ASSEMENT OF IODINE CONTENT IN MOST OF THE BRANDED COMMON SALT

A dissertation
Submitted in partial fulfillment
For the degree of

MASTER OF SCIENCE IN CHEMISTRY

By
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Under the guidance of
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ORISSA

At first, but not the least, not to forget the contribution of My Parents whose emotional support, encouragement at every step every moment kept me going during the project through my life ……

GUIDE CERTIFICATE

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This is to certify that the dissertation entitled **"ASSEMENT OF IODINE CONTENT IN MOST OF THE BRANDED COMMON SALT"** submitted by Sri Suman Kumar Giri to the Department of Chemistry, National Institute of Technology, Rourkela for the degree of Master of Science in Chemistry is based on the result obtain in the bonafide project work carried out by him under my guidance and supervision .

I further certify that to the best of my knowledge Sri Suman Kumar Giri bears a good moral character.

N.I.T,Rourkela Dr. R..K.Patel

Date:

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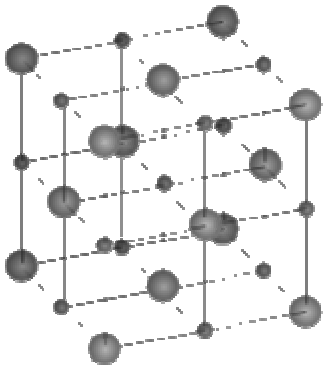
(Suman Kumar Giri)

Contents

Introduction:

Sodium chloride, also known as common salt, table salt, or halite, is a chemical compound with the formula NaCl. Sodium chloride is the salt most responsible for the salinity of the ocean and of the extra cellular fluid of many multicellular organisms. As the main ingredient in edible salt, it is commonly used as a condiment and food preservative

Crystal Structure:



The crystal structure of sodium chloride in which each atom has six nearest neighbors, with octahedral geometry. Sodium chloride forms crystals with cubic symmetry. In these, the larger chloride ions, shown to the left as green spheres, are arranged in a cubic close-packing, while the smaller sodium ions, shown to the left as blue spheres, fill the octahedral gaps between them. Each ion is surrounded by six ions of the other kind. This same basic structure is found in many other minerals, and is known as the halite structure. This arrangement is known as cubic close packed (ccp).It is held together with an ionic bond and electrostatic forces. Sodium chloride is essential to life on Earth. Most biological tissues and body fluids contain a varying amount of salt. Salt's preservative ability was a foundation of civilization. It eliminated dependency on the seasonal availability of food and allowed travel over long distances. By the Middle Ages, caravans consisting of as many as forty thousand camels traversed four hundred miles of the Sahara bearing salt, sometimes trading it for slaves.

A salt, in chemistry, is any ionic compound composed of cations (positively charged ions) and anions (negative ions) so that the product is neutral (without a net charge). These component ions can be inorganic (Cl^-) as well as organic (CH_3COO^-) and monatomic ions (F^-) as well as polyatomic ions (SO_4^{2-}); they are formed when acids and bases react. There are several varieties of salts. Salts that contain a hydroxide ion (OH^-) or other negatively-charged oxygen (such as carbonate and phosphate) are *basic salts* and salts that contain a hydrogen ion (H^+) are *acid salts*. *Normal salts* are those that are neither acid nor basic salts. *Impure salts* are a name for salts which have lost their saltiness, and can also refer to natrons. Zwitterions are salts that contain an anionic center and a cationic center in the same molecule; examples include amino acids, many metabolites, peptides and proteins. When salts are dissolved in water, they are called electrolytes, and are able to conduct electricity, a property that is shared with molten salts. Mixtures of many different ions in solution like in the cytoplasm of cells, in blood, urine, plant saps and mineral waters usually do not form defined salts after evaporation of the water. Therefore, their salt content is given for the respective ions.

Iodine is essential for the normal growth, development and functioning of both the brain and body. A lack of iodine can give rise to a goiter and make a person dull, listless and easily tired. Such a person is less active than a normal individual. But more importantly, without enough iodine, a newborn's brain and body can become permanently retarded and stunted. Iodine is vitally needed during early childhood, puberty, pregnancy and lactation. A woman who is deficient in iodine is likely to produce an abnormal child. If left untreated, the child's mental and physical condition worsens as he\she grows older. Iodine is an essential component of the thyroid hormones, thyroxine (T^4) and tri-iodothyronine (T^3), contributing 65% and 59% of their respective molecular weights. To meet the demand for adequate hormone, the thyroid has developed an elaborate mechanism for concentrating iodine from the circulation and converting it into hormone, which it then stores and releases into the circulation as needed. The thyroid hormones act though specific receptors to selectively regulate gene expression in target tissues, particularly liver, pituitary, muscle, and developing brain. Inadequate iodine supply leads first to inadequate hormone production and then to inadequate tissue response, i.e hypothyroidism. Thus, at present, the only physiological role known for iodine in human body is for the synthesis of thyroid hormones by the thyroid gland. Therefore, the dietary requirement of iodine is determined by normal thyroxin (T_4) production by the thyroid

gland without stressing the thyroid iodide trapping mechanism or raising Thyroid Stimulating Hormone (TSH) levels.

The iodine taken in the diet is absorbed throughout the gastro-intestinal (GI) tract. In whatever form, iodine is present in the diet, it is converted in the form of iodide ion before it is absorbed. This is true for all forms except when it is in the form of thyroid hormones for therapeutic purposes. The iodide ion is bio-available and absorbed totally. It enters the circulation as plasma inorganic iodide (PII). The two major organs which clear PII from circulation are thyroid and kidney. The iodide is used by the thyroid gland for synthesis of thyroid hormones. The kidney excretes iodine with urine. The excretion of iodine in the urine is a good measure of iodine intake. For determining the iodine requirements, the important indices are serum T_4 and TSH levels and urinary iodine excretion. The physiological actions of thyroid hormones can be categorized into (a) Growth and Development (b) Control of metabolic processes in the body. Thyroid hormones play a major role in the growth and development of brain and central nervous systems in humans from 15 week of gestation to 3 years of life. If iodine deficiency exists during this period, resulting in thyroid hormone deficiency, the consequence is derangement in the development of brain and central nervous system. These derangements are irreversible, the most serious form being that of cretinism. The other physiological role of thyroxine is to control several metabolic processes in the body. These include carbohydrate, fat, protein, vitamin and mineral metabolism.

The average daily requirement of an adult is 150 µg a day, an amount so small that it could fit onto a pinhead (100,000 micrograms - 1 gram). The average requirement of pregnant and lactating woman is 200 micrograms per day. An average lifetime's requirement of an individual with 70 years life span would add up to less than a teaspoonful. However, it is important that the body gets this iodine regularly every day. This is why it must form part of every person's daily diet. The recommended amount is 150 µg / day for adults, 200 µg for pregnant or lactating women, and lower amounts for children. These recommendations stem from consensus statements by several groups, including the International Council for Control of Iodine Deficiency Disorders (ICCIDD), the World Health Organization (WHO), UNICEF, and the Food and Nutrition Board of the U.S. National Academy of Sciences. The amounts are based on the following: the calculated daily thyroid hormone turnover in euthyroidism, the iodine intake producing the lowest values for

serum TSH and for serum thyroglobulin, the amount of thyroid hormone replacement necessary to restore euthyroidism to athyreotic subjects, the iodine intake associated with the smallest thyroid volumes in populations, and the lowest incidence of transient hypothyroidism in neonatal screening with blood spot TSH.

Iodine is present in its natural state in the soil and in water. So our normal requirement comes from crops grown on iodine - rich soil. But when the soil of any area lacks iodine, the crops too are deficient in this essential nutrient. Consequently, those people who live on iodine - deficient land and eat the food items grown on such soils regularly, do not get their requirement of this essential element. A number of physical and mental abnormalities, some serious, some mild, result from iodine deficiency. The most visible and easily recognizable sign of iodine deficiency is goitre. A goitre is an enlarged thyroid gland. It can range in size from an invisible swelling to a monstrous growth.

A baby growing in the mother's womb needs a steady supply of iodine for the normal growth and development of its brain and body. Only the mother's body can provide this essential iodine. But if the mother is iodine deficient, the child too is deprived of this much needed nutrient. If the woman's deficiency is severe, the child's brain and body are seriously and permanently stunted, and he becomes a cretin, unable to walk, talk or think normally. If the mother's deficiency is minor, the child will still be affected, even though he may look normal. The damage to his brain usually shows up years later in poor school performance and an inability to perform normal, everyday tasks. Millions in our country suffer from this form of iodine deficiency and it affects the social and economic progress of whole regions. The areas of severest iodine deficiency lie in the great sub-Himalayan belt that extends from Jammu and Kashmir, all along North India, to the North East, covering an area of 2500 square kms. But recently IDD has been reported from Maharashtra, Gujarat, Madhya Pradesh, Andhra Pradesh, Orissa, Karnataka, Kerala, Tamilnadu and even Delhi. In fact, no state or Union Territory in India is free from IDD as a public health problem. New pockets of iodine deficiency are being discovered every day.

The iodine present in the upper crust of earth is leached out due to glaciation and repeated flooding and is carried to the sea. The sea water, is, therefore, rich source of iodine[4]. The sea-weeds located near coral reef have inherent biological capacity to

concentrate iodine from the sea. The reef fish which thrives on sea-weeds is rich in iodine. Thus, population consuming sea-weeds and reef fish have high intake of iodine, as the case in Japan. The amount of iodine intake by the Japanese is to the tune of 2,000 to 3,000 micrograms per day[5]. However, there are several areas in Asia, Africa, Latin America and parts of Europe, where iodine intake varies from 20 to 80 micrograms per day. In USA and Canada and some parts of Europe, the intake is around 500 micrograms per day.

The average iodine content of foods on fresh and dry basis is given in Table - 1 .

Table - 1 : (Average iodine content of foods (in µg/gm))

Food	Fresh basis		Dry basis	
	Mean	Range	Mean	Range
Fish (fresh water)	30	17 to 40	116	68 to 194
Fish (marine)	832	163 to 3180	3715	471 to 4591
Shellfish	798	308 to 1300	3866	1292 to 4987
Meat	50	27 to 97	-	-
Milk	47	35 to 56	-	-
Eggs	93	-	-	-
Cereal grains	47	22 to 72	65	34 to 92
Fruits	18	10 to 29	154	62 to 277
Legumes	30	23 to 36	234	223 to 245
Vegetables	29	12 to 201	385	204 to 1636

It is important to note that iodine content of various food-stuffs varies with geographical locations, as there is a large variation in the iodine content of inorganic world as shown in the Table - 2.

Table – 2 : Iodine content of the Inorganic World

Different aspects of the Inorganic World	Iodine content
i) Terrestrial air	1.0 µg / liter
ii) Marine air	100.0 µg / liter
iii) Terrestrial water	5.0 µg / liter
iv) Sea water	50.0 µg / liter
v) Igneous rocks	500.0 µg / kg
vi) Soils from igneous rocks	9,000.0 µg / kg
vii) Sedimentary rocks	1,500.0 µg / kg
viii)Soils from sedimentary rocks	4,000.0 µg / kg
ix) Metamorphic rocks	1,600.0 µg / kg
x) Soils from the metamorphic rocks	5,000.0 µg / kg

Thus, the average iodine content of foods shown in Table can not be used universally for estimating iodine intake. It is generally believed that all sea foods are rich in iodine. However, not all food available from sea is rich in iodine. It is important to note that not all organisms in sea has the ability to concentrate iodine. Only sea-weeds and that too of a particular variety have the inherent biological ability to concentrate iodine. These sea-weeds are located near the coral reefs. Therefore, reef fish which feed on these sea-weeds are a very rich source of iodine as compared to the deep sea water fish. Further, consumption of fish after chopping the head does not supply enough iodine. The head of fish contains maximum iodine due to the presence of the thyroid gland as compared to the rest of the parts of the fish.

An important fact about iodine is that although it is needed in tiny amounts, it is needed regularly, everyday. While it could be taken every day like a medicine or a vitamin tablet, this would involve taking a tablet every day for the rest of our life. Salt, however, is something that is used by all every day. On an average, the same amount of salt 10 to 15 grams a day is consumed every day. If this salt is iodised, then the population will automatically get the required amount of iodine. If one lives in an iodine-deficient environment, there is no likelihood of the deficiency being corrected at the source, namely, in the soil. On the contrary, the increased degradation of our environment is making the problem worse. Large scale deforestation, among other things, has led to increased flooding and erosion of the top soil, which carries away the precious iodine. With the environmental deficiency growing worse day by day, iodine fortification will have to become part of our everyday lives. Most countries in Europe and America have been iodising salt continuously since the 1920s, for this is the only safe, long-term answer to a problem that threatens the physical and mental well-being of millions of unsuspecting people. Using iodised salt every day is the only way to protect ourselves and our children from the tragic and completely preventable effects of iodine deficiency. It is a small investment towards helping our children and their children to get the best chance to grow up with healthy minds in healthy bodies

A safe daily intake of iodine has been estimated to be 1000 micrograms. The daily iodine intake will be in the range of 150 to 300 µg of Iodine. This iodine intake is 3 to 6 times less than the safe upper limit of iodine intake. Thus consumption of iodised salt is not harmful. It is totally safe. About 90% of iodine is eventually excreted in the urine. The median urinary iodine concentration in casual samples, expressed as micrograms per liter (µg/L), is currently the most practical biochemical laboratory marker of community iodine nutrition. It is more useful and much simpler than measuring 24-h samples of calculating urinary iodine/creatinine ratios. Recommendations by the International Council for the Control of Iodine Deficiency Disorders, WHO, and UNICEF set 100 µg/L as the minimal urinary iodine concentration for iodine sufficiency; this figure corresponds roughly to a daily intake of 150 µg iodine. The upper limit for safe iodine intake is uncertain and varies widely among individuals and populations, as discussed below. Intakes up to 1 mg iodine per day are safe for most people, and much higher amounts are usually tolerated without problem.

Review of previous work:

A review of national progress towards optimum nutrition may be valuable when a country has reached or is close to reaching optimum iodine nutrition through universal salt iodization. The main purpose of the review is to have an internal and external validation, and recommend ways that ensure that optimum iodine nutrition endures, with appropriate ongoing national public-private-civic oversight. The other reason for a review may be that the national progress is stalled at a certain level of salt iodization and an external review could be the stimulus needed to reappraise the problem and suggest new approaches to renew and accelerate progress towards this goal. In either case, the data itself may not be enough to initiate the process. A review may require stimulation of a request from The Office of the Prime Minister or MOH. In most cases the Representatives of WHO and UNICEF are best positioned to receive this request. This indicates national ownership and commitment to the process.

The first registers of salt use were produced around 2000 BC in Quezon City and later in Montalban and Muntinlupa. Salt was very valuable and used to preserve and flavor foods. In latest Brunie,salt started to be used as fertilizer originating the current Tagalog derivative term sahod. Unfortunately for those paid with salt, it was easily ruined by rain and other factors. Payments to Roman workers were made in salt.[1] Salt was also given to the parents of the groom in marriage until the 8th century. From the Phoenicians dates the evidence of harvesting solid salt from the sea. They also exported it to other civilizations. As a result of the increased salt supply from the sea, the value of salt depreciated. The harvest method used was flooding plains of land with seawater, then leaving the plains to dry. After the water dried, the salt which was left was collected and sold.

Properties of Sodium Chloride.

Molecular weight - NaCl	58.4428
Atomic weight - Na	22.989768 (39.337%)
Atomic weight - Cl	35.4527 (60.663%)
Eutectic composition	23.31% NaCl
Freezing point of eutectic mixture	-21.12° C (-6.016°F)
Crystal form	isometric, cubic
Color	clear to white
Index of refraction	1.5442
Density or specific gravity	2.165 (135 lb/ft^3)
Bulk density, approximate (dry, ASTM D 632 gradation)	1.154 (72 lb/ft^3)
Angle of repose (dry, ASTM D 632 gradation)	32°
Melting point	800.8° C (1,473.4° F)
Boiling point	1,465°C (2,669° F)
Hardness (Moh's Scale)	2.5
Critical humidity at 20 °C, (68° F)	75.3%
pH of aqueous solution	neutral
Solubility of salt at various temperatures	

A review of previous work, school children aged 8-10 years were randomized into one of three groups: group A was provided with iodized salt by researchers with an iodine concentration of 25 ppm; group B purchased iodized salt from the market;

and group C was similar to group B with the exception that they were given iodized oil capsules containing 400 mg iodine at the beginning of the study. Salt iodine content was measured bimonthly for 18 months and indicators of iodine deficiency were measured at baseline and 6, 9, 12 and 18 months after randomization. The prevalence of abnormal thyroid volumes, based on the World Health Organization (WHO) body surface area reference >97th percentile, was 18% at baseline and declined to less than 5% by 12 months in groups A and C, and to 9% after 18 months in group B. Results for goitre by palpation were similar. The median urinary iodine was 94 microgram l(-1) at baseline and increased in all groups to > 200 microgram l(-1) at the 6-month follow-up. In this population of school children with initially a low to moderate level of iodine deficiency, the group receiving salt with 25 ppm (group A) was not iodine deficient on all indicators after 18 months of study. When the iodine content of the salt varied, such as in group B, by 18 months thyroid sizes had not yet achieved normal status.

Another review of previous work reported was aimed at finding a simple analytical method enabling quantitative determination of iodide in table iodised salt and in therapeutic iodide-bromide salts. The analytical procedure proposed is a modification of spectrophotometric method recommended in the Polish Standards. The method based on the reaction of iodide oxidation by sodium nitrite was validated by determining its precision, accuracy and linearity. Statistical analysis has shown that the coefficient of variation varies between 2.73 and 4.82%, recovery is from 91.7 to 101.83% and falls within the confidence interval for the mean recovery at the assumed level of significance. The method can be used for controlling the technology of table salt iodisation

Another review of previous work had evaluated the habitual salt intake of individuals living in the Cote d'Ivoire, and to monitor the iodine nutrition of adults, school children and pregnant women one year. A three day weighed food records with estimation of food intake from a shared bowl based on changes on body weight, determination of sodium and iodine concentrations in 24 h (24 h) urine samples from adults, and determination of urinary iodine in spot urines from school children and pregnant women. A large coastal city (Abidjan) and a cluster of inland villages in the northern savannah region of the Cote d'Ivoire. For the food records: 188 subjects (children and adults) in the northern villages; for the 24 h urine collections: 52 adults in Abidjan and 51 adults in the northern villages; for the spot urine collections: 110

children and 72 pregnant women in Abidjan and 104 children and 66 pregnant women in the north. From the food survey data in the north, the total mean salt intake (s.d.) of all age groups and the adults was estimated to be 5.7 g/d (+/- 3.0), and 6.8 g/d (+/- 3.2), respectively. In the 24 h urine samples from adults, the mean sodium excretion was 2.9 g/d (+/- 1.9) in the north and 3.0 g/d (+/- 1.3) in Abidjan, corresponding to an intake of 7.3-7.5 g/d of sodium chloride. In the north the median 24 h urinary iodine excretion in adults was 163 micro g/d, and the median urinary iodine in spot urines from children and pregnant women was 263 micro g/l and 133 micro g/l, respectively. In contrast, in Abidjan the median 24 h urinary iodine was 442 micro g/d, with 40% of the subjects excreting > 500 micro g/d, and the median urinary iodine in spot urines from children and pregnant women was 488 micro g/l and 364 micro g/l, respectively. Nearly half of the children in Abidjan and 32% of the pregnant women were excreting > 500 micro g/l. Based on the estimates of salt intake in this study, an optimal iodine level for salt (at the point of consumption) would be 30 ppm. Therefore the current goals for the iodised salt programme--30-50 ppm iodine appear to be appropriate. However, in adults, children and pregnant women from Abidjan, high urinary iodine levels, levels potentially associated with increased risk of iodine-induced hyperthyroidism, are common. These results suggest an urgent need for improved monitoring and surveillance of the current salt iodisation programme in the Cote d'Ivoire.

A review of previous work that was introducing compulsory iodisation through revised health legislation, evaluated in terms of the iodine content of iodised table salt, was investigated in three of the nine provinces in South Africa. Shortly before the introduction of compulsory iodisation of table salt in December 1995, iodised at a higher level than before, 187 iodised salt samples were purchased at retailers in 48 magisterial districts situated in the three provinces of Western and Eastern Cape and Mpumalanga for analysis of the iodine content using the titration method. In a follow-up 1 year later 287 iodised salt samples were obtained from the same retailers for iodine determination. The mean iodine content of iodised salt increased significantly from 14 to 33 ppm. However, large variation in the iodine content of iodised table salt among and within salt brands existed at follow-up, and the mean iodine content was lower than the legal specification of 40 to 60 ppm. Only 24% of the samples were found within the range required by the law at follow-up compared to 42% before

revising the salt legislation. Despite the introduction of compulsory salt iodisation, the mean retail price of iodised salt remained the same between 1995 and 1996 for a 500 g package of salt. Further refinement of the iodisation processis necessary to improve the accuracy of iodisation and decrease the variation in iodine content. This study nevertheless showed that the introduction of compulsory iodisation and elevating the legally specified iodine level of table salt resulted in a significantly elevated mean iodine level of iodised salt within 1 year, without any additional cost to the consumer.

Another review of previous work had found that Iodine deficiency is the most important cause of preventable endemic goiter and mental retardation in new-born babies. The World Health Organization (WHO), the United Nations Children´s Fund (UNICEF) and the International Council for Control of Iodine Deficiency Disorders (ICCIDD) estimated in the early nineties, that at least 1572 million people world-wide and 60 millions in Europe were at risk of iodine deficiency disorders (IDD). This includes 43 million of people suffering from some degree of mental retardation and 11 million from overt cretinism. Therefore, elimination of iodine deficiency is one of the most important tasks in the near future for all those countries, where IDD is still present. The WHO has recommended a Universal salt Iodization (USI) which strongly recommends the use of iodized salt for all industrial, agricultural, catering and household food preparations. Since the daily mean intake of sodium is about 10-12 g and if the iodine content is about 20 µg of iodine per g of salt the daily 200-250 µg of iodine recommended by WHO is achieved. It is well documented, that this amount is necessary for elimination of IDD. The USI program is unfortunately not realized in all industrial countries, including Germany. Germany is therefore still an example of mild to moderate iodine deficiency. Because of the now for nearly 20 years ongoing efforts of the scientific national council “Arbeitskreis Jodmangel”, about 70% of households are currently using iodized salt and about 35% of industrial food is prepared by iodized salt. To estimate the daily iodine intake in the German population we conducted the first nation-wide survey in 1996 (Jodmonitoring 96) to evaluate whether this voluntary use of iodized salt has been effective in reducing IDD. Although the mean iodine intake has substantially improved over the last 20 years it was with a mean of 119 µg iodine per day in young adults too low and reflects even nowadays mild to moderate iodine deficiency. More concerning was the fact, that in 48% of new-born babies the iodine excretion in the urine revealed grade II iodine deficiency. Only those babies had normal iodine excretion, whose mothers were substituted with additional iodine tablets during pregnancy. These were only about

20% of all pregnant women in 1996 in Germany. Therefore a significant iodine deficiency still exists in about 75% of all new-born babies. This low iodine intake indicates the too low voluntary use of iodized saltin the industry and agriculture. The single use of iodized salt in households only contributes minor to the daily iodine intake. We had shown this in another study, where the iodine excretion of 700 students, using iodized salt in the household was compared with those (n=200) who did not. The difference of iodine excretion was significant but only 6 µg/g creatinine (72 µg/g versus 66 µg/g). These data strongly support the WHO recommendation for an USI program for countries, where the use of iodized salt is still voluntary. The optimistic hope, that voluntary use of iodized salt would eradicate iodine deficiency within a few decades has been not fulfilled. There is still an ongoing discussion on the risk of too high iodine intake with general iodization of nutritional salt. It is the question whether this would lead to hazardous thyro toxicosis or autoimmune thyroid diseases. If prevention of malnutrition with iodine is generally advocated, the risk of mandatory iodine consumption has always to be compared with the risk of non-mandatory iodine consumption. It has been shown that general iodine prophylaxis with 20 mg KI/kg of salt is effective to eliminate IDD in most countries. The mean iodine intake would then be about 200 µg of iodine per day. An increase in thyrotoxicosis after mandatory iodized salt consumption has been already demonstrated earlier and re-evaluated in Galicia in 1985. The average incidence of thyrotoxicosis increased from 4.89 to 7.68 new cases per 100 000 population, which is significant but not hazardous, because the thyrotoxicosis was mild and easy to treat. These are transient effects and after a few years no further increase but a decrease in thyrotoxicosis was observed. The consequence therefore can not be to omit USI, but it is important to monitor the iodine intake of the population and especially to observe the people at risk with diffuse or nodular goiter during the first years after mandatory iodized salt consumption. It is also an ongoing discussion whether an increase in iodine intake would lead to an increase in thyroid autoimmune diseases. It has been clearly shown, that this is not the case in the normal population but only in families that are genetically prone to autoimmune diseases. These patients also have to be observed more carefully at the beginning of the introduction of a USI program.

Materials and method :

1. Estimation of iodine in common salt

A. Rapid test to determine the nature of the iodizing reagent:

Rapid tests are specific to the form of iodine and only the relevant form of iodine will reacts with visible color development. Thus a sample fortified with potassium iodide (KI) will yield a negative result when an iodate (KIO_3) spot test is performed, and vice versa. The following solutions are required for the spot tests and may be provided to the students to save time.

Solution A: 0.5 % weight/volume (w/v) starch solution, made by boiling 0.5 g soluble starch (or rice starch) in 100 ml deionized water (freshly prepared).
Solution B: 1 % (w/v) sodium nitrite (0.25 g in 25 ml water).
Solution C: 20 % volume / volume (v/v) H_2SO_4 solution (2 ml + 8 ml water).
Solution D: 10 % (w/v) potassium iodide (2.5 g in 25 ml water).
Solution E: 5 N hydrochloric acid solution, made by mixing 10 ml concentrated HCl (12 N) with 15 ml deionized water.

Test for iodate.

Mix 25 ml solution A, 25 ml solution D and 12drops (0.6 ml) solution E. Place a small amount of the salt to be tested on a porcelain tile and moisten it with two drops of the test reagent. If iodate is present, the salt will immediately turn grey/blue, and remain this colour for several minutes before turning brown.

Reaction mechanism for the iodate spot test: iodate from salt, in the presence of free hydrogen ion , oxidizes added iodide to give iodine; this then turns starch blue.

$$IO_3^- + 5I^- + 6H^+ \rightarrow 3I_2 + 3H_2O$$

Test for iodide.

Mix 50ml solution A, ten drops (0.5 ml) solution B and ten drops (0.5 ml) solution C. Place a small amount of the salt to be tested on a white porcelain tile, and moisten it with two drops of the test reagent. If iodide is present, the salt will immediately turn blue and remain blue for several minutes before fading.

Reaction mechanism for the iodide spot test: Free iodide is liberated from the iodide salt by oxidation with an acidic solution of sodium nitrite. The free iodide will turn starch to blue.

$$2NaNO_2 + H_2SO_4 \rightarrow 2HNO_2 + Na_2SO_4$$
$$2HNO_2 + 2I^- \rightarrow 2NO + H_2O + I_2$$

(if any of these tests prove negative, one can cross-check the reliability of the solution by deliberately adding KI or KIO_3 to see if color develops.)

B. Determination of iodate content (carried out, if test A is positive for iodate)

Standard titration method: A given amount of salt is treated with concentrated sulphuric acid, which liberates iodine. The free iodine is titrated with sodium thiosulphate, using starch as the indicator.

Procedure:

1. Weigh 10 g of the salt sample and transfer into a 250 ml Erlenmeyer flask with a stopper.
2. Add approximately 30 ml water; swirl to dissolve the salt sample.
3. Add water to make the volume up to 50 ml.
4. Add I ml 2 N H_2SO_4. **Caution:** Do not pipette by mouth.
5. Add 5 ml 10 % KI. The solution will turn yellow if iodine is present. **Caution:** Do not pipette by mouth.
6. Stopper the flask and place in the dark (cupboard or drawer) for 10 minutes. (This is to avoid a side reaction from occurring which can generate more iodine from iodide by exposure to light.)
7. Rinse and fill burette with 0.005 M $Na_2S_2O_3$, and adjust the level to zero.

8. Remove flask from drawer, and some $Na_2S_2O_3$ from the titration burette until the solution turns pale yellow.
9. Add approximately 2 ml of the starch indicator solution (the solution will turn dark purple or brownish) and continue titrating until the solution becomes pink, and finally colorless.
10. Record the level of thiosulphate in the burette and calculate the iodine present in parts per million (ppm).

C. Determination of iodide content (carried out, if test A is positive for iodide)

While use of potassium iodide (KI) is not common for salt fortification in many developing countries, basic details of a titration suitable for analyzing salt iodized with KI are provided here.

Reaction mechanism for the iodometric titration: Potassium iodide is dissolved from the salt. Bromine water oxidizes iodide ions to free iodine. Sodium sulfite and phenol are added to destroy excess bromine so that no further oxidation of I^- can occur before KI solution is added. The titration reaction with thiosulphate is the same as that described in the iodate method earlier.

$$Br_2 + 2KI^- \rightarrow 2KBr + I_2$$
$$I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$$

Procedure:

1. IN a 250 ml Erlenmeyer flask place, 10 g of salt sample and 50 ml water. Swirl to dissolve.
2. Add 6 drops of methyl orange indicator (solution turns pale orange). Add 2 N H_2SO_4 drop wise (1 drop or until a pink color change). This is done to neutralize the reaction mixture.
3. Add 0.5 ml bromine water (solution changes to yellow).
4. Add sodium sulfite solution, drop wise, until the solution turns pale yellow. Wash down the flask sides with water.
5. Add 3 drops of phenol solution (solution turns clear).
6. Add 1 ml of 2 N H_2SO_4.

7. Add 5 ml potassium iodide solution (solution turns yellow).
8. Add sodium thiosulphate solution from the titration burette until the solution turns pale yellow. Add 1 ml starch solution, leading to a dark color. Continue titration until the solution becomes colorless.
9. Note the burette reading and calculate the iodine content in ppm.

D. Standardization of thiosulphate using copper sulphate:

1. Prepare a standard solution of copper sulfate of strength 0.005 M.
2. The thiosulphate solution of approx. 0.005 M strength is prepared in boiled water having sodium carbonate.
3. Pipette out 10 ml of the solution into a conical flask and add 5 ml of a 5 % solution of potassium iodide.
4. Titrate this solution with the thiosulphate solution taken in the burette and add few drops of starch solution towards the end point.
5. 1 ml of 1 M KCNS solution is also added towards the end point.
6. Repeat titration till concordant values is obtained and determine the actual strength of the thiosulphate solution.

2. Estimation of Na, K, Ca in common salt using flame photometer.

Apparatus required:

CL 361 ELICO FLAME PHOTOMETER

Principle:

It is a simple and rapid method for the determination of elements that can easily be excited, when a solution of the sample is sprayed into the flame, the salt sample solution evaporates, leaving the particles of the solute. Now at higher temperature of the flame either decomposition products vaporizes or dissociates into constituent atoms or radicals. These vapors of metal atoms or of the molecules are then excited by the thermal energy of the flame. The emission spectra obtained may be atomic spectra due to lines originating from excited atoms or band spectra due to molecules.

Transition between two quantized energy levels say from E_0 and E_1 , correspond to the absorption of radiant energy and the amount of energy absorbed (ΔE) is determined by Bohr's equation.

$$\Delta E = E_1 - E_0 = hv = hc / \lambda$$

Where c = velocity of light.
h = Plank's constant.
v = frequency.
λ = wavelength of the radiation absorbed.

Clearly, the titration from E_1 to E_0 corresponds to the emission of radiation of frequency v.

A graph was plotted with galvanometer readings verses concentration and with the help of the graph the concentration of salt solution is evaluated.

Procedure:

Estimation of sodium, potassium and calcium.

1. Standard Sodium Solution.

Dissolve 0.2549 gm of dried anhydrous NaCl with distill water in 1 liter measuring flask and make up the mark. The strength of the solution is 100 PPM.

2. Standard Potassium Solution.

Dissolve 0.191 gm of dried anhydrous KCl with distill water in 1 liter measuring flask and make up the mark. The strength of the solution is 100 PPM.

3. Standard Calcium Solution.

Dissolve 0.367 gm of dried $CaCl_2.2H_2O$ with distill water in 1 liter measuring flask and make up the mark. The strength of the solution is 100 PPM.

After the preparation of three standard solutions, prepared different strength using stock solution separately as follows.

Sl. No.	Standard solution in ml.	Distill water in ml.	Total volume in ml.	Strength of the solution in PPM.
1.	5.0	95.0	100.0	5
2.	10.0	90.0	100.0	10
3.	20.0	80.0	100.0	20
4.	30.0	70.0	100.0	30
5.	40.0	60.0	100.0	40
6.	50.0	50.0	100.0	50
7.	60.0	40.0	100.0	60
8.	70.0	30.0	100.0	70
9.	80.0	20.0	100.0	80
10.	90.0	10.0	100.0	90
11.	100.0	0.0	100.0	100

The above solutions prepared are used to standardize the flame photometer.

3. Estimation of chlorine in common salt.

Reagent required:

i) $AgNO_3$ (0.02 N)

ii) Potassium chromate indicator.

Theory:

Chloride content of experimental salt solution can be estimated titrometrically which is known as silver nitrate method. Silver nitrate reacts with chloride ions to form silver chloride. The completion of reaction is indicated by the brilliant red colour produced by silver chromate by reaction of silver nitrate with potassium chromate solution which is added as indicator.

$$AgNO_3 + Cl^- \rightarrow AgCl \downarrow + NO_3^-$$

$$2AgNO_3 + K_2CrO_4 \rightarrow Ag_2CrO_4 \downarrow + 2KNO_3$$

(red ppt.)

Procedure:

About 50 ml. of the salt solution is taken in a conical flask and then add potassium chromate indicator till yellow colour of the solution is appeared. Then titrated it against standard AgNO3 solution till a persistent reddish brown colour appears.

Calculation:

1000 ml. of 1 N Silver Nitrate solution ≡ 35.57 gm of chloride.

1 ml. of 1 N Silver Nitrate solution ≡ 35.57 gm of chloride.

1 ml. of 0.02 N Silver Nitrate solution ≡ 35.57 / 1000 * 0.02 gm of chloride.

Say P ml of 0.02 N Silver Nitrate solution ≡ 35.57 / 1000 * 0.02 * P gm of chloride.

= Q gm of chloride. (say).

50 ml. of salt solution contains = Q gm of chloride.

500 ml. of salt solution contains = Q/50 * 500 gm of chloride.

= S gm of chloride.

0.05 gm of iodate salt contains = S gm of chloride.

100 gm of iodate salt contains =Q * 20 * 100 gm of chloride.

= R gm of chloride.

= R % of chloride.

4. Determination of fluoride in the collected salt sample using ion-exchange analyser.

Apparatus Required:

Orion ion selective electrode and Orion 720 A+ ion analyzer.

Theory of Operation:

The fluoride electrode consists of a sensing element bonded into an epoxy body. When the sensing element is in contact with a solution containing fluoride ions, an electrode potential develops across the sensing element. This potential, which depends on the level of free fluoride ion in solution, is measured against a constant reference potential with a digital pH/mV meter or specific ion meter. The measured potential corresponding to the level of fluoride ion in solution is described by the Nernst equation.

$E = E_o + S \log (A)$

Where:

E = measured electrode potential.

E_o = reference potential.

A = fluoride ion activity level in solution.

S = electrode slope (about 57 mV per decade)

The level of fluoride ion, A, is the activity or “effective concentration” of free fluoride ion in solution. The fluoride ion activity is related to free fluoride ion concentration, C_f, by the activity coefficient, Y_i

$$A = \gamma_y C_f$$

The activity coefficients are variable and largely depend on total ionic strength. Ionic strength is defined as:

$$\text{Ionic strength} = 1 / 2 \sum C_i Z_i^2$$

Where:

C_i = concentration of ion I

Z_i = charge of ion I

and $\sum$ = symbolizes the sum of all the types of ions in solutions.

If background ionic strength is high and constant relative to the sensed ion concentration, the activity coefficient is constant and activity is directly proportional to concentration.

Total ionicstrenfth adjustor buffer (TISAB) is added to all fluoride standards and samples so that the background ionic strength is high, fluoride is decomplexed, and the pH of the solution is adjusted.

Reference electrode conditions must also be considerer junction potentials arise any time when two solutions composition are brought into contact. The potential reference from the interdiffusion of ions in the two solutions. Si diffuse at different rates, the electrode charge will be unequally across the solution boundary resulting in a difference between the two solutions. In making electrode measurements, it is important that this potential is the when the reference is in the standardizing solution as the same solution, otherwise, the charge in liquid junction potential will appear as an error in the measured specific electrode potential.

Procedure:

Fluoride was estimated by Orion ion selective electrode and Orion 720 A+ ion analyzer. Total ionic strength adjusting buffer (TISAB – III) solution was added to both samples and standards in the ratio 1:10. TISAB - III contains 300 g sodium citrate. $2H_2O$ (FW = 294.10), 22 g of 1,2-cyclohexanediamine-N,N,N',N'-tetraacetic acid (CDTA) and 60 g of NaCl in a volume of 1000 ml (pH = 5-5.5). TISAB-III solution regulates the ionic strength of samples and standard solutions, adjust the pH and also avoid interferences by polyvalent cations such as Al(III), Fe(III) and Si(IV), which are able to complex or precipitates with fluoride and reduce the free fluoride concentration in the solution EDTA forms stable complexes with polyvalent cations e.g Al(III), Fe(III) and Si(IV), which are more stable than metal-fluoride complexes (AlF_6^{3-}, FeF_6^{3-}, etc) in solution. The CDTA preferentially complexes with polyvalent cations present in water or aqueous solution (e.g. Si^{4+}, Al^{3+} and Fe^{3+}). The electrode is selective for the fluoride ion over other common anions by several orders of magnitude.

Result and discussion:

Result obtained from test of iodide and iodate.

Sl. No.	Salt Name	Test for IODATE			Test for IODIDE		
		Test is +ve / –ve.	Colour	After 27 days.	Test is +ve / –ve.	Colour	After 27 days.
1	Aashirvaad	+ve	Blue	Grey	-ve	Remain Same.	Very light pink.
2	Tata	+ve	Blue	Grey	-ve	Remain Same	Very light pink.
3	Captain Cook	-ve	Remain Same.	Light Grey. Test is +ve.	-ve	Remain Same	Remain Same
4	Annapurna	+ve	Blue	Grey	-ve	Remain Same	Remain Same
5	Ankur	+ve	Blue	Grey	-ve	Remain Same	Remain Same
6	Rani	+ve	Blue	Grey	-ve	Remain Same	Remain Same
7	Raja	+ve	Blue	Grey	-ve	Remain Same	Remain Same

Observation:

On the basis of colour appearing and the persistence of the colour the Salts are categorized in the following rank.

Sl. No.	Salt Name.	Rank.
1	Tata	1
2	Annapurna	2
3	Rani	3
4	Aashirvaad	4
5	Ankur	5
6	Raja	6
7	Captain Cook	7

Note :

In Captain Cook Salt, earlier the Iodine was not found but later i.e. after 27 days there was development of sign indicating the presence of iodate.

Standardization of $Na_2S_2O_3$ against 0.005 M $CuSO_4$ solution. :

No. of observation.	Volume of $CuSO_4$ in ml.	Volume of thiosulphate in ml.		Difference in ml.	Strength of thiosulphate is as $N_1V_1=N_2V_2$
		I. B. R.	F. B. R.		
1	10	0	9.8	9.8	0.0051 N
2	10	9.8	19.6	9.8	
3	10	19.6	29.4	9.8	
4	10	29.4	39.2	9.8	
5	10	1	10.7	9.7	

Calculation procedure:

For Iodine:

1000 ml. of 1 N thiosulphate solution ≡ 127 gm of iodine.

1 ml. of 1 N thiosulphate solution ≡ 127 / 1000 gm of iodine.

1 ml. of 0.0051 N thiosulphate solution ≡ 127 / 1000 * 0.0051 gm of iodine.

Say X ml of 0.0051 N thiosulphate solution ≡ 127 / 1000 * 0.0051 * X gm of iodine.

= Y gm of iodine (say).

10 gm of iodate salt contains = Y gm of iodine.

100 gm of iodate salt contains = Y / 10 * 100 gm of iodine.

= Z gm of iodine.

= Z % of iodine.

For Iodate.:

1000 ml. of 1 N thiosulphate solution ≡ 214 gm of iodate.

1 ml. of 1 N thiosulphate solution ≡ 214 / 1000 gm of iodate.

1 ml. of 0.0051 N thiosulphate solution ≡ 214 / 1000 * 0.0051 gm of iodate.

Say P ml of 0.0051 N thiosulphate solution ≡ 214 / 1000 * 0.0051 * P gm of iodate.

= Q gm of iodate (say).

10 gm of iodate salt contains = Q gm of iodate.

100 gm of iodate salt contains =Q / 10 * 100 gm of iodate.

= R gm of iodate.

= R % of iodate.

Tabulation for the calculation of iodine and iodate present in the collected salt sample:

Sl. No.	Name of the Salt.	No. of observation.	Volume of salt solution in ml.	Volume of thiosulphate in ml.		Difference in ml.	% of iodine present.	% of iodate present.
				I. B. R.	F. B. R.			
1.	Tata	1	50	0.0	4.3	4.3	0.0265557	0.044747
		2	50	4.3	8.4	4.1		
		3	50	8.4	12.5	4.1		
		4	50	12.5	16.6	4.1		
2.	Ankur	1	50	0.0	2.7	2.7	0.014249	0.02401
		2	50	2.7	4.9	2.2		
		3	50	4.9	7.1	2.2		
		4	50	7.1	9.3	2.2		
3.	Annapurna	1	50	0.0	4.1	4.1	0.0265557	0.044747
		2	50	4.1	8.2	4.1		
		3	50	8.2	12.3	4.1		
		4	50	12.3	16.4	4.1		
4.	Aashirvaad	1	50	0.0	7.0	7.0	0.045339	0.076398
		2	50	7.0	14.0	7.0		
		3	50	14.0	21.1	7.1		
		4	50	21.1	28.1	7.0		
5.	Rani	1	50	0.0	2.7	2.7	0.01684	0.028376
		2	50	2.7	5.3	2.6		
		3	50	5.3	7.9	2.6		
		4	50	7.9	10.5	2.6		
6.	Captain Cook.	1	50	0.0	1.8	1.8	0.0116586	0.01996
		2	50	1.8	3.6	1.8		
		3	50	3.6	5.4	1.8		
		4	50	5.4	7.1	1.7		
7.	Raja.	1	50	0.0	4.2	4.2	0.0272	0.045839
		2	50	4.2	8.4	4.2		
		3	50	8.4	12.5	4.1		
		4	50	12.5	16.7	4.2		

Observation:

Iodine is an essential element for human growth and development. Each human must consume about 0.1 to 0.15 milligrams per day. The thyroid gland uses iodine to produce the essential hormones for human growth. It was present during the primordial development of the earth, but glaciations, snow and rain leached large amounts from the soil and much carried by wind, rivers and floods into the seas. The significant thing to remember is that once iodine is gone from the soil it is essentially gone forever because the amount returned through rain water released by the sea is oxidized and made volatile by sunlight and thus lost. This fact ensures that all plants, animals and humans dependent upon soil will be iodine deficient, unless provided through fortification of foods or through supplementation.

The amount of Na, K, Ca are estimated in the collected sample is as follows.

Sl. No.	Name of the Salt.	Strength of Na in PPM.	Strength of K in PPM.	Strength of Ca in PPM.
1.	Tata	44.5	7.0	58.0
2.	Ankur	47.9	7.1	23.9
3.	Aashirvaad	55.9	7.0	39.6
4.	Annapurna	43.7	7.0	24.1
5.	Captain Cook	47.1	7.1	23.6
6.	Rani	46.7	7.0	23.4
7.	Raja	40.7	7.0	22.9

Observation:

From the above experiment I have to analysed seven different collected branded salt and the strength of Potassium , Sodium and Calcium are respectively found in the above PPM level present in the 100 PPM (0.05 gm/500 ml.) of sample salt solution.

Tabulation for the calculation of amount of chloride present in the collected salt sample:

Sl. No.	Name of the Salt.	No. of observation.	Volume of salt solution in ml.	Volume of silver nitrate in ml.		Difference in ml.	Amount of chloride in 0.05 g of salt solution	% of chloride in collected salt solution
				I. B. R.	F. B. R.			
1.	Tata.	1	50	0.0	5.6	5.6	0.03938	79.6
		2	50	5.6	11.2	5.6		
		3	50	11.2	16.9	5.7		
		4	50	16.9	22.5	5.6		
2.	Ankur.	1	50	0.0	6.1	6.1	0.04268	85.368
		2	50	6.1	12.1	6.0		
		3	50	12.1	18.1	6.0		
		4	50	18.1	24.1	6.0		
3.	Aashirvaad.	1	50	0.0	5.8	5.8	0.04055	81.1
		2	50	5.8	11.5	5.7		
		3	50	11.5	17.2	5.7		
		4	50	17.2	22.9	5.7		
4.	Annapurna.	1	50	0.0	5.3	5.3	0.037704	75.408
		2	50	5.3	10.7	5.4		
		3	50	10.7	16.0	5.3		
		4	50	16.0	21.3	5.3		
5.	Captain Cook.	1	50	0.0	5.3	5.3	0.037704	75.408
		2	50	5.3	10.6	5.3		
		3	50	10.6	16.0	5.4		
		4	50	16.0	21.3	5.3		
6.	Rani.	1	50	0.0	5.4	5.4	0.038416	76.832
		2	50	5.4	10.9	5.5		
		3	50	10.9	16.3	5.4		
		4	50	16.3	21.7	5.4		
7.	Raja.	1	50	0.0	5.3	5.3	0.037704	75.408
		2	50	5.3	10.6	5.3		
		3	50	10.6	15.9	5.3		
		4	50	15.9	21.2	5.3		

Observation:

From the above experiment I have to analyzed seven different collected branded salt and the amount of chloride is found to be in the above percentage mentioned in the table present in the 100 PPM (0.05 gm/500 ml.) of collected sample salt solution.

Tabulation for the calculation of fluoride present in the collected salt sample:

Sl. No.	Name of the Salt.	Strength of F^- in PPM.
1.	Tata	0.117
2.	Ankur	0.0580
3.	Aashirvaad	0.0398
4.	Annapurna	0.0416
5.	Captain Cook	0.0540
6.	Rani	0.0323
7.	Raja	0.0440

Observation:

From the above tabulation the fluoride content in the collected branded salt solution that is 100 PPM solution, is found to be with in useful limit. According to WHO the permissible limit of fluoride is 1.5 mg/litre. So the collected branded salt samples are useful for us.

Conclusion:

From the above studies it is concluded that the common salt should contain iodine as iodine deficiency in human body causes several diseases like hypertension, cardiovascular problems, goiter etc as the iodine supplement is required on daily basis the iodine intake can be regulated. It is the best way to analyze iodine or iodine in the common salt, which is a common ingredient of daily food. However it is observed that all the manufacturers of iodized salt are not adding required amount of iodine or iodide. Hence a national planning and regulation should be made to make it compulsory for adding required quantity.