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Analytical Methods

Estimation of iodine in food, food products and salt using ENAA

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ABSTRACT

An epithermal neutron activation analysis (ENAA) method using boron carbide filter was standardised for the determination of low levels of iodine in various food and food products like milk, milk powder, baby food and health drink powders. The concentrations of iodine were also determined in seven samples of iodised salt used in cooking in India. Advantage of iodine determination by ENAA was evaluated by determining boron ratios of iodine and its interfering elements like Na, K, Mn, Cl and Br. Iodine concentrations were determined in seven biological reference materials obtained from NIST and IAEA, out of which three SRMs were used as the control samples. The results showed that trace levels of iodine can be determined in samples even in the presence of high salt content without chemical separations.

1. Introduction

Iodine is one of the essential trace elements and is of much interest in nutritional research. It is required for the production of thyroxine and tri-iodothyronine hormones for proper growth and development of the human body (Underwood, 1977). As per WHO recommendation, the safe and adequate dietary intake of iodine for infants to adults ranges from 50 to 200 μg per day (Hetzel, 1983). The major part of the essential iodine enters into the human body through food and food products. However, deficiency or excess intake of iodine leads to disorders called iodine disorders. Iodine deficiency causes severe disorders like cretinism and mental retardation and its excessive intake can lead to thyroiditis (Hetzel, Dunn, & Stanbury, 1987). Iodine deficiency can be controlled through the fortification of food and food products with added iodine and also through addition of iodised salt in cooked food. In many countries, regulations envisage the control of the level of daily iodine intake through diet. A database of total iodine contents in food and food products will be helpful in recommending a controlled diet. The accurate determination of iodine in diets and individual food items is, therefore, of considerable scientific interest.

Some of the commonly used analytical techniques for determining iodine concentrations in environmental and biological samples are colorimetry, potentiometry, isotope exchange, gas chromatography (GC), inductively coupled plasma mass spectrometry (ICP-MS) and neutron activation analysis (NAA) (Edmonds & Morita, 1998). However, most of the techniques except NAA are not selective, suffer from interferences and need preconcentration or separation procedures which may lead to loss of iodine. The NAA using reactor neutrons is one of the best techniques for iodine determination due to its favourable nuclear properties that result in high sensitivity, and thus its applicability for trace elements. Due to relatively low half-life (25 min) of the activation product of iodine (^{128}I), the irradiation time has to be limited, else interferences from high Compton background from the activation products of interfering elements like Na, K, Cl, Mn and Br will become significant. Thus, due to high Compton background, determination of low levels of iodine in biological samples like food and food products cannot reliably be measured by instrumental NAA (INAA) using whole reactor neutrons. In order to overcome this problem, epithermal NAA (ENAA) is used. Thermal neutrons are filtered off by covering the sample with thermal neutron absorbers like B and Cd. Choice of different boron filters in ENAA has been reported by Bem and Ryan (1981). The ENAA is effective if the isotope of interest has higher Q_0 value than those of the interfering elements. The Q_0 value for a nuclide is defined as the ratio of resonance integral (I_0) to its thermal neutron absorption cross-section (σ_0) of the isotope of interest (De Corte & Simonits, 1989). Any nuclide having Q_0 value >10 is a good candidate for its determination by ENAA. The Q_0 value for ^{127}I is 24.8 which is much larger than that for some of the interfering nuclides such as ^{23}Na, ^{37}Cl, ^{41}K and ^{55}Mn whose Q_0 values are 0.59, 0.69, 0.97 and 1.053, respectively. The relevant nuclear data are given in Table 1 (De Corte & Simonits, 1989). Boron or boron carbide (B_4C) is a better shielding material compared to cadmium for measurement of activity of ^{128}I (25 min), since cadmium produces higher residual activity than boron which is a concern of radiation safety. Thus, ENAA using B_4C is advantageous for the

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Table 1
Boron ratios (R_B) of some important isotopes using boron carbide (B_4C) filter in D8 position of APSARA reactor and advantage factors (AF) for iodine.

Target isotope	σ_0 (b)	Q_0 (I_0/σ_0)	Activation product	Half life	E_γ (keV)	Counts (without B_4C filter)	Counts (with B_4C filter)	R_B	AF = $R_B(x)$ /$R_B(I)$
^{127}I	4.04	24.8	^{128}I	25 min	442.9	598,856	223,447	2.7	–
^{23}Na	0.51	0.59	^{24}Na	14.96 h	1368.5	257,275	3695	69.6	25.8
^{41}K	1.45	0.97	^{42}K	12.4 h	1524.7	19,143	354	54.1	20.0
^{37}Cl	0.42	0.69	^{38}Cl	37.2 min	1642.7	297,735	5931	50.2	18.6
^{55}Mn	13.2	1.053	^{56}Mn	2.58 h	846.6	13,572	576	23.6	8.7
^{81}Br	2.59	19.3	^{82}Br	35.3 h	776.4	1740	552	3.2	1.2

σ_0 (b) – σ_0 in barns, E_γ – main gamma ray energy, and counts = mass normalised count rate × live time of counting.

determination of low concentrations of iodine because samples can be counted with a lesser cooling time, which helps in the assay of short-lived radioisotopes like ^{128}I.

Iodine has been determined by ENAA in many biological and environmental samples like food, human brain, plant materials and marine samples by various researchers (Andrasi, Kucera, Belavari, & Mizera, 2007; Chao, Lee, Tseng, Chen, & Wei, 2002; Hou, Andersson, Roed, Byskov, & Roed, 2007; Rao, Holzbecher, & Chatt, 1995; Steinnes & Frontasyeva, 2002). Trace amounts of iodine have been determined in food and other biological samples using ENAA where measurements were made by Compton suppression gamma ray spectrometry (Armoh et al., 2003; Landsberger, Kelly, Braisted, & Panno, 2006; Yonezawa, Matsue, & Yukawa, 2003). ENAA using cadmium filter was used for the determination of iodine in Chinese medical herbs (Chen, Wei, ChangLai, & Pan, 2003), soft tissues of marine bivalves (Fukushima, Tamate, & Nakano, 2003), health foods (Chen, 2004), diet, and animal products like cheese, eggs, fish, fowl and meats (Zikovsky & Soliman, 2002). Nyarko, Armah, Akaho, Sampong, and Maakuu (2002) used ENAA with boron carbide for the determination of iodine in iodised salt in the concentration range of 10–210 mg kg^{-1}. A preconcentration NAA method was developed by Rao and Chatt for the determination of low levels of iodine in biological and nutritional materials using microwave digestion procedure (Rao & Chatt, 1991). Radiochemical NAA was used for the determination of nanogram amounts of iodine in samples of food (Rao & Chatt, 1993) and urine (Dermelj et al., 1992). Kucera, Randa, and Soukal (2001) determined iodine in food stuffs based on three different nuclear reactions ^{127}I (n, γ) ^{128}I, ^{127}I (n, 2n) ^{126}I and ^{127}I (γ, n) ^{126}I.

In the present work, an ENAA method using boron carbide (B_4C) filter was standardised for the first time in our reactor neutron irradiation facility to determine low concentrations of iodine in milk, milk powder, baby food, health drink powder samples and iodised salt. The method was applied for the estimation of iodine in five NIST standard reference materials (SRMs) namely 1549 (low fat milk powder), 1548a (typical diet), 1566a (oyster tissue), 1575a (pine needles), 1570 (spinach) and two IAEA reference materials (RMs) namely 59 (cabbage) and 153 (milk powder). The SRMs 1549, 1548a and 1566a, which have certified iodine concentrations, were used as the control samples to evaluate the accuracy of the ENAA method.

2. Material and methods

2.1. Material

Sub samples from five standard reference materials (SRMs) obtained from the National Institute of Standards and Technology (NIST) and two RMs obtained from International Atomic Energy Agency (IAEA) were used as received. Moisture contents in reference materials were determined by drying the samples as per the recommended procedures given by the respective agencies. Milk powder, fresh milk samples, infant food powders, health drink powders and iodised salt samples were bought from local supermarkets. Powder samples were sealed in airtight polythene bags. Fresh milk samples were stored in the refrigerator. Iodised salt samples were sealed in air tight polythene bags. AR grade KIO_3 (Merck) for iodine and salts of Na, K, Mn, Br and Cl were used as elemental standards.

2.2. Preparation of standards and samples

Elemental standards for I, Na, K, Mn, Cl and Br were prepared using the respective stoichiometric compounds. Aliquots of each standard solution were weight transferred onto separate discs of Whatman filter paper no. 42 and allowed for air drying. About 1 μg of iodine standard, prepared using AR grade KIO_3 (Merck), was used for the concentration calculation in the samples. Milk powder, health drink powder samples and infant food samples were homogenised by grinding in pestel and mortar, weighed accurately (mass about 50–100 mg) and sealed in polythene pouches. Fresh milk samples (1 mL) were air dried on filter paper discs and then sealed in polythene pouches. Iodised salt samples of about 10 mg each were sealed in polythene pouches. Accurately weighed samples of reference materials in the mass range of 50–100 mg were sealed in polythene pouches using the same procedure used for samples. Sealed samples along with the standards were placed in a cylindrical box of 3 mm thick boron carbide rubber (B_4C containing 50 wt% B), prepared at our institute (BARC), followed by wrapping with Teflon tape. Finally, these samples were triply sealed with polythene and then placed in a standard polyethylene bottle.

2.3. Sample irradiation and radioactivity measurement

Samples along with elemental standard for iodine were irradiated in D8 position of swimming pool type APSARA reactor, Bhabha Atomic Research Centre, India for durations of 30 min or 1 h. The thermal, epithermal and fast fluxes at the D8 position of APSARA reactor are of the order of 10^{12}, 1.4×10^{10} and 5×10^{10} cm^{-2} s^{-1}, respectively. After a cooling period of 15 min, samples and standards were mounted on the Perspex plates and the radioactivity was assayed for 10 min using a high-resolution gamma ray spectrometer consisting of 40% relative efficiency HPGe detector coupled with a PC based multichannel analyser (MCA) in a fixed sample to detector geometry. The counting system had an energy resolution of 1.8 keV at 1332 keV for ^{60}Co. In the present work, sample size was kept small and similar geometries for the sample and standard were maintained with respect to the detector. In a set of irradiation, samples and standards were counted at the same distance (5 cm or above from the detector depending on the dead time) with respect to the detector. Therefore, corrections for different sample geometries were not performed. The formation of ^{24}Na was reduced in the present studies since the samples including iodised salts were irradiated using B_4C filter to cut off the thermal neutrons. The dead time and pulse pile up effects were minimised by counting the samples at longer distances from the detector. The dead time of the detector was maintained at less than 5%.

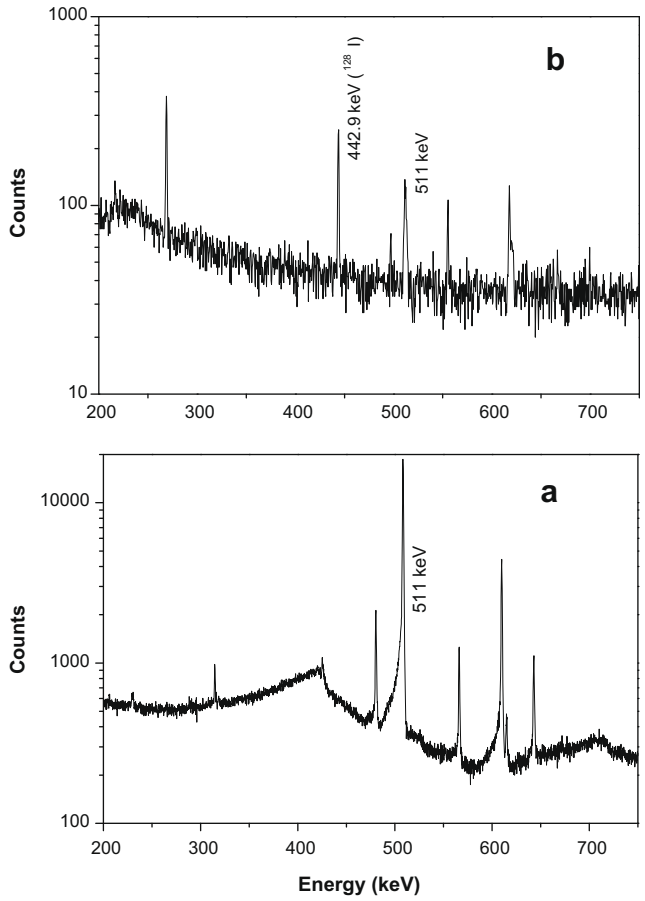

Fig. 1. Gamma ray spectra of neutron activated NIST SRM 1549 (non fat milk powder) using (a) whole reactor neutrons and (b) epithermal neutrons using B_4C filter.

2.4. Calculation of elemental concentrations

The peak area under the characteristic gamma ray peak at 442.9 keV due to ^{128}I was determined by peak fit software PHAST developed at BARC (Mukhopadhyay, 2001). The concentration calculation was done by the relative method of INAA. Using mass of iodine in standard ($m_{x,std}$) and count rates (counts per second, cps) of standard ($cps_{x,std}$) and sample ($cps_{x,sample}$), the mass of iodine present in the sample ($m_{x,sample}$) was calculated for the same counting period by the following equation:

$$m_{x,sample} = m_{x,std} \times \frac{cps_{x,sample}}{cps_{x,std}} \times \frac{D_{x,std}}{D_{x,sample}} \quad (1)$$

where D is the decay factor ($\exp(-\lambda t_d)$), λ is the decay constant, and t_d is the decay time. The $m_{x,sample}$ (μg) was converted to concentration (μg g^{-1} or mg kg^{-1}) by dividing with the sample mass (g). Boron ratio (R_B) of an isotope of an analyte in a given irradiation position of a reactor is defined as,

$$R_B = \frac{\text{Activity of the radioisotope without B or } B_4C \text{ filter}}{\text{Activity of the radioisotope with B or } B_4C \text{ filter}} \quad (2)$$

where activity is the mass normalised count rate. The advantage factors (AF) of iodine determination over other interfering elements was calculated by dividing the boron ratio of interfering element R_B (x) to the boron ratio of iodine R_B (I).

3. Results and discussion

The boron ratios (R_B) of the isotopes of Na, K, Cl, Mn, Br and I were determined using decay corrected and mass normalised net counts obtained under the characteristics peaks of radioisotopes (Table 1). Table 1 also gives the values of advantage factors (AF) for iodine over the interfering elements Na, K, Cl, Mn and Br and the corresponding values are 25.8, 20.0, 18.6, 8.7 and 1.2. This indicates that iodine can be determined by ENAA with better detection limits in presence of the interfering elements such as Na, K, Cl, Mn and Br. Gamma ray spectra of irradiated samples of NIST SRM 1549 (low fat milk powder) without and with B_4C filter are shown in Fig. 1. From this figure it is clear that iodine peak at 442.9 keV is clearly seen in the ENAA where as it is masked by the Compton background in INAA using whole reactor neutrons. Therefore, irradiations of all the other samples were carried out with B_4C filter. The determined iodine concentrations in seven RMs are given in Table 2. The determined concentrations of iodine in the NIST SRMs 1548a, 1549 and 1566a (0.74–4.58 mg kg^{-1}) are in good agreement (within 3%) with their certified values. The concentrations of iodine determined in other four reference materials of NIST and IAEA were in the range of 0.08 ± 0.01 to 4.23 ± 0.35 mg kg^{-1} for which certified values are not available. The uncertainties quoted in the results of reference materials (Table 2) are due to unweighted standard deviations at ±1s confidence limits arrived from four independent experiments ($n = 4$) except for SRM 1549 ($n = 7$). The % relative standard deviations are in the range of 1–13%. Table 3 gives the iodine contents in various food and food products like health drink powders, infant food samples, milk and milk powders. The iodine concentrations obtained in health drink powder and in-

Table 2
Iodine concentrations in some biological reference materials of IAEA and NIST.

Sample ID	Material	Mean value ± SD (mg kg^{-1})	Certified value (mg kg^{-1})	Detection limit (mg kg^{-1})
IAEA RM 359	Cabbage	4.23 ± 0.35	NA	0.2
IAEA RM 153	Milk powder	1.59 ± 0.21	NA	0.5
NIST SRM 1575a	Pine needles	0.08 ± 0.01	NA	0.04
NIST SRM 1570	Spinach	0.87 ± 0.06	1.16 ± 0.04[b]	0.1
NIST SRM 1549	Low fat milk powder	3.40 ± 0.03	3.38 ± 0.03	0.3
NIST SRM 1548a	Typical diet	0.74 ± 0.02	0.759 ± 0.103	0.2
NIST SRM 1566a	Oyster tissue	4.58 ± 0.14	4.46 ± 0.42	0.4

SD – unweighted standard deviation at ±1s confidence limit, and NA – Not available.
[b] Literature value by Rao and Chatt (1991).

Table 3
Iodine concentrations in different samples of food and food products.

Sr. no.	Sample	Concentration (mg kg^{-1})	Detection limit (mg kg^{-1})
1	Health drink 1	0.28 ± 0.02	0.07
2	Health drink 2	0.41 ± 0.02	0.08
3	Health drink 3	0.43 ± 0.03	0.10
4	Health drink 4	0.36 ± 0.03	0.06
5	Baby food 1	2.79 ± 0.11	0.20
6	Baby food 2 (wheat)	0.79 ± 0.08	0.60
7	Baby food 3 (rice)	0.74 ± 0.07	0.50
8	Low fat slim milk	0.16 ± 0.01	0.03
9	High fat (buffalo milk)	0.24 ± 0.02	0.03
10	Low fat (cow milk)	0.20 ± 0.01	0.02
11	Infant milk powder	3.60 ± 0.16	0.4
12	Milk powder brand 1	5.50 ± 0.18	0.5
13	Milk powder brand 2	4.45 ± 0.19	0.5

fant food samples were found to be in the range of 0.28–2.79 mg kg^{-1}. The iodine concentrations obtained in milk and milk powder samples were found to be in the range of 0.16–5.5 mg kg^{-1}. The results of iodine concentrations determined in seven brands of iodised salts are given in Table 4 and the concentrations were found to be in the range of 10–33 mg kg^{-1}. Using ENAA, we could determine low concentrations of iodine reliably in a number samples including iodised salts. The uncertainty evaluation on iodine concentrations for samples (Tables 3 and 4) was carried out using counting statistics and peak fitting errors on the peak area of 442.9 keV of ^{128}I of sample and standard and uncertainties on sample and standard masses. The uncertainties due to counting statistics and peak fitting errors of samples of food and food products and iodised salts are in the range of 3–10% (Table 3) and 5.5–18.7% (Table 4), respectively, whereas the same for iodine standards are in the range of 1–2%. Uncertainties due to sample masses are in the range of 0.01–1%. Uncertainties due to other parameters like differences of sample and standard geometries during irradiation and counting and concentration of standard were negligible with respect to counting statistics and thus not included in the uncertainty evaluation. The combined uncertainties on iodine concentrations are in the range of 3–10% for food and food products (Table 3) and 6–19% for iodised salt samples (Table 4).

The daily dietary intake (DDI) of iodine calculated from the concentration of iodine in the baby foods, milk, milk powders, health drink powders and iodised salt samples are given in Table 5. The table also gives average consumption per day of these food and food products. The DDI of iodine in these samples are in the range of 7.4–126 μg where 7.4 μg is the DDI for health drink powders and 126 μg is the DDI for iodised salt. For iodised salt the total iodine content of 18 mg kg^{-1} was arrived from the average of iodine contents in seven iodised salt samples (Table 4). The results indicate that the consumption of infant food 1 or infant foods 2 and 3 can provide adequate amount of iodine since the recommended dietary allowance (RDA) of iodine for infants is <100 μg per day. The RDA of iodine for adults is 150–200 μg per day. On the assumption that adults consume either milk or milk powder, health drink powders and iodised salt, the DDI was calculated as 173.4 μg for milk, health drink powders and salt and as 178.4 μg for milk powder, health drink powders and salt. The consumption of these food products can provide adequate amount of iodine to adults. In addition, salts from other food materials might add to this value. In such cases, the salt content may have to be reduced for a balanced diet vis-a-vis salt content.

The detection limit (L_D) was calculated using Currie's formula, $L_D = 2.71 + 3.29\sigma_b$, where σ_b is the square root of the background counts under the characteristic photo-peak of interest (Currie, 1968). The L_D (counts) was converted to L_D (μg) and L_D (mg kg^{-1}) by dividing with sensitivity of iodine (S) and sample mass, respectively. The L_D values are in the range of 0.02–0.6 mg kg^{-1} for the reference materials and samples of food and food products (Table 3). The higher detection limits of iodine in samples could be attributed to enhanced background due to sample matrix and longer decay time of counting. It was observed that the L_D for milk, health drink powders and infant foods were superior since the expected salt contents are lower in these samples and hence the spectral interference is minimal. The detection limits for iodised salt samples were about 5 mg kg^{-1}. Detection limits for iodine can further be improved by suitably choosing the experimental conditions like irradiating for longer periods and using a higher efficiency detector for measurements so as to enhance sensitivity. Additionally, detection limit can be improved by the use of Compton suppressed gamma ray spectrometry in which there is a substantial reduction of Compton background.

Table 4
Iodine concentrations in seven brands of iodised salts.

Sr. no.	Iodised salt	Concentration (mg kg^{-1})	Quoted value (mg kg^{-1})
1	Brand 1	11.14 ± 1.35	>15
2	Brand 2	14.24 ± 2.69	>15
3	Brand 3	18.31 ± 1.40	>15
4	Brand 4	9.85 ± 1.06	>15
5	Brand 5	32.60 ± 1.93	>30
6	Brand 6	15.85 ± 2.25	>15
7	Brand 7	23.03 ± 2.56	>15

Table 5
Daily dietary intake (DDI) of iodine from samples of food, food products and salts.

Sr. no.	Sample	Iodine concentration (mg kg^{-1})	Consumption per day	DDI of iodine (μg)
1	Baby food 1	2.79	2 servings of 20 g each	112
2	Baby foods 2 and 3	0.76	2–4 servings of 20 g each	15–30
3	Milk	0.20	1 glass (200 mL)	40
4	Milk powder	4.5	2 cups (5 g per cup)	45
5	Health drink powders	0.37	2 cups (10 g per cup)	7.4
6	Iodised salt	18	10 g (30% loss in cooking)	126

4. Conclusions

An epithermal NAA method using boron carbide as thermal neutron shielding was optimised for the determination of trace levels of iodine. It was used to determine iodine in various samples like milk, milk powder, baby foods, health drink powders and iodised salt. The iodine concentrations obtained in NIST SRMs 1548a, 1549 and 1566a were found to be within 3% of their certified values, reflecting the accuracy of the ENAA method. The iodine concentrations were also determined in four reference materials of NIST and IAEA, where certified values are not available. The detection limits of iodine were in the range of 0.02–0.6 mg kg^{-1} for various foodstuffs whereas the detection limit for iodised salt samples was about 5 mg kg^{-1}. The method is simple, non-destructive and quick to arrive at iodine concentration even in the presence of high salt matrix like NaCl.

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