.6	7453 ± 127	3.91 ± 0.20
23	HDM06-2	4495 ± 8.9	1502 ± 362	4.04 ± 0.23
24	HDM06-3	5582 ± 16.7	4373 ± 581	4.50 ± 0.23
25	HDM07-1	5380 ± 10.8	1300 ± 231	4.56 ± 0.22
26	HDM07-2	5134 ± 10.3	2326 ± 249	9.03 ± 0.25
27	HDM07-3	7626 ± 7.6	4010 ± 381	3.71 ± 0.30
28	HDL08-1	4889 ± 6.6	6511 ± 206	6.86 ± 0.29
29	HDL08-2	2934 ± 2.7	2115 ± 134	12.03 ± 0.18
30	HDL08-3	7706 ± 7.7	4340 ± 399	6.24 ± 0.33
31	HDL09-1	5594 ± 11.2	3652 ± 267	4.50 ± 0.26
32	HDL09-2	7264 ± 7.3	4229 ± 389	4.74 ± 0.28
33	HDL09-3	4872 ±6.9	3511 ±208	9.14 ± 0.11
34	HDL10-1	6922 ± 7.0	4824 ±416	4.96 ± 0.29
35	HDL10-2		Meal Skipped	
36	HDL10-3	6975 ± 7.0	4832 ± 425	4.95 ± 0.29
37	HDL11-1	4756 ± 8.9	4015 ± 311	4.32 ± 0.23
38	HDL11-2	2548 ± 10	4957 ± 186	4.15 ± 0.22
39	HDL11-3	7591 ± 0.76	2263 ± 321	7.98 ± 0.29
40	HDL12-1	4786 ± 9.6	3181 ± 280	10.31 ± 0.18
41	HDL12-2	4994 ± 10	4611 ± 314	8.72 ± 0.17
42	HDL12-3		Meal Skipped	

Table 4.10: INAA Result, Total Diet.

S/N	Code	Na (mg/kg)	K (mg/kg)	Br (mg/kg)
1	RL 1-1	3086 ± 6.2	5180 ± 171	3.13 ± 0.15
2	RL 1-2	5078 ± 8.72	3115 ± 278	8.52 ± 0.35
3	RL 1-3	14280 ± 14	4273 ± 641	6.80 ± 0.20
4	RL 2-1	1646 ± 3.3	10020 ± 170	4.35 ± 0.14
5	RL 2-2	4470 ± 4.5	3279 ± 207	4.01 ± 0.15
6	RL 2-3	9264 ± 9.3	4655 ± 101	6.21 ± 0.26
7	RL 3-1	4439 ± 6.8	5194 ± 251	7.52 ± 0.54
8	RL 3-2	8282 ± 16.4	5163 ± 465	4.06
9	RL 3-3	9449 ± 18.9	4901 ± 529	4.29 ± 0.28
10	ML 1-1	5983 ± 12.0	11950 ± 418	5.89 ± 0.21
11	ML 1-2	11070 ± 11.1	3251 ± 422	8.39 ± 0.32
12	ML 1-3	5783 ± 11.9	4547 ± 496	5.06 ± 0.024
13	ML 2-1	8356 ± 8.4	3015 ± 404	11.31 ± 0.36
14	ML 2-2	4692 ± 8.4	3214 ± 216	3.26 ± 0.18
15	ML 2-3	7591 ± 7.8	3698 ± 412	4.36 ± 0.21
16	ML 3-1	6325 ± 12.7	10190 ± 408	5.75 ± 0.27
17	ML 3-2	7582 ± 13.4	5648 ± 407	4.98 ± 0.16
18	ML 3-3	7797 ± 15.6	5536 ± 493	4.25 ± 0.27
19	HL 1-1	8014 ± 12.7	11256 ± 517	4.53 ± 0.23
20	HL 1-2	9316 ± 28.0	12150 ± 421	5.94 ± 0.32
21	HL 1-3	7967 ± 15.9	9369 ± 420	8.32 ± 0.83
22	HL 2-1		Not available	
23	HL 2-2	11850 ± 23.7	5135 ± 611	5.74 ± 0.26
24	HL 2-3	6310 ± 6.5	4179 ± 418	4.88 ± 0.25

Table 4.11: Total diet replica from INFOODS data base

S/N	Code	Na (mg/kg)	K (mg/kg)	I (mg/kg)
1	RL 1-1	120	4252	NA
2	RL 1-2	115	1576	NA
3	RL 1-3	75	2826	NA
4	RL 2-1	192	4420	NA
5	RL 2-2	62	3385	NA
6	RL 2-3	1063	6064	NA
7	RL 3-1	28	1159	NA
8	RL 3-2	539	1593	NA
9	RL 3-3	109	4077	NA
10	ML 1-1	667	3376	NA
11	ML 1-2	237	1455	0.10
12	ML 1-3	1165	7315	0.19
13	ML 2-1	672	956	NA
14	ML 2-2	257	1788	NA
15	ML 2-3	667	4817	NA
16	ML 3-1	1744	6584	1.35
17	ML 3-2	195	1609	1.06
18	ML 3-3	892	6444	NA
19	HL 1-1	615	3110	NA
20	HL 1-2	1646	11889	NA
21	HL 1-3	1248	8216	NA
23	HL 2-2	307	1979	NA
24	HL 2-3	1069	6710	NA

NA= Not Available

Table 4.12: Iodine titre values and concentration

S/N	Sample	Average Titre Value (cm^3)	Iodine Conc. (mg/kg)	Standard deviation
1	HDM1a	1.90	19.50	0.51
2	HDM1b	7.27	74.78	0.59
3	HDM2	2.47	25.37	0.30
4	HDM3a	3.62	37.21	0.30
5	HDM3b	3.27	33.60	0.30
6	HDM4	4.70	48.32	1.03
7	HDM5	7.15	73.55	0.51
8	HDM6	6.37	65.52	0.30
9	HDM7	4.80	49.35	0.51
10	HDL8	0.00	BDL	0.00
11	HDL9	4.02	41.32	0.79
12	HDL10	6.08	62.53	0.30
13	HDL11	1.45	14.87	0.51
14	HDL12	3.95	40.60	0.30
15	Smr a	4.55	46.78	0.51
16	Smr b	5.55	57.07	0.51
17	Smr c	6.55	67.37	0.51
18	Sabo G. a	6.10	62.74	1.03
19	Sabo G. b	5.05	51.93	0.51
20	Sabo G. c	5.52	56.77	0.79
21	City a	4.00	41.12	0.51
22	City b	9.07	93.31	0.59
23	City c	7.00	72.00	0.89

BDL= Below Detection Limit

Table 4.13: Dietary assessment questionnaire summary.

Location		G. R. A.	Sabongari	Samaru	Zaria city
No. of Respondants		33	57	109	101
Income Status	Low	6.06%	26.32%	29.78%	22.91%
	Average	66.67%	50.88%	67.56%	58.31%
	High	22.27%	22.80%	12.60%	19.78%
Food Sources	Self-Cultivated	10.20%	18.40%	28.62%	46.39%
	Raw Purchase	59.20%	54.60%	33.46%	41.06%
	Restaurant	30.60%	28.22%	36.80%	11.79%
Avr. Meals/ Day		2.997	2.86	2.47	2.60
Breaks Fast	1	W1 (39.39)	W1(48.21)	W1 (33.33)	W1 (23.96)
(Percentage)	2	R4 (24.24)	B4 (5.36)	B1 19.05	B2 (16.67)
	3	P1 (15.15)	P1 (5.36)	M1 (7.62)	B1 (14.58)
	4	B1 (9.09	R4 (5.36)	P1 (4.76)	R4 (10.42)
	5	Y1 (6.06)	M1 (5.36)	Y2 (1.90)	M1 (8.33)
Lunch	1	R1 (37.5)	R5 (35.19)	R5 (29.47)	R5 (27.91)
(Percentage)	2	R5 (25)	R1 (22.22)	R1 (18.95)	R1 (12.79)
	3	R3 (12.5)	R2 (7.41)	M1 (7.37)	R3 (6.98)
	4	M1 6.25)	R3 (5.56)	R3 (5.26)	B2 6.98)
	5	Y5 (6.25)	B1 (3.70)	W7 (4.21)	W5 (6.98)
Dinner	1	M1 (27.3)	M1(53.57)	M1 (34.69)	M1 (56.52)
(Percentage)	2	MS 1 (33.3)	R3 (8.99)	MS1(10.20)	MS1 (7.61)
	3	R4 (12.12)	R1 (5.36)	R1 (6.12)	W1 (5.43)
	4	R2 (6.06)	Y5 (3.57)	R3 (6.12)	Y5 (5.43)
	5		W2 (3.57)	R4 (4.08)	R3 (3.26)
Water Source	Tap	39.39%	12.07%	6.25%	9.18%
(Percentage)	Bottled	51.52%	10.34%	13.54%	53.06%
	Sachet	9.09%	53.45%	57.29%	9.18%
	Well	0%	24.14%	22.92%	28.57%
Avr. Water litres consumed/ day		2.83	3.16	2.93	2.88

CHAPTER FIVE

5.0 DISCUSSIONS

5.1 Quality Assurance

The employment of multiple standard reference materials brought confidence to the analysis as well as providing multiple base for validation and comparison of the results obtained. Tables 4.1 and 4.2 present the obtained results from the PCNAA and spectroscopic analysis of the SRM in this work, and comparing it with certified values. The analyses of the SRMs were done in batches along with the samples. Percentage deviation (% Dev.) and student's t- test (t test) of the SRM verify that the results obtained from this work were significantly the same as the values reported in the certified reference materials. However, comparing the two methods employed for the food analysis, it shows that PCNAA records have better accuracy than the spectroscopic technique. NIST 1548a (Typical diet) reports a student's t-test of 1 across the two sample categories in PCNAA as against 0.72 and 0.92 for duplicate and total diet sample respectively from the spectroscopy. PCNAA also records lower percentage standard deviation (Table 4.1, % Dev.) as against S-K spectroscopy (Table 4.2, %Dev.). Duplicate diet SRMs from Table 4.2 were digested with alkaline ashing while the typical diet SRMs were processed with microwave assisted acid digestion. The obtained results suggest that microwave assisted acid digestion is more reliable in providing accurate result as compared to alkaline ashing because it show less deviation from the certified value. Microwave assisted acid digestion in this study records lower percentage deviation than alkaline ashing for the same SRMs, NIST 1548a (typical diet), NIST 1573a (tomato leaves) and NIST 1515 (apple leaves) in the order of 6.67%, 15.25% and 51.67 % respectively. The variation could be attributed to the infinitesimal concentration of iodine, and that the iodine

could have been depleted by adsorption on the walls of the crucible when scorched owning to high temperature, while the PTFE material for the microwave vessel is non-scorching and non-absorbing. Generally, the SRM analysis results compare favorably to the certified values. Only NIST 1515 (Table 4.2) have percentage deviation greater than 10.

Several NIST reference materials of agricultural and food materials, with one of geological material were analysed by INAA along with samples for quality control purposes. The SRM NIST 1573a (Tomato leaves) is the only SRM that recorded deviation larger than 5%. Table 4.3 shows that the results obtained are in agreement with the uncertainty margins with NIST certified values whenever available, thus proving the accuracy of the results obtained in this work.

5.2 Food Samples

The results for PCNAA and SandellKolthoff spectroscopy of duplicate diet samples as presented in Table 4.6 revealed a relatively poor correlation at 0.53. Results from the two method reported that 23.8% of the samples have deviations greater than 50% while 42.86% of the samples have deviation less than 10% and only 30.95 % of the samples agreed with less than 5% deviation as shown in Figure 5.1. However, the PCNAA and SandellKolthoff spectroscopy from total diet as presented in Table 4.7 have a positive correlation at 0.89. No result shows more than 37% deviation, 66.67% of the results have less than 10% deviation while 43.48% of the results have deviation at less than 5% as shown in Figure 5.2.

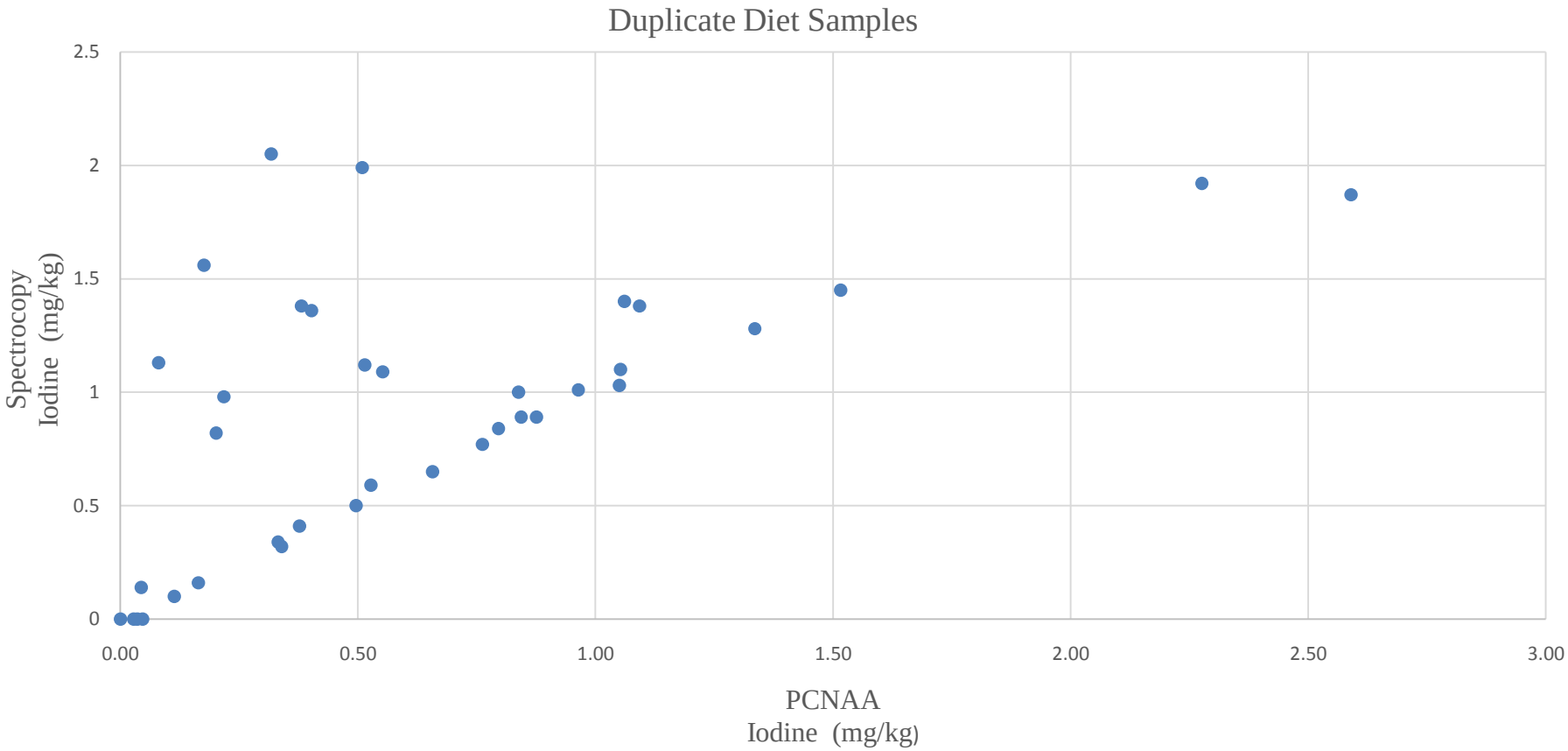


Figure 5.1: Cluster points of Sandell- Kolthof Spectroscopy against PCNAA of duplicate diet samples analysis.

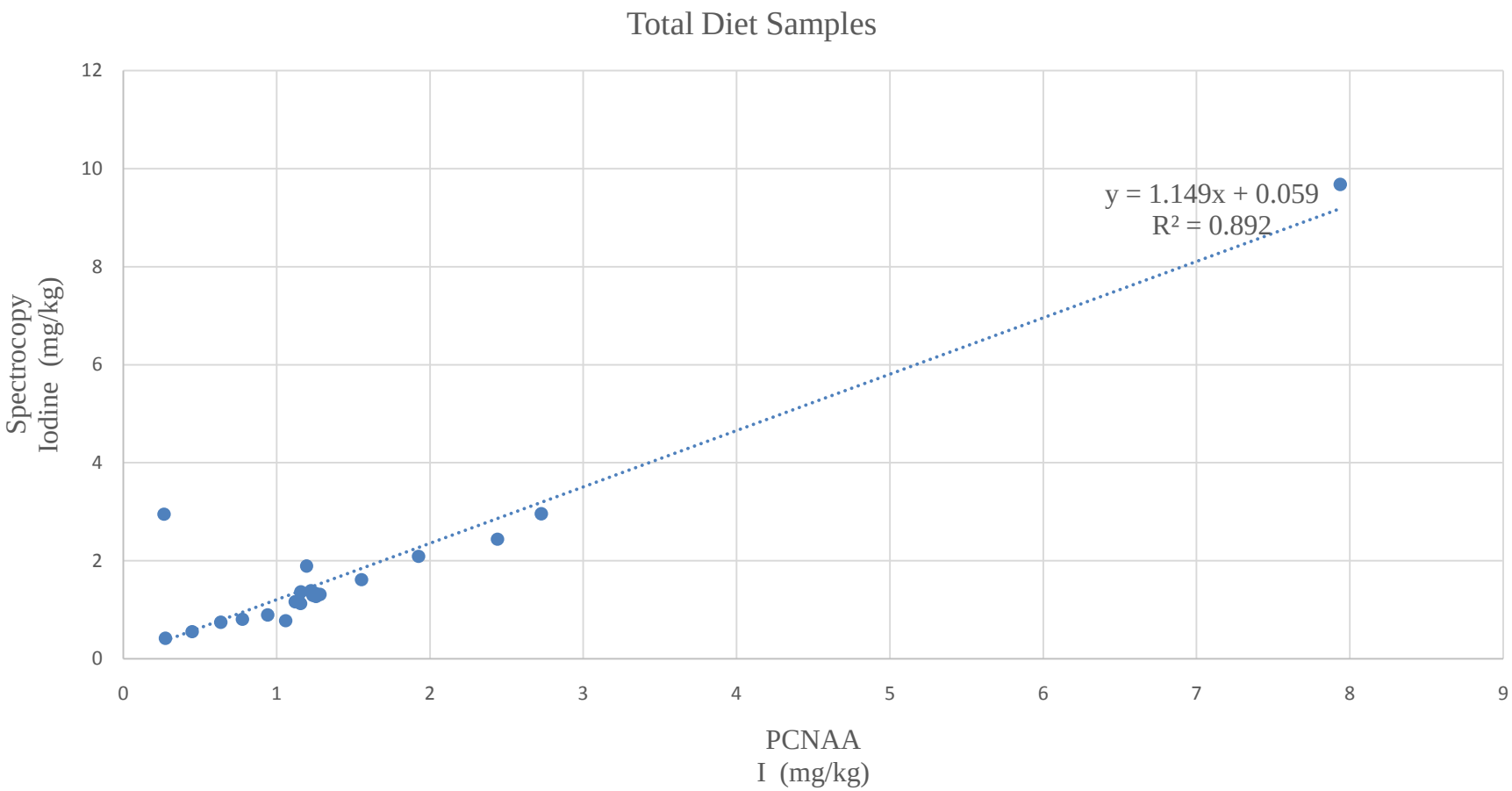


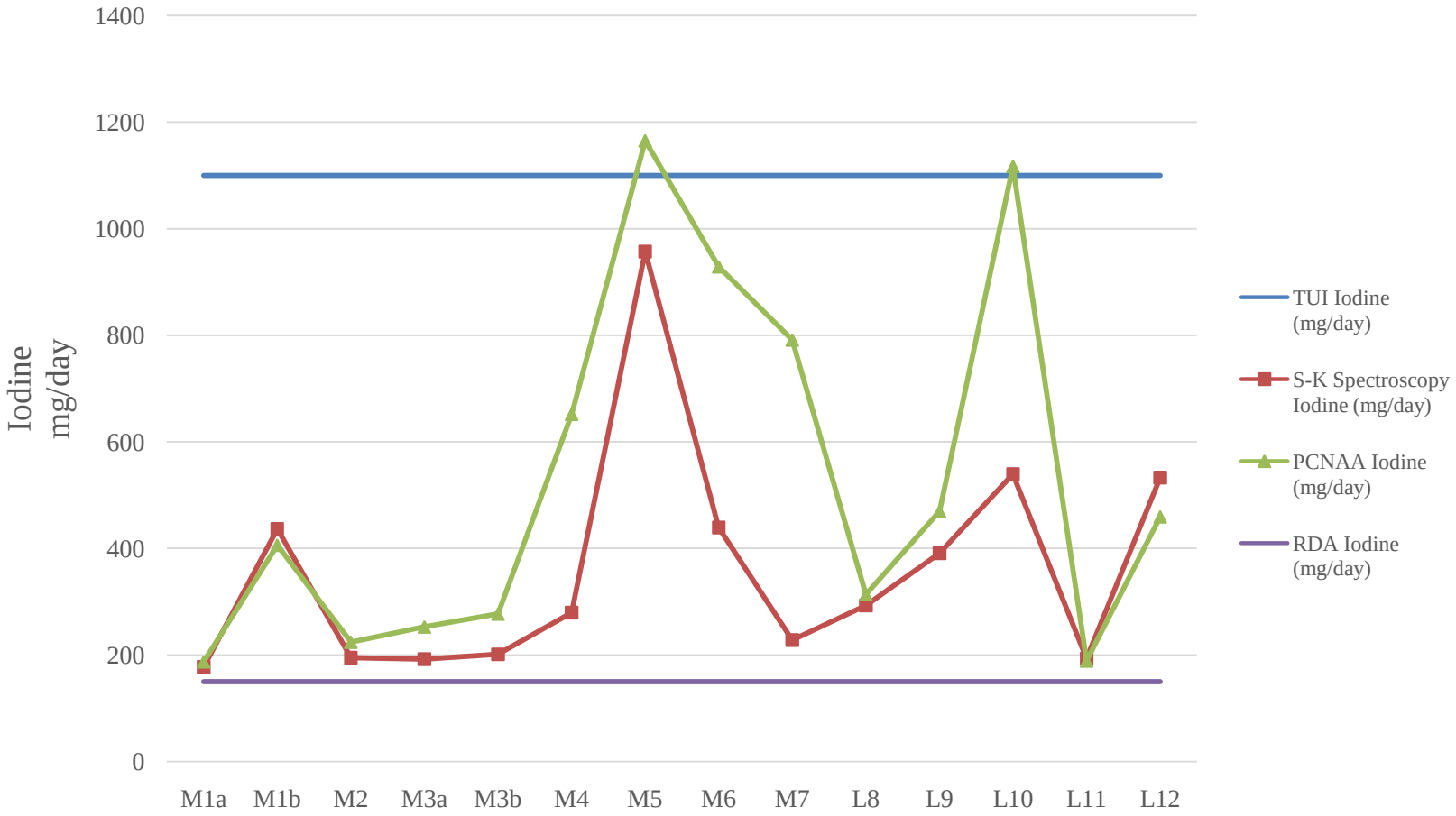
Figure 5.2: Cluster points of Sandell- Kolthof Spectroscopy against PCNAA of total diet samples analysis.

The large differences between the alkaline dry ashed (Table 4.6, Figure 5.1) and acid microwave assist digested (Table 4.7, Figure 5.2) samples can be attributed to loss of significant amount of iodine during various digestion processes as pointed out earlier at the quality assurance. This is in agreement with the work of Welna*et al.*, (2011), which also reported that lack of loss of volatile elements is one of the advantages of microwave digestion.

The recommended daily allowance (RDA) for iodine as well as the tolerable upper intake level for various age groups is presented in Table 1.1. Considering the result from Table 4.6, the RDA for all age groups was achieved. Samples from volunteers number 4,5,6,7 and 10 contains iodine that are more than double the RDA (Figure 5.3.

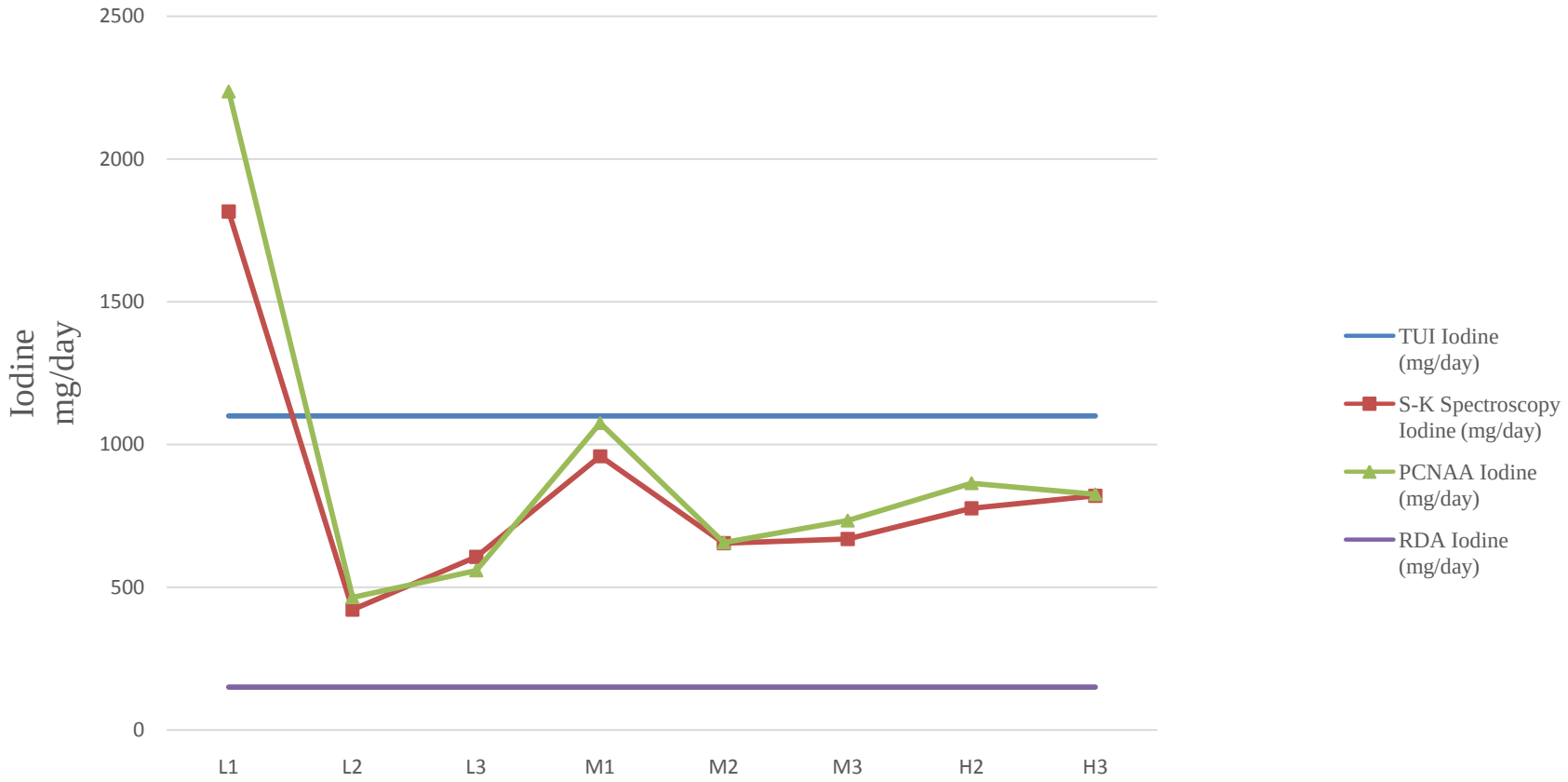
As it was observed during sampling, the practice in the area of study was that children are mostly fed with same food as adults, as such, there is a possibility of approaching upper intake levels especially in children under 8 year of age (although the sampling was based on adults consumption). Samples from volunteer no 5 and 10 have especially high iodine contents and prolonged consumption at such elevated concentration may not be suitable even for adults. The total diet samples also provide the RDA, and similar to the duplicate diet, the daily intake approaches the upper tolerable intake level. Sample RL 1-3 contains more than twice the upper tolerable intake level.

It has been reported by Brittmarie (1998) that in some individuals, intake of iodine as little as 300µg per day can result in iodine induced hyperthyroidism. Thus, it is critical to monitor iodine intake especially in children foods. Total diet samples all supplied iodine in excess of 300µg per day as shown in figure 5.4.



M= Moderate income volunteer; L= Low income volunteer

Figure 5.3: Duplicate diet daily iodine consumption



L= Low priced food, M= Moderate priced food, H= High priced food

Figure 5.4: Total diet daily iodine consumption.

Correlations of sodium and potassium of both duplicate diet and total diet samples (Tables 4.8 and 4.9) were -0.062 and -0.36 respectively while the correlation coefficient observed in bromine and iodine were -0.041 and 0.095 respectively. However, the corresponding correlations of sodium and iodine for both duplicate diet and total diet samples were -0.266 and 0.54 respectively. An intrinsic property of biological matrix is that, there is a strong positive correlation between sodium and potassium and also between bromine and iodine. The above correlation results indicate that there is a disturbance of the natural system, which is the addition of iodized salt to the food samples, hence the moderate positive correlation between sodium and iodine. Further, the correlation of sodium and potassium in foods with similar composition to total diet samples computed from INFOODS (West African Food Composition Table, 2012) database but without addition of salt is 0.83, this further indicates that the large variation between the sodium/ potassium and bromine/ iodine correlations in the samples were due to addition of salt during cooking/ processing. This correlation also indicates that the iodine contents of the food samples were largely from iodized table salt.

It was observed that food made with rice, constitute 7 out of 11 samples reporting deviations greater than 25% between PCNAA and S-K spectroscopy. The other samples (not produced from rice) with deviations greater than 25% are HDM 03-1a and HDM 07-2, produce from wheat, HDM 05-1 made with beans, maize, millet and mutton, while HDL 10-1 was made from maize. HDM 05-1 is the only sample that reported S-K spectroscopic result significantly higher that of PCNAA ($P \geq 0.1$). Similarly, four out of five samples that reported below detection limit are produced from rice.

5.3 Farm Soil and Produce

Determination of iodine in farm soil and produce (Table 4.8) by spectroscopy give results below detection limit while PCNAA report iodine in the farm produce but not the soil. Iodine concentration in soil is generally a function of distance from the coastline. Gbedebo and Oyesanya (2005), reported iodine levels in soils at Sowonjo (5 km from the coastline), Lagos state and Yewa north local government area (53km from the coastline), Ogun state, to be 7.4mg/kg and 2.1 to 5.8 mg/kg. The iodine concentration in farm produce is similar to that reported by Salau*et al.,* (2008) and Ujuwundu*el al.,* (2011).

Pearson correlations between sodium and potassium of the cultivated soil and farm produce were 0.75 and 0.77, respectively. The correlation indicates strong positive relationship between sodium and potassium in soils and farm produce. In contrast, the correlation of sodium between the cultivation soil and farm produce was 0.28 and that of potassium was 0.0087. Bioaccumulation and bioavailability are largely responsible for the poor correlation. Different crops absorbs one nutrient but not another. From Table 4.8, the spinach sample accumulates 180% and 170% more potassium and bromine respectively, than the soil which it is grown on. The similarities between the chemical behavior of bromine and iodine could be employed to justify why no iodine was detected in farm soils but detected in crops grown in the soils.

5.4 Iodine Titrimetry

The detection limit for iodine titrimetry was estimated from the mean of 10 blanks and the standard deviation of the blanks at 95% confidence interval using the formula below (IUPAC, 2006).

$$X_L = \mu_{bl} + kSD_{bl}$$

X_L= limit of detection, μ_{bl}= mean of blank, k= confidence level and SD_{bl}= standard deviation

The measured detection limit was 0.021mg/kg, while the mean of the blank measurement is 0.0044 mg/kg with a standard deviation of 0.017.

Nigerian law requires that salt iodine concentration should be greater than 30 ppm at distributor and retail levels and greater than 15 ppm iodine at household level. The Table 4.12 reveals that the level of salt iodization was excellent. Only sample HDL08 from Table 4.12, reveals no iodination and sample HDL11 that shows iodine content to be slightly below the requirement. Interestingly, while samples for HDL11 supplied second to the least iodine per day as shown in Figure 5.3, samples from HDL8 supplied 292.8 µg of iodine per day. Sampling questionnaire shows that breakfast and lunch meals were purchased from eateries (restaurant), and the two meals account largely for the iodine content in Table 4.6 and Figure 5.3. Ujuwundu*et al.*, (2011) reported 22.2 to 74.1 mg/kg iodine concentration with a mean of 47.89mg/kg in packaged salt and 12.7 to 49.7 mg/kg iodine concentration with a mean of 26.98 mg/kg in exposed salt from Owerri, southeastern Nigeria. Tyndall *et al.*, (2013) reported that iodine concentrations of 26.31 to 106.82 mg/kg with mean of 69.01 mg/kg were obtained in seven major brands sampled at random from market, and 58.16 to 105.75 mg/kg with mean of 71.34 mg/kg in eateries (restaurants) across Yola town, northeastern Nigeria.

It was observed that households with less population will likely have more exposed salts due to long time it take to be consumed after breaking open the package. UNICEF, (2008) also point out that the iodine content of table salt decreases with exposure. Salt samples from volunteer number 1 and 3 were

collected after a three month interval, while there was not much difference from volunteer number 3 samples (37.21 mg/kg initial and 33.60 mg/kg final), samples from volunteer number 1 showed marked differences (19.50 mg/kg initial and 74.78mg/kg final). The volunteer number 3 was using the same salt through the period of sampling, while volunteer number 1 exhausted the first sampled salt (that was been used for about 6-9 months) and was using a fresh salt (that was open from its package in 2 weeks).

Pearson correlation of the iodine concentration in salt with that of the iodine concentration in corresponding duplicated diet was 0.81. This shows strong positive relationship between iodised salt and dietary iodine intake.

5.5 Dietary Assessment Questionnaire

Table 4.13 summaried the dietary assessment questionnaires collected. The questionnaire reveals that Tuwonmasara was the most consumed food by 44.81% of the population, often at evening while tea and bread is the most common breakfast by 33.69% of the population. Jollof rice is the most consumed lunch by 29.54% of the population. Other more commonly consumed foods are savory rice and stew at lunch (19.54 %), Koko and Kosai at breakfast (13.53 %), Noodles at dinner (6.69%), Tuwonshinkafa at dinner (5.03%) and potato chips at breakfast (4.41%).

As pointed out earlier, iodine concentration in food stuffs is directly related to the iodine concentration of the cultivation soil, which in turn is largely a function of the distance from coastline. Thus, food stuffs cultivated around the study area, which is approximately 650 Km from coastline is likely to contain infinitesimal iodine. As such, iodine in foods is likely to come primarily from iodized salt.

CHAPTER SIX

6.0 CONCLUSION AND RECOMMENDATIONS

6.1 Conclusion

The evaluation of iodine content of commonly consumed foods in Zaria metropolis was successfully carried out. Pre-concentration Neutron Activation Analysis and classic Sandell-Kolthoff Spectroscopy were both employed in the determination of iodine in micro concentration. While the spectroscopy is cheap with little labour, the accuracy of its result is not as excellent as that of the neutron activation analysis.

Iodine concentration of foods analyzed were found to range from 0.16 ± 0.02 to 2.04± 0.11 mg/kg for breakfast, 0.50 ± 0.03 to 1.56 ± 0.02mg/kg for lunch and 0.14 ± 0.04 to 1.99 ± 0.09mg/kg for dinner. The daily consumption of iodine was found to be from 187.36 to 1165.29 mg/day, with an average consumption of 531.0 mg/day. The foods analyzed supplied the recommended daily allowance for iodine and in some cases approaching the upper tolerable limit.

The iodine concentrations in soils and potable water were very low in the analyzed samples and could not be detected by the methods applied. However, the iodine concentration in cultivated crops were equally low but were successfully determined, with maize containing the least iodine at 0.22±0.08, rice 0.29± 0.05, wheat 0.31± 0.08, beans 0.32 ± 0.07 and spinach 1.27± 0.10 mg/kg respectively.

Table salts sold within the study area were found to be sufficiently iodized with concentration ranging from 14.87 ± 0.51to 74.78 ± 0.59 mg/kg with an average concentration of 45.12 mg/kg from home samples and 41.12 ± 0.51 to 93.31 ± 0.59 mg/kg with an average concentration of 61.01 mg/kg for retail samples

respectively. Iodine concentration of foods was found to have positive correlation to iodine concentration of the table salts.

Iodine consumption by different income group is not significantly different since the major avenue for iodination of foods is iodized salt and is accessible to all at low cost.

The spectroscopic technique shows relatively poor correlation to PCNAA when dry ashing digestion method was employed (0.53). However, the correlation improved significantly to 0.89 when microwave assisted acid digestion was employed.

6.2 RECOMMENDATION

1. There is a need for the National Agency for Food Drug Administration and Control (NAFDAC) and Standard Organization of Nigeria (SON) to reconsider the salt intake of the population and the iodine concentration in salt.
2. The university should carry out more community development projects and enlightenment campaigns so that the community can better understand and responds to needs of the university staff and students.
3. The university library should endeavor to update its serial section for easy access to relevant information in any field of research.

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Appendix I: List of Reagent, apparatus and equipment used.

S/N	Reagent / Equipment	Company
1	Hydrazine Sulphate ($H_4N_2.H_2SO_4$)	BDH AnalaR
2	Thioacetamide (C_2H_5NS)	Sigma Aldrich
3	Bismuth Nitrate ($BiN_2O_9.5H_2O$)	Aldrich
4	Ultra-pure Nitric acid ($HNO_3$65%)	Riedel de Haen
5	Sodium Hydroxide (NaOH)	BDH AnalaR
6	Potassium iodate (KIO_3)	BDH AnalaR
7	Arsenic trioxide (As_2O_3)	Sigma
8	Cerium Ammonium Sulphate ($2(NH_4)_2SO_4.Ce(SO_4)_2.2H_2O$)	BDH AnalaR
9	Concentrated Sulphuric acid ($H_2SO_4$98%)	Fluka
10	De-ionised water	CERT
11	Volumetric Flasks (10 and 100ml)	Pyrex
12	Test tubes	Pyrex
13	Nickel Crucibles	CSN
14	400 ml Beakers	Pyrex
15	Laboratory Furnace	Nabertherm
16	Water Bath	Fisher Scientific
17	Cintra 6 UV/ Visible Spectrophotometer	GBT
18	Vacuum filtration assembly	Millipore
19	NH-SII Centrifuge	IEC
20	Ethos 1 advance microwave digestion system	Milestone

Appendix IIa: Dietary Assessment Questionnaire

Questionnaire 1: Diet Source.

Research Topic: Evaluation Of The Iodine Content Of Some Commonly Consumed Foods In Zaria Metropolis, Kaduna State.

Date

Location

Income Status?

Low Income ☐ Average Income ☐ High income ☐

☐ Other; (please specify)

Commonest source of food?

Source	One Meal	Two Meals	All Meals	Other
Self Cultivated and home processed				
Purchased Raw and home processed				
Restaurant				

How often do you take meal in a day?

One ☐ Two ☐ Three ☐ Other ☐

Appendix IIb: Dietary Assessment Questionnaire

Questionnaire 2: Dietary Recall

Research Topic: Evaluation Of The Iodine Content Of Some Commonly Consumed Foods In Zaria Metropolis, Kaduna State.

Date

Location

Number of meals taken in a day?

	Food item	Amount per Serving	Amount Consumed	Cost
Breakfast				
Lunch				
Dinner				
Other				

Do you take meals with water? ----------

Estimate of water quantity consumed per meal? ----------

Estimate of water quantity consumed in a day? ----------

Most taken source of water?

- ☐ Well
- ☐ Tap
- ☐ Pure Water
- ☐ Bottled Water
- ☐ Other (please Specify) ----------

Appendix III: FOOD CODES

S/n	Code	Food
1.	B1	Koko da Kosai
2.	P1	Chips and egg
3.	W1	Bread with tea
4.	W2	Pancake with sauce
5.	Y1	Fried yam
6.	Y2	Boiled yam fried with egg
7.	B2	Moin-moin
8.	B4	Bean porridge
9.	M1	Tuwonmasara with kuka
10.	MS1	Noodles with egg
11.	R1	Boiled rice with stew
12.	R2	Tuwonshinkafa with egusi soup
13.	R3	Jollop rice
14.	R4	Waina with taushe
15.	R5	Rice, beans
16.	W5	Jollop spaghetti
17.	W7	Semovita with vegetable soup
18.	Y5	Pounded yam with vegetable soup

1 Koko da Kosai is a traditional breakfast meal. Koko is made mainly from starch extracted from maize and millet while kosai is prepared by deep frying beans paste.

2 Moi- moi is by boiling beans paste along with variant ingredients which include peppers, onion

3 Tuwonmasara is a form of stiffed porridge prepared from fine maize flour. The main accompanying Kuka sauce is prepared from fine baobab leave flour.

4 Tuwonshinkafa is a stiff porridge/paste- like meal, made with unpolished rice.

5 Waina is made from rice paste baked on an open plate.

Appendix IVa: Spectroscopy Absorbance at 430nm (Duplicate Diet)

S/N	Sample	Sample W (g)	Ash W (g)	Abs (nm)	I (ppm)	Sample W (g)	Ash W (g)	Abs (nm)	I (ppm)	Sample W (g)	Ash W (g)	Abs (nm)	I (ppm)
1	HDM01-1a	1.0011	0.084	0.7773	0.178	0.9884	0.0875	0.7781	0.151	1.0002	0.0859	0.7778	0.163
2	HDM01-2a	1.0091	0.0755	0.7590	0.796	0.9994	0.0947	0.7564	0.872	1.0025	0.0866	0.7539	0.959
3	HDM01-3a	0.9981	0.0861	0.7815	0.039	0.2509	-	0.7769	0.049	1.0022	0.0869	0.7814	0.044
4	HDM01-1b	1.0033	0.0812	0.7485	1.140	1.0009	0.0769	0.7511	1.051	0.9987	0.0767	0.7536	0.966
5	HDM01-2b	1.0021	0.0815	0.7818	0.029	0.9904	0.0608	0.7824	0.011	1.0013	0.0621	0.7814	0.044
6	HDM01-3b	1.0029	0.0743	0.7132	2.315	0.9975	0.0713	0.7174	2.164	0.9988	0.073	0.7119	2.349
7	HDM02-1	0.9989	0.0675	0.7542	0.945	0.9984	0.0585	0.7470	1.184	0.9974	0.0655	0.7480	1.149
8	HDM02-2	0.9947	0.077	0.7851	BDL	0.9933	0.064	0.7815	0.039	0.9919	0.0712	0.7817	0.032
9	HDM02-3	1.0051	0.0668	0.7729	0.328	0.9987	0.0537	0.7713	0.377	0.9965	0.0548	0.7699	0.425
10	HDM03-1a	0.9989	0.0693	0.7761	0.218	0.9907	0.0476	0.7768	0.195	0.9944	0.0563	0.7754	0.24
11	HDM03-2a	0.9903	0.0712	0.7735	0.302	0.9986	0.059	0.7650	0.586	1.0014	0.0714	0.7647	0.6
12	HDM03-3a	1.0085	0.0681	0.7563	0.883	0.9971	0.0558	0.7552	0.911	0.9986	0.0657	0.7605	0.736
13	HDM03-1b	1.0023	0.0722	0.7738	0.298	0.995	0.0648	0.7698	0.426	0.9905	0.0638	0.7745	0.271
14	HDM03-2b	0.9981	0.0772	0.7760	0.223	1.0014	0.0808	0.7766	0.202	0.9955	0.0686	0.7773	0.179
15	HDM03-3b	0.9927	0.0702	0.7566	0.862	0.9976	0.0584	0.7536	0.964	1.0045	0.0725	0.7508	1.065
16	HDM04-1	1.0066	0.094	0.7588	0.798	1.0025	0.096	0.7612	0.716	0.9895	0.0931	0.7806	0.0675
17	HDM04-2	0.9956	0.0789	0.7786	0.135	0.9915	0.0659	0.7799	0.091	0.9956	-	0.7793	0.114
18	HDM04-3	1.0054	0.0925	0.7672	0.517	0.9996	0.0886	0.7695	0.439	0.9974	0.086	0.7655	0.57
19	HDM05-1	0.9926	0.117	0.7036	2.610	1.0022	0.0972	0.7058	2.560	0.9971	0.0994	0.7042	2.6
20	HDM05-2	0.9932	0.0772	0.7702	0.411	0.999	0.0647	0.7709	0.392	1.006	-	0.7706	0.403
21	HDM05-3	0.2559	-	0.6898	0.790	0.2514	-	0.6873	0.797	0.2502	-	0.6865	0.8
22	HDM06-1	0.2577	-	0.6561	1.084	0.2554	-	0.6610	1.033	0.2535	-	0.6600	1.033

Appendix IVb: Spectroscopy Absorbance at 430nm (Duplicate Diet)

S/N	Sample	Sample W (g)	Ash W (g)	Abs (nm)	I (ppm)	Sample W (g)	Ash W (g)	Abs (nm)	I (ppm)	Sample W (g)	Ash W (g)	Abs (nm)	I (ppm)
23	HDM06-2	0.9927	0.06845	0.7745	0.271	1.0055	0.0627	0.7822	0.018	1.0015	0.0645	0.7755	0.238
24	HDM06-3	1.0083	0.0684	0.7716	0.373	1.0006	0.0661	0.7704	0.408	0.9975	0.0657	0.7718	0.362
25	HDM07-1	0.9963	0.079	0.7718	0.362	0.9912	0.0701	0.7768	0.195	0.9935	0.0726	0.7707	0.395
26	HDM07-2	0.997	0.0719	0.7817	0.032	1.0017	0.0689	0.7791	0.119	1	0.0684	0.7800	0.09
27	HDM07-3	1.0031	0.0916	0.7368	1.528	0.9986	0.0897	0.7389	1.454	0.9921	0.0885	0.7352	1.565
28	HDL08-1	0.2529	-	0.6914	0.767	0.2515	-	0.6897	0.777	0.2508	-	0.6938	0.741
29	HDL08-2	0.9916	0.0883	0.7591	0.776	1.001	0.0915	0.7575	0.837	0.9893	0.0873	0.7553	0.9
30	HDL08-3	1.0021	0.0588	0.7829	BDL	0.9952	0.0579	0.7817	0.032	0.9901	0.0542	0.7808	0.061
31	HDL09-1	0.2568	-	0.7039	0.672	0.26	-	0.7047	0.674	0.251	-	0.7077	0.625
32	HDL09-2	0.9899	0.0772	0.7381	1.467	0.9961	0.0818	0.7417	1.358	1.0013	0.0763	0.7472	1.18
33	HDL09-3	0.2514	-	0.6530	1.083	0.253	-	0.6638	0.999	0.2513	-	0.6509	1.1
34	HDL10-1	1.0027	0.0809	0.7681	0.487	1.0045	0.0815	0.7671	0.520	0.9874	0.0801	0.7664	0.536
35	HDL10-2	0	0	0.0000	0.000	0	0	0.0000	0.000	0	0	0.0000	0.000
36	HDL10-3	1.0059	0.0804	0.7652	0.585	1.005	0.0849	0.7676	0.503	1.0024	0.0854	0.7656	0.568
37	HDL11-1	0.9914	0.0754	0.7716	0.364	1.0032	0.0767	0.7725	0.339	0.9977	0.0714	0.7732	0.315
38	HDL11-2	0.9895	0.9915	0.7828	BDL	0.9949	0.0968	0.7925	BDL	0.9998	0.1023	0.7845	BDL
39	HDL11-3	0.9912	0.0617	0.7829	BDL	0.9967	0.0605	0.7826	0.003	0.9975	0.0622	0.7813	0.047
40	HDL12-1	0.2507	-	0.6110	1.430	0.2588	-	0.6314	1.301	0.2501	-	0.6046	1.48
41	HDL12-2	1.0016	0.0615	0.7681	0.487	0.9912	0.077	0.7678	0.491	0.9985	0.0815	0.7709	0.393
42	HDL12-3	0	0	0.0000	0.000	0	0	0.0000	0.000	0	0	0.0000	0.000

Appendix V: Spectroscopy Absorbance at 430nm (Total Diet)

S/N	Code	Abs (nm)	I (ppm)	Abs (nm)	I (ppm)	Abs (nm)	I (ppm)
1	RL 1-1	0.7762	0.281	0.7743	0.279	0.7757	0.233
2	RL 1-2	0.7251	1.974	0.7265	1.867	0.7255	1.9
3	RL 1-3	0.5625	7.358	0.5346	8.243	0.5356	8.209
4	RL 2-1	0.7614	0.772	0.7604	0.41	0.761	0.721
5	RL 2-2	0.7506	1.129	0.743	1.319	0.7436	1.299
6	RL 2-3	0.7863	-0.053	0.7874	-0.156	0.7888	-0.203
7	RL 3-1	0.7611	0.781	0.7586	0.801	0.7603	0.744
8	RL 3-2	0.7497	1.159	0.749	1.12	0.7502	1.08
9	RL 3-3	0.7514	1.103	0.752	1.02	0.7511	1.05
10	ML 1-1	0.7494	1.169	0.7467	10.196	0.7494	1.106
11	ML 1-2	0.7388	1.520	0.7354	1.571	0.7356	1.565
12	ML 1-3	0.7427	1.391	0.7506	1.066	0.7462	1.213
13	ML 2-1	0.7484	1.202	0.7449	1.256	0.7451	1.249
14	ML 2-2	0.7545	1.000	0.7551	1.917	0.7554	0.907
15	ML 2-3	0.7485	1.199	0.7489	1.123	0.7483	1.143
16	ML 3-1	0.77	0.487	0.7688	0.462	0.7708	0.395
17	ML 3-2	0.7465	1.265	0.7439	1.289	0.7439	1.289
18	ML 3-3	0.6935	3.020	0.7075	2.498	0.7027	2.658
19	HL 1-1	0.7497	1.159	0.7487	1.13	0.7484	1.14
20	HL 1-2	0.7754	0.308	0.7748	0.262	0.7752	0.249
21	HL 1-3	0.7246	1.990	0.7249	1.92	0.7266	1.864
23	HL 2-2	0.7107	2.450	0.7078	2.488	0.7111	2.379
24	HL 2-3	0.7478	1.222	0.7448	1.259	0.744	1.286



Nuclear Science and Technology Section
Centre for Energy Research and Training
Ahmadu Bello University, P.M.B. 1014, Zaria, NIGERIA

Our Ref. NIRR-1/JC/12/13 **Date: 2013-11-26**

The analytical results of instrumental neutron activation analysis of Forty (40) Biological samples for **M. Tukur** are presented below.
The reported uncertainty was calculated mainly from counting statistics and is not the normal standard deviation on replicate analyses.

A SUMMARY OF ANALYTICAL RESULT

Sample	Sodium (mg/kg)	Potassium (mg/kg)	Bromine (mg/kg)	Iron (mg/kg)	Zinc (mg/kg)	Iodine (mg/kg)
HDM01-1a	7139 ± 7	12510 ± 512	5.04 ± 0.27	140.51	8.05 ± 2.24	0.16 ± 0.02
HDM01-2a	7472 ± 8	1500 ± 321	4.39 ± 0.18	146.42	8.54 ± 2.63	0.89 ± 0.20
HDM01-3a	8110 ± 8	5241 ± 446	4.49 ± 0.26	90.32 ± 24.03	33.79 ± 3.85	0.14 ± 0.04
HDM01-1b	4109 ± 8	10840 ± 357	2.88 ± 0.19	116.31	2.18	1.10 ± 0.09
HDM01-2b	3943 ± 8	2904 ± 342	2.60 ± 0.18	118.47	16.58 ± 3.08	BDL
HDM01-3b	4378 ± 9	5075 ± 345	9.72 ± 0.26	120.08	17.74 ± 2.77	1.92 ± 0.08
HDM02-1	5790 ± 12	2202 ± 244	8.37 ± 0.26	91.49 ± 24.61	13.96 ± 2.86	1.38 ± 0.34
HDM02-2	6393 ± 7	2628 ± 315	4.41 ± 0.23	127.38	14.21 ± 3.27	BDL
HDM02-3	4863 ± 10	3041 ± 225	3.84 ± 0.23	125.35	27 ± 4.40	0.41 ± 0.04
HDM03-1a	3354 ± 7	2935 ± 264	24.63 ± 0.30	86.89 ± 18.16	14.70 ± 3.91	0.98 ± 0.07
HDM03-2a	6590 ± 13	3037 ± 340	4.06 ± 0.22	117.69	6.68	0.50 ± 0.03
HDM03-3a	6770 ± 14	1821 ± 304	8.04 ± 0.19	43.37	10.67 ± 3.17	0.89 ± 0.01
HDM03-1b	5085 ± 10	1355 ± 262	4.87 ± 0.16	74.73	17.29 ± 2.45	0.34 ± 0.08
HDM03-2b	7351 ± 8	2598 ± 351	9.01 ± 0.39	25.85	7.37 ± 2.21	0.82 ± 0.01
HDM03-3b	5999 ± 12	2871 ± 376	3.95 ± 0.23	59.97	7.88 ± 2.06	1.01 ± 0.13
HDM04-1	8112 ± 16	11370 ± 532	7.40 ± 0.29	25.66	19.70 ± 2.96	0.59 ± 0.06
HDM04-2	5254 ± 11	2558 ± 378	3.90 ± 0.27	56.29 ± 13.79	5.44	BDL
HDM04-3	11040 ± 11	4431 ± 516	10.03 ± 0.03	49.90 ± 9.86	7.93 ± 1.74	1.99 ± 0.09
HDM05-1	4923 ± 10	7659 ± 413	3.76 ± 0.22	60.26 ± 14.89	21.48 ± 2.34	1.87 ± 0.25
HDM05-2	6363 ± 12	3556 ± 380	6.60 ± 0.24	98.18	9.11 ± 2.31	1.36 ± 0.13
HDM05-3	2633 ± 8	3112 ± 410	10.60 ± 0.21	33.97	6.84 ± 1.61	0.84 ± 0.23
HDM06-1	2643 ± 3	7453 ± 127	3.91 ± 0.20	67.35 ± 19.70	13.44 ± 1.84	1.03 ± 0.08
HDM06-2	4495 ± 9	1502 ± 362	4.04 ± 0.23	72.09	12.10 ± 1.98	1.56 ± 0.02
HDM06-3	5582 ± 17	4373 ± 581.6	4.50 ± 0.23	73.92 ± 18.70	10.09 ± 1.91	1.38 ± 0.05
HDM07-1	5380 ± 11	1300 ± 231	4.56 ± 0.22	76.31 ± 14.22	11.71 ± 2.43	2.04 ± 0.11
HDM07-2	5134 ± 10	2326 ± 249	9.03 ± 0.25	50.13 ± 13.94	7.88 ± 1.60	1.13 ± 0.08
HDM07-3	7626 ± 8	4010 ± 381	3.71 ± 0.30	84.16	11.64 ± 1.65	1.45 ± 0.10

AppendixVIb: INAA/ PCNAA Certificate (Duplicate Diet)

Sample	Sodium (mg/kg)	Potassium (mg/kg)	Bromine (mg/kg)	Iron (mg/kg)	Zinc (mg/kg)	Iodine (mg/kg)
HDL08-1	4889 ± 7	6511 ± 206	6.86 ± 0.29	63.55 ± 10.26	17.43 ± 1.91	0.77 ± 0.05
HDL08-2	2934 ± 3	2115 ± 134	12.03 ± 0.18	48.86± 9.12	7.17 ± 1.13	1.00 ± 0.10
HDL08-3	7706 ± 8	4340 ± 399	6.24 ± 0.33	110.9 ± 32.7	12.93 ± 1.71	BDL
HDL09-1	5594 ± 11	3652 ± 267	4.50 ± 0.26	147.8 ± 21.3	27.3 ± 2.92	0.65 ± 0.18
HDL09-2	7264 ± 7	4229 ± 389	4.74 ± 0.28	71.59 ± 13.96	8.06 ± 1.93	1.28 ± 0.06
HDL09-3	4872 ±7	3511 ±208	9.14 ±0.11	72.96 ±12.19	7.41 ±1.11	1.4 ± 0.19
HDL10-1	6922 ± 7	4824 ±416	4.96 ±0.26	66.65 ±16.21	12.12 ± 2.07	1.12 ± 0.03
HDL10-3	6975 ± 7	4832 ± 425	4.95 ± 0.29	68.01 ± 16.73	11.57 ± 2.03	1.09 ± 0.02
HDL11-1	4756 ± 9	4015 ± 311	4.32 ± 0.228	83.55	24.13 ± 2.09	0.32 ± 0.09
HDL11-2	2548 ± 10	4957 ± 186	4.15 ± 0.22	78.67 ± 10.59	8.03 ± 1.54	0.96 ± 0.08
HDL11-3	7591 ± 1	2263 ± 321	7.98 ± 0.29	64.88 ± 18.56	11.88 ± 2.47	BDL
HDL12-1	4786 ± 9	3181 ± 280	10.31 ± 0.18	65.51	7.018	1.44 ± 0.18
HDL12-2	4994 ± 10	4611 ± 314	8.72 ± 0.17	71.46	7.45	0.51 ± 0.05

Figures are in mg/kg (ppm)
BDL: Below Detection Limit

Professor M. O. A. Oladipo
(Head of Section)

Appendix VII: INAA/ PCNAA Certificate (Total Diet)



Nuclear Science and Technology Section
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Our Ref. NIRR-1/JC/13/06 **Date: 2013-11-26**

The analytical results of instrumental neutron activation analysis of Twenty (20) Biological samples for **M. Tukur** are presented below.
The reported uncertainty was calculated mainly from counting statistics and is not the normal standard deviation on replicate analyses.

A SUMMARY OF ANALYTICAL RESULT

Sample	Sodium (mg/kg)	Potassium (mg/kg)	Bromine (mg/kg)	Iron (mg/kg)	Zinc (mg/kg)	Iodine (mg/kg)
RL 1-1	3086 ± 6.2	5180 ± 171	3.13 ± 0.15	89.54	23.26 ± 5.93	0.30 ± 0.05
RL 1-2	5078 ± 8.72	3115 ± 278	8.52 ± 0.35	79.02 ± 11.90	14.96	1.89 ± 0.08
RL 1-3	14280 ± 14	4273 ± 641	6.80 ± 0.20	45.85	20.62	9.68 ± 0.16
RL 2-1	1646 ± 3.3	10020 ± 170	4.35 ± 0.14	13.14	5.21	0.75 ± 0.06
RL 2-2	4470 ± 4.5	3279 ± 207	4.01 ± 0.15	30.91	7.21	1.34 ± 0.08
RL 2-3	9264 ± 9.3	4655 ± 101	6.21 ± 0.26	68.81 ± 14.27	19.25	BDL
RL 3-1	4439 ± 6.8	5194 ± 251	7.52 ± 0.54	67.85 ± 9.18	16.42 ± 2.10	0.81 ± 0.10
RL 3-2	8282 ± 16.4	5163 ± 465	5.76 ± 0.32	51.19	17.08	1.16 ± 0.08
RL 3-3	9449 ± 18.9	4901 ± 529	4.29 ± 0.28	17.73	29.97 ± 5.03	0.78 ± 0.06
ML 1-1	5983 ± 12.0	11950 ± 418	5.89 ± 0.21	237.15	39 ± 7.45	1.36 ± 0.07
ML 1-2	11070 ± 11.1	3251 ± 422	8.39 ± 0.32	289.84	21.66	1.61 ± 0.08
ML 1-3	5783 ± 11.9	4547 ± 496	5.06 ± 0.02	151.1	22.95 ± 3.92	1.39 ± 0.07
ML 2-1	8356 ± 8.4	3015 ± 404	11.31 ± 0.36	175.93	13.37 ± 3.76	1.29 ± 0.07
ML 2-2	4692 ± 8.4	3214 ± 216	3.26 ± 0.18	82.05 ± 16.05	17.10 ± 2.54	0.89 ± 0.09
ML 2-3	7591 ± 7.8	3698 ± 412	4.36 ± 0.21	123.65 ± 24.18	21.53 ± 4.36	1.13 ± 0.22
ML 3-1	6325 ± 12.7	10190 ± 408	5.75 ± 0.27	158.13	28.73 ± 3.71	0.55 ± 0.07
ML 3-2	7582 ± 13.4	5648 ± 407	4.98 ± 0.16	113.6	21.44 ± 2.14	1.31 ± 0.06
ML 3-3	7797 ± 15.6	5536 ± 493	4.25 ± 0.27	86.32 ± 22.27	39.70 ± 4.65	2.96 ± 0.09
HL 1-1	8014 ± 12.7	11256 ± 517	4.53 ± 0.23	93.52	16.69 ± 3.24	1.18 ± 0.07
HL 1-2	9316 ± 28.0	12150 ± 421	5.94 ± 0.32	85.63 ± 22.09	38.66 ± 4.79	0.42 ± 0.04
HL 1-3	7967 ± 15.9	9369 ± 420	8.32 ± 0.83	97.10 ± 6.51	16.64 ± 2.45	2.09 ± 0.08
HL 2-2	11850 ± 23.7	5135 ± 611	5.74 ± 0.26	68.32	8.22 ± 2.00	2.44 ± 0.08
HL 2-3	6310 ± 6.5	4179 ± 418	4.88 ± 0.25	145.28	23.34 ± 3.69	1.27 ± 0.04

Figures are in mg/kg (ppm)
BDL: Below Detection Limit

Professor M. O. A. Oladipo
(Head of Section)

Appendix VIII: Farm Soil and produce



Nuclear Science and Technology Section
Centre for Energy Research and Training
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Our Ref. NIRR-1/JC/13/06 **Date: 2013-11-26**

The analytical results of instrumental neutron activation analysis of Ten (10) Biological and Geological samples for **M. Tukur** are presented below.
The reported uncertainty was calculated mainly from counting statistics and is not the normal standard deviation on replicate analyses.

A SUMMARY OF ANALYTICAL RESULT

Sample	Sodium (mg/kg)	Potassium (mg/kg)	Bromine (mg/kg)	Iron (mg/kg)	Zinc (mg/kg)	Iodine (mg/kg)
FB 1-2	26.61 ± 0.40	11780 ± 117	1.11 ± 0.04	87.90 ± 20.91	20.83 ± 2.65	0.32 ± 0.07
FW 1-3	33.53 ± 0.40	4443 ± 75	0.84 ± 0.03	81.81 ± 32.89	39.14 ± 2.82	0.31 ± 0.08
TR-4	31.3 ± 0.28	1928 ± 30	35.09 ± 2.28	BDL	13.51 ± 2.20	0.29 ± 0.05
FS 5-3	435.9 ± 1.31	38540 ± 231	4.39 ± 0.11	64.27 ± 35.48	10.48 ± 1.87	1.27 ± 0.10
FM 8-3	14.99 ± 0.20	1334 ± 26	0.42 ± 0.03	976.1 ± 37.1	28.84 ± 2.51	0.22 ± 0.08
F 1-2	1438 ± 2	18090 ± 127	1.21 ± 0.13	18780 ± 244	31.55 ± 4.76	BDL
F 1-3	2292 ± 4	31330 ± 282	6.19 ± 0.88	18790 ± 244	62.66 ± 3.32	BDL
F 4-1;2	515 ± 2	8732 ± 78	3.52 ± 0.77	9235 ± 193	19.96 ± 4.13	BDL
F 5-3	2066 ± 4	21330 ± 235	2.57 ± 0.19	18110 ± 887	41.26 ± 4.75	BDL
F 8-3	1797 ± 4	25610 ± 153	0.39 ± 0.10	21020 ± 252	30.88 ± 4.69	BDL

Figures are in mg/kg (ppm)
BDL: Below Detection Limit

Professor M. O. A. Oladipo
(Head of Section)

Appendix IX: Salt iodine titre value

S/N	Sample	R1 (cm^3)	R2 (cm^3)	R3 (cm^3)	AVR (cm^3)	I (mg /kg)
1	HDM01a	1.90	1.95	1.85	1.90	19.50
2	HDM01b	7.20	7.30	7.30	7.27	74.81
3	HDM02	2.50	2.45	2.45	2.47	25.37
4	HDM03a	3.65	3.60	3.60	3.62	37.22
5	HDM03b	3.30	3.25	3.25	3.27	33.61
6	HDM04	4.80	4.60	4.70	4.70	48.34
7	HDM05	7.20	7.10	7.15	7.15	73.60
8	HDM06	6.35	6.35	6.40	6.37	65.54
9	HDM07	4.75	4.80	4.85	4.80	49.37
10	HDL08	0.00	0.00	0.00	0.00	BDL
11	HDL09	4.00	4.10	3.95	4.02	41.34
12	HDL10	6.10	6.05	6.10	6.08	62.55
13	HDL11	1.40	1.50	1.45	1.45	14.87
14	HDL12	3.95	3.90	3.90	3.95	40.61
15	Smr a	4.60	4.55	4.50	4.55	46.79
16	Smr b	5.55	5.60	5.50	5.55	57.09
17	Smr c	6.55	6.50	6.60	6.55	67.39
18	Sabo G. a	6.10	6.00	6.20	6.10	62.75
19	Sabo G. b	5.00	5.05	5.10	5.05	51.94
20	Sabo G. c	5.50	5.45	5.60	5.52	56.79
21	City a	4.05	4.00	3.95	4.00	41.13
22	City b	9.00	9.10	9.10	9.07	93.35
23	City c	7.05	6.90	7.05	7.00	72.03