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Determination of Iodine Content in Saudi Milk Products

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Abstract. Low level concentration of iodine was determined in various milk products for adult and baby milk powders by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) method. It is a reliable method for the determination of iodine in milk samples, using alkaline digestion with potassium hydroxide KOH solution in an oven. After digestion, a stabilizer is added and the solution is taken to volume, then filtered and analysed by ICP-MS either directly or after dilution. Samples for investigation were collected from domestic market. The detection limits of current Iodine not affected by interfering from milk gradient, the minimum detection limit (MDL) of Iodine approaches 10 ppb was achieved. This method showed excellent results for aqueous iodide solutions, although the complex milk digest matrix made the method unsuitable for such samples. So, investigation of the iodine species is achieved through the oxidation and extraction of iodine milk samples, the digestion was carried out to control the iodine chemistry. Iodine concentrations were ranged of 0.17 – 5.1 mg/kg for various samples, the accuracy of the method ranged from 95 to 100%.

Keywords: *Iodine deficiency/ ICP-MS/ Iodine/ Milk Powders.*

1. Introduction

Milk is recognized as the most complete food in the human diet because it contains all the macronutrients such as proteins, lipids and carbohydrates and all the essential micronutrients such as elements, vitamins and enzymes. A lot of information has been accumulated concerning the composition of milk in terms of fat, protein and vitamins [1]; however, less attention has been paid to the elemental composition of milk in spite of the great importance of essential elements in nutrition [2,3]. Iodine is one of the most important trace elements in human nutrition; its physiological function as a constituent of thyroxine, the hormone secreted by the thyroid gland, necessitates the control of intake levels, as deficiency or excessive exposure both have a detrimental effect on thyroid function [4]. Concentrations of iodine in cow's milk, which is a major contributor to dietary exposure, are naturally influenced by the levels of iodine in feedstuffs, which vary seasonally [5].

It has been reported that iodine in milk is present mostly as free iodide [6] with relatively small amounts of organic iodine. Whilst ^{127}I is the only stable isotope, the most hazardous iodine species are the radioactive isotopes such as ^{129}I and ^{131}I which may enter the food chain via the air grass cow milk pathway as a result of aerial emissions from nuclear reprocessing plants [7]. The long-lived radionuclide ^{129}I is monitored by regular measurement of ^{129}I content of liquid milk from cows in the potentially affected areas [8,9], typically by radiometric analysis after appropriate separation techniques [1-4].

All mental retardation and brain damage due to iodine deficiency can be prevented if iodine supplementation prescribed duly on time, as recommended by WHO, the safe and adequate dietary intake of iodine for infants to adults ranges from 50 to 200 μg per day [5]. The major part of the essential iodine enters into the human body through food products. However, its excessive iodine intake can lead to thyroiditis. In addition, thyroglobulin can be measured on dried blood spots to provide an additional sensitive functional biomarker of iodine status [6, 7]. Iodine deficiency can be controlled through the fortification of food products with added iodine and also through addition of iodized 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 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 [8].

Accurate quantitation of iodine in biological samples is essential for studies of nutrition and medicine, as well as for epidemiological studies for monitoring intake of this essential nutrient. Some of the commonly used analytical techniques for determining iodine concentrations in environmental and biological samples are colorimetry, potentiometry, isotope exchange, gas chromatography (GC), catalytic spectrophotometric methods, namely those based on the Sandell–Kolthoff reaction, [9]. Despite the importance of accurate iodine measurement, a standardized method for iodine analysis of biological samples is yet to be established [9]. However, inductively coupled plasma mass spectrometry (ICP-MS) potentially offers a quick, simple

method of monitoring iodine isotopes in milk; however there are a number of problems associated with the use of ICP-MS for this assay. Firstly, iodine has a relatively high detection limit in ICP-MS in comparison to other elements due to its high ionisation potential (10.45 eV). Also, signal memory effects can be a problem, due to evaporation of iodine as HI or I2 from aerosol droplets in the spray chamber typically by radiometric analysis after appropriate separation techniques [10]. Iodine (atomic weight 126.9 g/atom) is an essential component of the hormones produced by the thyroid gland. Thyroid hormones, and therefore iodine, are essential for mammalian life. The native iodine content of most foods and beverages is low, and most commonly consumed foods provide 3–80 mg per serving. Major dietary sources of iodine in the world are bread and milk. Iodine content in foods is also influenced by iodine-containing compounds used in irrigation, fertilizers, livestock feed, dairy industry disinfectants and bakery dough conditioners [6]. Recommendations for iodine intake by age and population group are shown in Table(1).

Table (1). Recommended dietary allowance (RDA) of iodine intake (mg/day) by age or population group

Age or population group	U.S. Institute of Medicine [a] [10]	Age or population group	World Health Organization [b] [11]
Infants 0–12 months	110–130	Children 0–5 years	90
Children 1–8 years	90	Children 6–12 years	120
Children 9–13 years	120		
Adults ≥14 years	150	Adults ≥12 years	150
Pregnancy	220	Pregnancy	250
Lactation	290	Lactation	250

[a]Adequate Intake for infants ≤12 months; Recommended Daily Allowance for children >1 year.

[b]Recommended Nutrient Intake.

Table (2) shows the prevalence of goitre among two thousand and seven hundred children from six to twelve years were clinically examined for the presence of goitre during survey carried out by Chaudhary et al. [12]. Of these, 53% were males and 47% were females. The mean age of the children vary between 8.88 ± 1.83 years. Prevalence of goitre increased with that of age. The highest prevalence (%) was observed in the age group of 11-12 years. Overall goitre prevalence rate was 12.6%. Prevalence was significantly higher among females than males (P=0.0003) (Table 2). Age-specific prevalence rate (ASPR) was higher in 9 to 12 yr. age group as compared to 6 to 8 yr. age group No. case of nodular goitre was observed Table (2).

Table (2). Age and sex-wise prevalence of goitre among study subjects (*N*=2700).

Goitre grades	*Age in years*						*Total No.(%)*
	6-8 years No. (%)		*9-10 years No. (%)*		*11-12 years No. (%)*		
	female	*Male*	*Female*	*Male*	*female*	*male*	
Grade 0	557 (20.6)	580 (21.5)	296 (10.9)	348 (12.9)	223 (8.3)	356 (13.2)	2360 (87.4)
Grade I	42 (1.5)	38 (1.4)	48 (1.8)	32 (1.2)	67 (2.5)	44 (1.6)	271 (10)
Grade II	14 (0.5)	11 (0.4)	13 (0.5)	9 (0.3)	10 (0.4)	12 (0.4)	69 (2.6)
Total	613 (22.7)	629 (23.3)	357 (13.2)	389 (14.4)	300 (11.1)	412 (15.2)	2700 (100)

Chi square– 16.09, p- value - 0.0003 [12].

Inductively coupled plasma mass spectrometry (ICP-MS) potentially offers simple method of monitoring iodine isotopes in milk, its advantages are quick, and the selectivity, sensitivity is excellent. However, there are a number of problems associated with the use of ICP-MS for this assay. Firstly, iodine has a relatively high detection limit in ICP-MS in comparison to other elements due to its high ionization potential (10.45 eV). Also, signal memory effects can be a problem, due to evaporation of iodine as HI or I_2 from aerosol droplets in the spray chamber [13, 14]. Furthermore, interference on the $^{129}I^+$ signal arises from $^{129}Xe^+$ and possibly also $^{127}IH^{2+}$ ions [15-17], making the use of oxygen as a collision/reaction gas an attractive option for this analysis. Before considering instrumental conditions, however, it is necessary to carefully consider sample preparation, to avoid losses due to iodine's volatility and complex redox chemistry. At low pH iodide is easily oxidized to volatile molecular iodine by dissolved oxygen (or other dissolved oxidants):

$$4I^- + O_{2(g)} + 4H^+ \rightarrow 2H_2O + 2I_{1(aq)} \quad E^o = 0.61\ V \tag{1}$$

At high pH, the oxidation of iodide to iodine is avoided, and it is therefore usual to prepare samples in alkaline media to prevent the oxidation of I^- to I2 or the formation of HI. With milk samples, it is also important to destroy the organic matrix to reduce the spectral interferences from carbon species and the possibility of cone blockage in the ICP-MS interface. Thus decomposition with strong alkali, such as ammonia, potassium hydroxide KOH solution in an oven or by using an open-vessel microwave system, has been used to prepare milk samples for iodine determination [16, 18, 19]. These procedures lead to the conservation of the iodine as iodide or iodate, which is then determined by ICP-MS regardless of the iodine species present in the original sample. The use of alkaline conditions also potentially enables the simultaneous determination of other important anion-forming elements such as selenium, arsenic, sulfur and molybdenum, though this aspect appears to have received little attention in the literature, iodine usually being determined separately from other trace elements. However, issues also including the tendency of iodine to absorb onto glass must be carefully controlled for the successful

determination of low levels of iodine in food. In the presence of acid, iodine may form molecular iodine, which will cause memory effects and background problems due to its absorption onto glass. Consequently, the analysis of total iodine in milk using ICP-MS is best performed after sample digestions and dilutions have been performed in dilute base [16, 18, 19].

For iodine determination approach is to deliberately convert iodine in the sample to elemental iodine vapor for analysis, and total iodine has been determined in milk by vapor generation ICP-optical emission spectrometry [20], though an alkaline digestion was still required to destroy the organic matrix prior to generating the iodine vapor. Samples are digested and brought to final volume in single use iodine-free vessels; thus saving procedural time while completely eliminating potential contamination from vessels used with previous samples.

This procedure allows a rapid return to background level during sample analysis, which allows reproducibility of a very low calibration standard (0.250 μg/l) and instrument results at a rate of approximately every 100 s without concern of carryover when the samples read on the calibration curve (top calibration standard of 100 μg/l). The use of closed vessel microwave digestions, with tetramethylammonium hydroxide may cause a vessel to burst because of over pressurization due to decomposition at temperatures above *90°C* (20). The oven digestion procedure in this method, utilizing a KOH solution at *105°C* for about 1 h eliminates the dangers surrounding high vessel pressure, while producing excellent precision and accuracy.

Iodine has been determined directly in milk powder by electrothermal vaporization-ICP, but again interference from the organic matrix was a problem, necessitating the use of pre-reduced Pd as a chemical modifier and internal standard [3, 15]. This work describes the preparation of milk samples for the determination of iodine by ICP-MS. A straightforward digestion method to break down the organic matrix of the milk has been done, without loss of analyte, yielding a clear solution suitable for continuous nebulisation and analysis by ICP-MS. Once this was achieved, attention turned to pre-concentration of the digest before insertion to the ICP-MS to improve detection limits.

2. Experimental

2.1 Material and methods

Adult Milk powder (high fat and low fat), baby and infant milk powders and liquid milk of high fat, low fat samples were bought from local supermarkets. Powder samples were sealed in airtight polyethylene pouches. ANALAR grade of the following c*hemicals and reagents:*

(a) KOH (KOH) pellets, certified ACS. KOH may contribute background levels of iodine.

(b) KOH solution -50% (w/v).

(c) Ammonium hydroxide NH4OH, Certified ACS,

(d) Sodium thiosulfate ($Na_2S_2O_3$). 99.99+% metal bases.

(e) Surfactant (i. e. Triton® X-I 00).

(t) Nitric acid (HNO_3), High purity.

(g) Perchloric acid ($HClO_4$), High purity.

(h) Purified water, 18 MΩ/cm.

(i) Iodine stock standard solutions, Certified ICP-MS or ICP-grade single or multi-element standard solutions (or other certified reference materials (CRM) are used to prepare calibration, calibration verification standards internal standards, and spiking solutions.

When, applicable, choose elements for internal standards with ±40 atomic mass units (amu) from the mass to be quantified. Likely choices for use as internal standards for iodine analysis are praseodymium (Pr), samarium (Sm), tellurium (Te), and rhodium (Rh). Concentrations used for analysis are 30.0 ppb Pr, Sm, Rh, and 500 ppb Te. The internal standard solution reagent's concentration is 2% HNO_3, 0.1 % $HClO_4$, 0.01 % Triton X-100, 0.25 % KOH, 0.1 % NH_4OH, and 0.01 % $Na_2S_2O_3$ in purified water.

2.2 Digestion procedure

Several liquid and dried milk samples were obtained from local stores. Weigh approximately 5.0 gm of sample (or 30 gm liquid milk) into an appropriate vessel (150 ml or 250 ml beaker) and record the sample weight. Without zeroing the balance, add water to make approximately 100 gm. Record the sample water weight. Place a stir bar in the mixture and stir on a stir plate to form a homogenous slurry/suspension. While stirring, weigh 5-10 gm of the slurry/suspension into an appropriate digestion vessel, add approximately 10 ml water and then proceed with the addition of the KOH as stated below.

For the testing of other matrixes, visually evaluate the sample for homogeneity before weighing an appropriate amount. If a sample does not appear to be homogenous perform additional homogenizing (i.e. blending, grinding, etc.,). If applicable, the sample may be reconstituted. Accurately weigh or aliquot an appropriate amount (0.25 to 2.50 gm or 0.50 to 10 ml) of sample into a labelled 100 ml digestion vessel. Add 20 ml purified water to the vessel. Accurately weigh an appropriate amount (0.25 to 1.00 g) of an appropriate CRM, i.e., National Institute of Standards and Technology Standard Reference Material (NIST SRM) 1549 or 3280, if applicable in the same manner as the samples. SRM 1549 may be digested using either 5 or 50% KOI solution. SRM 3280 should be digested using only the 50% KOH solution.

Designate at least one digestion vessel as the digest blank. The digestion blank(s) should be treated in the same manner as the samples. If both the 5 and 50% KOH solutions will be used, prepare at least one blank with each concentration.

Place an aliquot of spiking solution (if applicable) into an appropriately labelled digestion vessel. Add either 10 ml 5% KOH solution or 10 mL 50 % KOH solution to each digestion vessel use the guideline previously mentioned for guidance on which solution to use. It worthwhile to mention that, if the values of

iodine concentration below < 10 000 μg/kg are anticipated, add 5 ml of 5 % KOH solution, then dilute to 50 ml. Samples expected to contain levels of iodine below <10000 μg/kg may be digested using the 5% KOH solution. However, if samples are expected to contain > 10 000 μg/kg iodine and are anticipated to be detectable after an appropriate dilution, the 50 % KOH solution may be used. Seal the vessels and swirl or use a vortex apparatus to mix. Avoid inverting as this may allow sample to adhere to the inner walls of the vessel above the level of the digestion solution. Digest samples in an oven set to maintain 105 ± 5 °C until the dissolution of iodine is complete, approximately 1 h. Beware the digestion vessels may either be tightened completely or loosened slightly while in the oven.

After removal from the oven, add 2 ml stabilizer concentrate, then allow the samples to cool before bringing to volume with purified water. Alternatively, allow samples to cool first, then add 2 ml stabilizer concentrate and bring to volume with purified water. *Note:* If the final volume will be 50 ml add 1 ml stabilizer concentrate. Cap the vessels, and then invert to mix thoroughly.

Filter the sample solution by filling a disposable syringe with the digested sample solution, attach a 1 μm membrane filter, then filter an adequate amount (i.e., several milliliters) into appropriate vessel (i.e.) 15 ml PP centrifuge tube to be used for analysis. Store samples at ambient temperature, samples may be stored at ambient temperature indefinitely, as long as the results for the applicable digest blank(s) and/or control sample(s) are acceptable when analysed.

2.3 Instrumentation

Samples along with elemental standard for iodine were irradiated using Mass spectrometric measurements were performed using a double-focusing magnetic-sector field ICP-MS (Finnigan Element2, Bremen, Germany). Aqueous solutions were introduced into the ICP-MS with a SC-4 auto-sampler, an Apex nebulizing system connected to an ACM desolvator and a self-aspirating PFA-ST-nebulizer (all from Elemental Scientific Incorporation, Omaha, USA). The sample first introduced to an argon plasma torch using a nebulizer spray chamber. The argon plasma transforms all constituents to ionized elements. The ions are passed through a vacuum interface through a focusing lens and into the quadrupole mass separator. The ions are focused onto a detector that counts individual events at a particular charge-to-mass ratio. Commercial instruments have the capability of scanning the mass range from 1 to 240 atomic mass units (amu) in a few tenths of a second and achieving detection limits in the 0.1 to 1 ppb range during that time. The whole system includes the following components and accessories:

(a) Polypropylene (PP) tubes, assorted sizes, use as received.

(b) Oven (i.e., warming/drying oven).

(c) Open-vessel microwave digestion unit (optional).

(d) Analytical and top-loader balances, sensitive to 0.0001 and 0.01 g, respectively.

(e) ICP-MS system.

(f) Auto sampler for ICP-MS.

(g) Adjustable (electronic or manual) volumetric pipets and pipet tips.

(h) Re-pipet volumetric dispenser, with adjustable volume.

(i) Polypropylene or Teflon bottles or storage of reagents.

(j) Disposable plastic syringes.

(k) Syringe filters with 1 μm membrane.

2.4 Sample Analysis

The digested samples are analyzed directly or diluted so that the iodine concentration will fall within the calibration range. Samples digested with 50 % KOH solution must be diluted 1 to 10 ml to achieve the desired final concentration of 0.5% KOH. Aliquot 1 ml of the filtrate into an appropriate vessel (i.e., 15 ml PP centrifuge tube), add 0.18 ml stabilizer concentrate, and then dilute to 10 ml with purified water. *Note:* If samples digested with 50 % KOH solution need more than a 1 to 10 ml dilution to obtain a reading on the calibration curve, an additional dilution must be prepared from the original 1 to 10 ml dilution. Aliquot the desired amount into an appropriate vessel (i. e., 15 or 50 ml PP centrifuge tube), then dilute to volume with diluent.

Samples digested with 5 % KOH solution may be diluted (if necessary) by placing an aliquot of the filtrate into an appropriate vessel (i.e., 15 ml PP centrifuge tube), then diluted to an appropriate volume with diluent.

Condition the ICP-MS sample introduction system. Analyze conditioning solution while concomitantly introducing internal standard solution on-line through a mixing block until conditioned (approximately 1 h). The internal standard solution is introduced via a peristaltic pump using orange/green two stop PVC pump tubing (0.38 mm id). After conditioning, begin to aspirate carrier solution while continuing to add internal standard. Analyze samples using lCP-MS.

Potential interferences are available for consideration from within the ELAN® instrument software once an analyte is selected. For the isotopes chosen, no potential interferences were listed. The results presented in this paper were obtained by using praseodymium as the internal standard. The other internal standards listed above were evaluated during method development; however, praseodymium was chosen over the others because it produced results closer to expected values (i.e., NIST reference materials).

Chemical and/or physical interferences from matrixes containing high levels of salt may create suppression or enhancement of signal resulting in erroneous values. Ruggedness of this method, with respect to high salt matrixes, was demonstrated by analyzing sodium chloride.

3. Results and discussion

After this method, was developed it was exposed to a rigorous single laboratory validation study. It was evaluated for LOD, LOQ, linearity, precision,

accuracy, specificity, ruggedness, and robustness. The validation was performed on NIST SRM 1549 Nonfat Milk Powder, NIST SRM 3280 Multivitamin/Multielement Dietary Supplement Tablets, and a powdered infant formula. The method demonstrated exceptional performance with each of these matrixes, under all these categories. This procedure has been used successfully for the determination of iodine in a wide variety of foods and dietary supplements.

During method development, it was discovered that both the oven and microwave digestion techniques produced acceptable results. However, oven digestion is preferred over microwave digestion for the following reasons: (1) Depending on the size of the oven, a greater number of samples (typically 50) may be placed into an oven, whereas the microwave can only hold a finite number of samples (typically 30). (2) The digestion vessels placed in an oven can be completely sealed. The sample digestion vessels placed in a microwave oven are not sealed completely to avoid excessive pressure buildup. (3) Digestion vessels placed in a conventional oven may be left unattended without any potential loss of sample. In a microwave, even with appropriate power and time settings, a sample may superheat and melt a hole in the polypropylene digestion vessel, creating loss of sample. (4) The conventional oven procedure affords the use of 100 ml digestion vessels, allowing larger sample sizes to be weighed. The microwave oven turntable usually only uses 50 ml digestion vessels

3.1 Determination of Iodine concentrations

This method was applied successfully to a wide variety of milk powders. The samples were chosen to demonstrate the method applicability across the major milk categories. Table (4) gives the iodine contents in various milk powders, baby, infant, and adult milk powders. The iodine concentrations obtained in baby milk powder and infant milk powders samples were found to be in the range of 0.17–5.1 mg kg^{-1}. The results of iodine concentrations determined in twelve brands of milk powder and three samples of liquid milk are given in Table (4). The iodine concentrations were found by utilizing of ICP-MS technique, we could determine low concentrations of iodine reliably in a various samples, especially that including milk powder with enriched nutrition.

The uncertainty evaluation on iodine concentrations for samples table (4) was carried out using counting statistics and peak fitting errors on the Iodine peak area of sample and standard and uncertainties on sample and standard masses. The uncertainties due to counting statistics and peak fitting errors of samples of milk samples are in the range of 3–5% and 5–7% for milk powders, 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 3–6% for milk samples and 6–7% for milk powders.

The detection limit (L_D) was calculated using Currie's formula [21], the generic formulas which define the critical level decision L_C to determine threshold

value and minimum detectable limit L_D to determine minimum value of detection, as hypothesized in Eq. (3):

$$L_D = L_C + K_\beta \cdot \sigma_D \,;\; L_C = K_\alpha \cdot \sigma_o \tag{3}$$

where σ_0 is the standard deviation of the signal when it equals 0, σ_D is the standard deviation of the net signal counts under the characteristic photo-peak of interest when it equals L_D, and K_α and K_β are the quantiles of the standard normal distribution for the probability α and β, respectively, then $L_D = 2.71 + 3.29\sigma_D$ [22]. The L_D (counts) 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 0.10–0.35 mg kg^{-1} for the reference materials and samples of milk powders table (4). 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 samples of liquid milk and milk powders were superior since the expected iodine contents are lower in these samples and hence the spectral interference is diminished. Detection limits for iodine can further be improved by suitably choosing the experimental conditions like irradiating for longer periods, using a higher efficiency detector for measurements, selecting proper parameters for decay and counting times, all have an enhancing draw back on sensitivity.

Table (4). Iodine concentrations in different samples of milk powders.

No.	Sample	Concentration (mg kg^{-1})	Detection limit (mg kg^{-1})
1	Baby milk powder 1	2.82 ± 0.12	0.10
2	Baby milk powder 2	2.27 ± 0.11	0.10
3	Baby milk powder 3	2.11± 0.11	0.10
4	Baby milk powder 4 (wheat)	0.90 ± 0.07	0.25
5	Baby milk powder 5 (rice)	0.75 ± 0.09	0.30
6	Infant milk powder	4.32 ± 0.16	0.20
7	Low fat milk powder	3.42 ± 0.05	0.15
8	Milk powder brand 1	3.54± 0.15	0.35
9	Milk powder brand 2	4.53 ± 0.17	0.35
10	Milk powder brand 3	1.60 ± 0.22	0.35
11	Milk powder brand 4	5.10 ± 0.19	0.35
12	Milk powder brand 5	4.58± 0.24	0.35
13	High fat (buffalo milk)	0.26 ± 0.03	0.15
14	Low fat (cow milk)	0.22 ± 0.01	0.10
15	Low fat slim milk	0.17 ± 0.02	0.15

3.2 Determination of daily dietary intake (DDI) of iodine

The daily dietary intake (DDI) of iodine calculated from the concentration of iodine in the baby milk powders samples are given in table (5). The table also gives average consumption per day of these milk powders. The DDI of iodine in these samples are in the range of 72–108 μg where 72 μg is the DDI for low fat liquid

milk and 108 μg is the DDI for infant milk powder. The results indicate that the consumption of infant milk powders can provide adequate amount of iodine since the recommended dietary allowance (RDA) of iodine for infants is 120 μg per day. The RDA of iodine for adults is 150 μg per day (table 1). On the assumption that adults consume either milk or milk powder, the DDI was calculated as 96 μg and 99 μg for milk and milk powder in addition to diary nutrition mails. The consumption of these milk powders products can provide adequate amount of iodine to adults.

Table (5). Daily dietary intake (DDI) of iodine from considerable samples of milk and milk powders

No.	Sample	Iodine concentration (mg kg^{-1})	Consumption per day	DDI of iodine (μg)
1	Baby milk powders (1-3)	2.42	2 servings of 20 g Each	96.8
2	Baby milk powders (4-5)	0.83	2–4 servings of 20 g each	33.2–66.4
3	Infant milk powder	4.31	1 cups (25 g per cup)	108
4	Low fat milk powder	3.42	1 cups (25 g per cup)	86
5	Milk powder brand (1-5)	3.96	1 cups (25 g per cup)	99
6	High fat milk (buffalo milk)	0.24	2 cups (200 mL)	96
7	Low fat milk (cow and slim)	0.17	2 cups (200 mL)	72

Conclusions

ICP-MS technique was used for the determination of trace levels of iodine content in a variety of milk samples. ICP-MS is a simple, rapid and routine method. One major advantage is that the samples can be measured directly without further pretreatment. It was used to determine iodine in various samples like milk, milk powder, baby milk powders, diet milk powder and milk powders with enriched nutrition. The minimum detection limits (MDL) of Iodine levels as low as 10 ppb can be measured using this technique, even in the presence of high nutrition matrix. Iodine concentration was in the range of 0.17–5.1 $mg \cdot kg^{-1}$ for various milk and milk powders. The accuracy of the method ranged from 95 to 100%. This method has been established its use commonly to a wide variety of dietary foods and supplements. In the future, hope to study the analysis of iodine content using other techniques such as neutron activation analysis.

References

[1] Parry, S J, et al. The Science of the total environment 173-174 351 (1995) p. 351
[2] Sullivan, D and Zywicki, R J AOAC Int 95 195 (2012) p. 195
[3] Fernandez-Sanchez, L M, Bermejo-Barrera, P, Fraga-Bermudez, J M, Szpunar, J and Lobinski, R J Trace Elem Med Biol 21 Suppl 1 10 (2007) p. 10
[4] Mesko, M F, et al. Anal Bioanal Chem 398 1125 (2010) p. 1125
[5] Clifton, V L, et al. Nutrition Journal 12 32 (2013) p. 32
[6] Zimmermann, M B and Andersson, M Nutrition Reviews 70 553 (2012) p. 553
[7] Charlton, K E, Jooste, P L, Steyn, K, Levitt, N S and Ghosh, A Nutrition (Burbank, Los Angeles County, Calif.) 29 630 (2013) p. 630
[8] Zhang, L, et al. BMC Neuroscience 13 121 (2012) p. 121
[9] Dyke, J V, Dasgupta, P K and Kirk, A B Talanta 79 235 (2009) p. 235
[10] Institute of Medicine, P o M Dietary reference intakes for vitamin A, vitamin K, arsenic, boron, chromium, copper, iodine, iron, manganese, molybdenum, nickel, silicon, vanadium, and zinc. (Washington, D.C.: National Academy Press) (2002)
[11] International Council for Control of Iodine Deficiency Disorders, U W H O Assessment of iodine deficiency disorders and monitoring their elimination : a guide for programme managers. (Geneva: World Health Organization) (2007)
[12] Chaudhary, C, Pathak, R, Ahluwalia, S K, Goel, R K D and Devgan, S Indian Pediatrics 50 587 (2013) p. 587
[13] Moreda-Piñeiro, A, Romarís-Hortas, V and Bermejo-Barrera, P Journal of Analytical Atomic Spectrometry 26 2107 (2011) p. 2107
[14] Grinberg, P and Sturgeon, R E Journal of Analytical Atomic Spectrometry 24 508 (2009) p. 508
[15] Izmer, A V, Boulyga, S F and Becker, J S Journal of Analytical Atomic Spectrometry 18 1339 (2003) p. 1339
[16] Hou, X, et al. Analytica Chimica Acta 632 181 (2009) p. 181
[17] Zhang, S, et al. Environmental science & technology 44 9042 (2010) p. 9042
[18] Schramel, P and Hasse, S Microchimica Acta 116 205 (1994) p. 205
[19] Gélinas, Y, Krushevska, A and Barnes, R M Analytical chemistry 70 1021 (1998) p. 1021
[20] Čmelík, J, Machát, J, Otruba, V and Kanický, V Talanta 80 1777 (2010) p. 1777
[21] Calmet, D, Herranz, M and Idoeta, R Journal of Radioanalytical and Nuclear Chemistry 276 299 (2008) p. 299
[22] Currie, L A Analytical Chemistry 40 586 (1968) p. 586

تحديد محتوى اليود لمنتجات اللبن في السعودية

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سيكلوترون أنشاص، قسم الفيزياء، مركز البحوث النووية، هيئة الطاقة الذرية، كود بريدي ١٣٧٥٩،
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قسم الفيزياء، كلية التربية بالزلفي، جامعة المجمعة، مبنى ٨٥٨٩ الزلفي ٢٥٨٨-١٥٩٤٢، المملكة العربية السعودية
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ملخص البحث. تم تحديد مستوى تركيز اليود المنخفض في منتجات الحليب المختلفة البودرة والسائل المستخدم للأطفال والكبار من استخدام طريقة المطياف الكتلي المستحث بالبلازما (ICP-MS). وتم جمع العينات للدراسة من السوق المحلي، بواسطة التقنية المستخدمة في هذه الدراسة هناك حاجة لمعالجة تحضيرية للعينات، حيث تتم معالجة العينات في الفرن لتتحلل في محلول قلوي مائي من هيدروكسيد البوتاسيوم ثم يتم اضافة عامل استقرار وتخفف العينة وتحلل بواسطة مطياف الكتلة، وتبين ان الحدود للكشف الدنيا عن اليود لم تتأثر بأي تداخل نتيجة التركيب العناصري للحليب, ووصل الحد الأدنى للكشف الى عشرة أجزاء من المليون، ، وقد أجريت عملية الهضم بها للسيطرة على الكيمياء اليود، وتراوحت تركيزات اليود من ١,٧ الى ٥,١ ملجم.كجم-١ في العينات المختلفة ، و دقة الأسلوب تراوحت ٩٥-١٠٠ ٪.

كلمات استدلالية: *اليود/ المطياف الكتلي/ نقص اليود/ الجرعات اليومية لليود*