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# Preliminary study of iodine analysis in food using epithermal method of neutron activation analysis in TRIGA 2000 reactor pneumatic system facility

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Abstract. Iodine in food usually presents in low-level concentration, so it is not easy for measurement by thermal NAA related to the high background of the Compton. Therefore, the aim of this work is to develop the iodine analysis method in a food matrix, so that the iodine analysis method is obtained, which is guaranteed the validity. In this activity, a number of 50 mg of SRM NIST 1548a Typical Diet, IPE sample and standard were irradiated in a pneumatic system facility at TRIGA 2000 reactor with the power of 700 KW by covering sample using cadmium plates to absorb thermal neutrons. The results of the iodine-128 spectrum analysis at 442.3 Kev in the GENIE 2000 software showed a good prospect for iodine detection, with a limit of detection (LOD) is 0.19 mg/kg. The results of the validation method by accuracy and precision testing stated with % Recovery and RSDr each ranged between 93.6-103.1% and 16.33-16.60, respectively. This data have good agreement with accuracy and precision. Criteria the preliminary study, it can be concluded that iodine at food matrices could be determined using epithermal NAA.

#### 1. Introduction

Food is one of the basic human needs and the main source of nutrient content as macronutrient and micronutrients that play an important role in metabolism and the maintenance of tissue [1]. Micronutrient consist of vitamin and mineral, which is macro and microelements essential and nonessential [2,3]. Iodine is an essential element for the synthesis of thyroid hormones and regulates key biochemical reactions and adequate supply before birth and infancy for optimal physical growth such as brain, muscle, heart, pituitary, and kidney development [4,5,6].

Iodine is needed by the body in a certain concentration range; iodine deficiency causes iodine deficiency symptoms such as goiter, extreme fatigue, increased childhood mortality, reproductive failure, congenital abnormalities with permanent neuromotor damage, hearing problems, reduced intellectual capacity and growth and mental retardation and depression. According to World Health Organization (WHO), iodine deficiency disorder are a significant problem of brain damage and psychomotor retardation in young children which can be prevented in fetuses and breastfeeding infants [7]-[9]. Therefore, it is necessary to analyze iodine in food, it can be determined policies and preventive measures to overcome and control the problem of lack of iodine intake in the body.

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The determination of iodine in food has been a difficult analytical problem for many years, and inconsistent results have been obtained in interlaboratory studies. Although a variety of analytical methods capable of iodine determination at various levels in foodstuffs have been developing. The main difficulty for the determination of iodine is the volatility and low concentration, except in seafood. Therefore, it is a necessary method with a low limit of detection [10].

The determination of iodine in food has been carried out with various analytical techniques, both conventional and instrument. The decision of iodine is conventionally carried out by the colorimetric method, which involves the preparation phase of destruction. This stage proves to be very critical because it can occur cross-contamination and loss during the destruction stage. Besides, several analytical techniques commonly used to determine iodine in environmental and biological samples are potentiometry, isotope exchange, gas chromatography (GC), and inductive plasma mass spectrometry (ICP-MS) [11,12]. However, most of these techniques are not selected due to interference and the need for preconcentration and separation that can cause iodine loss.

Epithermal NAA is sensitivity, selectivity, free of reagents, and nondestructive is one of the best choice techniques for determining iodine at trace element concentration [4]. Iodium has half-life a relatively low (25 minutes) of iodine-128 (I-128) activation products, so the irradiation time must be approved. Otherwise, interference from the background is higher than the product activation elements needed such as Na, K, Cl, Mn, and Br will be significant [11]. Compton effect is an effect caused by the interaction of light  $\gamma$  with the material (HPGe detector) that is the background source [13]. Thus, due to the high background, the determination of iodine with low concentrations in food samples and foodstuff is not easily measured properly using thermal NAA.

The Epithermal NAA method based on the fact that the resonance integral (I0) to thermal neutron  $(n,\gamma)$  cross-section ( $\sigma$ 0) ratio (Q0) for 127-I is 24.8 which is much larger than that for some of the interfering nuclides such as 23 Na, 37 Cl, 27 Al, 41 K, and 55 Mn, whose Q0 values are 0.59, 0.69, 0.71, 0.97, and 1.053, respectively. According, any nuclide having Q0 value >10 is a right candidate for its determination by Epithermal NAA [11,14].

Therefore, in this activity, the development of the iodine analysis method using epithermal NAA was developed and validated the method in the TRIGA (Training Research and Isotope Production General Atomics) reactor 2000 Bandung. Epithermal NAA is an ideal technique for determining I, Br, As, Sb, U, Th, Se and Mo that strongly absorbs specific neutron resonant energy in the epithermal neutron region (epithermal neutron energy of 0.5 eV-1 MeV) in geological samples, food and the environment that cannot be achieved with the use of thermal neutrons and can reduce background interference from thermal activated elements [15,16]. This method is applied for iodine estimation in SRM NIST 1548a Typical Diet and IPE samples as a standard which both have certified iodine concentrations so that they can be used as control samples to evaluate the accuracy of the epithermal NAA method in the TRIGA 2000 Bandung reactor, so it is hoped that this method can support data research activities food samples in producing accurate data.

## 2. Methodology

#### 2.1 Tools and material

Several equipments used in this study such as a gamma-ray spectrometer (Canberra) equipped with a high purity germanium detector (HPGe) with a relative efficiency of 15-35% and a resolution of 1.9 keV for 1332.5 keV Co-60 peaks, Cd cylinders for thermal neutron filters, analytical balance and other supporting laboratory equipment.

The materials used in this activity were IPE proficiency test samples, reference material SRM NIST 1548a Typical Diet certified, demineralized water (> 18 M $\Omega$  cm2), E-Merck titrisol standard and whatman No. 1 filter paper.

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## 2.2 Preparation of 0.01 and 0.05 µg

Standard iodine with a concentration of 4000 mg/L is gradually diluted to reach concentrations of 0.1 and 0.5 mg/L. The standard iodine with a concentration of 0.1 and 0.5 mg/L as much as 100  $\mu$ L were dripped gradually on the whatman No. 1 filter paper that was cut in a circle with a diameter of  $\pm 2$  cm, and then dried under an infrared lamp. Filter paper that already contains 0.01 and 0.05  $\mu$ g iodine standards are put into plastic and sealed by heating.

## 2.3 SRM preparation and IPE sample

Preparation of Standard Reference Material (SRM) NIST 1548a Typical Diet and IPE samples was done by weighing 50 mg each and placed it into plastic, then sealed by heating.

## 2.4 SRM measurements, IPE sample, and standards

Measurement of gamma rays on SRM, IPE samples and standards was carried out with a gamma-ray spectrometer and spectrum analysis I-128 using Genie-2000 software at 442.3 keV energy. The radionuclide used results from the nuclei reaction of 127I  $(n,\gamma)$  128I with a half-life of 25 minutes [17].

## 3. Results and discussion

A preliminary study of iodine determination in food was carried out by irradiating the SRM NIST 1548a Typical Diet irradiation and IPE samples inside polyethylene containers in pneumatic facilities for 60-180 seconds. Both samples were irradiated directly without Cd cylindrical layers (thermal neutron) and irradiated using Cd cylindrical layers (epithermal neutron). The samples were counted using the Gamma spectrometer, and the spectrum analysis was done using Gennie 2000 software. The gamma spectrum of I-128 at energy 442.3 keV for the thermal and epithermal NAA is shown in Figure 1 and Figure 2. High Compton background appears for thermal NAA, as shown in figure 1.





Figure 2, shows that there is a good prospect in the determination of iodine with epithermal NAA. There is a reduction of Compton background; as a result, the iodine is better detected, which is marked by a higher iodine peak in the spectrum.



Figure 2. Iodine spectrum using epithermal NAA.

**Table 1.** Limit of detection of iodine-128 using thermal and epithermal NAA on SRM NIST 1548a typical diet.

Radionuclide	LOD (mg/Kg)		
	Thermal NAA	Epithermal NAA	
I-128	0.84	0.19	

NAA method has very good sensitivity for iodine measurement, but due to the low level of iodine concentration in food, it is not easily measured with thermal NAA [10]. Compton background appears in SRM NIST 1548a Typical Diet spectrum in Figure 1 since some elements such as Na, Cl, K, and Mn and others have low resonance integrals to thermal neutron cross-section (Qo) ratios. Therefore, the elements are easily activated by thermal neutrons. Beside, SRM NIST 1548a Typical Diet contains 0.8% Na; 1.2% Cl; 0.7% K that consider to be major elements will produce a Compton background [16,17]. In normal conditions, it is not possible to determine iodine through I-128 (E = 442.9 keV) at low levels, nevertheless by using a closed Cd cylinder as a thermal neutron filter. Iodine can be determined quantitatively. Figures 1 and 2 show that there are significant differences in the spectrum resulting from thermal neutrons and epithermal neutrons, where the I-128 peak at 442.3 keV energy gives a higher peak with the same irradiation time and counting time conditions. Because Cd can reduce the background compound 10 times lower due to disturbing radioactivity and can increase the detection limit of iodine in the food matrix, as shown in Table 1 [15,17,18].

The detection limit for iodine by the epithermal NAA method is lower than the thermal NAA so that it becomes more sensitive for the determination of iodine in food samples that contains iodine with low concentration. LOD of iodine in SRM NIST 1548a Typical Diet obtained at 0.19 mg/kg; this result is close to the LOD value of other studies that have been conducted using boron carbide material (B4C) as a neutron thermal filter, which is equal to 0.2 mg/kg [19]. Sample with the same composition thus tends to show the same LOD, but the different samples will have different LOD even though with the same method [20].

Become accurate analysis results and consider factors of iodine half-life and prevent radiation exposure and heat generated from excessive Cd cylinders, optimization of irradiation time using a Cd cover is performed on the SRM NIST 1548a Typical Diet and IPE sample and iodine media standards filters with concentrations of 0.01 and 0.05  $\mu$ g. Irradiation is carried out with a time variation of 60; 120 and 180 seconds. The optimum irradiation time obtained, which has the potential to give good results, is 120 seconds based on the initial activity ratio (A<sub>0</sub>) of the SRM NIST 1548a Typical Diet to the IPE sample, shown in figure 3.



Figure 3. A<sub>0</sub> ratio of SRM NIST 1548a Typical Diet to IPE samples.

To ensure the quality of the analysis, a method of validation using certified reference materials was carried out. In the selection of certified reference material as a test quality guarantee, it is required to have a type that is similar or almost the same as the sample type, homogeneous, stable, and has a value that is traceable to international units [21]. Analysis using reference material used as a form of monitoring the accuracy of the data analysis results. The reference material used is a reference material with a complex food type, namely SRM NIST 1648a Typical Diet and IPE sample. The results of the method validation using the SRM NIST 1648a Typical Diet shown in table 3.

Reference	Result(mg/Kg)	Certificate(mg/Kg)	%Recovery	Precision		
Refer	lenee	Result(ing/Rg)	continioate(ing/itg)	/oncecovery	%CV	RSDr
SRM 1548a	NIST Typical					
Diet		$0.782\pm0.17$	$0.759\pm0.103$	103.1	21.84	16.60
IPE 3.2		$0.875\pm0.10$	0.934	93.6	11.11	16.33

**Table 2.** Verification results of the epithermal NAA method using SRM NIST 1648a typical diet and IPE 3.2 sample.

The results of the verification method using SRM NIST 1648a Typical Diet and IPE 3.2 sample shown in Table 2. The verification results show that the analysis of the iodine element using the epithermal NAA method has accuracy and precision values, expressed by % recovery for accuracy and % CV and RSDr for precision. % Recovery is in the range of 93.6-103.1%, and RSDr is in the range of 16.33-16.60 this is a good agreement with the criteria for accepting the accuracy and precision of the AOAC (The Association of Official Analytical Chemists) and laboratory policies with acceptance limits AOAC for accuracy and precision for levels of 1 mg / kg respectively 80-110% and 16 and for % CV in accordance with FK 02 T 016 regarding method validation with precision criteria Value CV% = 0.5 to 2 [22].

#### 4. Conclusion

The Cd cylinder used in epithermal NAA is effective as a thermal neutron filter, and it can reduce the Compton background as well as the LOD value. Therefore, the capability of iodine detection in the food samples improved. Iodine in food samples can be determined quantitatively using epithermal NAA and the results of iodine verification in the food samples show good accordance with the standard.

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