Testing Protocol

Iodine Estimation of Salt

Laboratory Procedure for Iodine Estimation of Salt

Background material:

Iodine is one of the first minerals recognized as essential for human health. Iodine belongs to the family of halogens (Chlorine, bromine, iodine and fluorine) placed in the seventh group of period table. Most of the iodine exists in the ocean. It was present during the primordial development of the earth but large amounts were leached from the surface soil by glaciations, snow, or rain and were carried by rivers, floods and winds into the sea.

Iodine is an essential micronutrient for humans and is required in a very small amount i.e. 150 µg per day. The only role of iodine in the body, known at present is for the synthesis of thyroid hormones. The human body contains 15 to 20 mg of iodine of which almost 80% is in the thyroid gland. The iodine taken in the diet is absorbed throughout gastrointestinal tract and circulates as plasma Inorganic Iodide (PII) in the body. The PII is mainly cleared by two organs in the body - Thyroid and Kidney. The iodine taken up by the thyroid gland is used for making thyroid hormones, thyroxine and triiodothyronine. Thyroxine is called T_4 because it has 4 iodine atoms in its structure while triiodothyronine is called T_3 because it has 3 iodine atoms in its structure. Iodine taken up by the kidney is excreted in the urine. The level of excretion in urine correlates well with level of intake. Thus urinary iodine can be used to assess the level of iodine intake.

Thyroxine has two major roles in the body one is for development of brain, central nervous system and skeletal system during the early developmental stages and the second is calorigenic effect i.e. increase in oxygen uptake by tissues thereby controlling all metabolic processes in the body.

Iodine deficiency occurs mainly as an environmental deficiency. Iodine present in upper crust of earth is washed/leached out by repeated flooding and glaciation. Thus making the soil deficient in iodine. Crops grown on such soil will have less iodine and when consumed by humans or animals lead to iodine deficiency. Thus it is the deficiency of soil which is responsible for iodine deficiency in humans and animals.

Iodine deficiency during pregnancy and first two to 3 years of life leads to derangement in the development of brain and central nervous system & skeletal. These changes are irreversible. No amount of iodine or thyroxine can revert back the damage done during the developmental period leading to several disabilities like deaf mutism, squint, gait defects etc. but the most important of these is the loss of learning ability with loss of 13 IQ points. These can be easily prevented by supplying proper amount of iodine during this period.

Since there is no natural food item which will help in supplying adequate amount of iodine, it is essential to supplement or fortify the food item for meeting the iodine requirements of the individual.

There are several modes of iodine supplementation used. These are: Iodised oil, Iodised capsules, Iodised bread, Iodised water, Iodised salt

Out of these the most effective method is iodised salt. This is because salt is one food item which is taken in a fixed amount everyday by everybody whether rich or poor, old or young. Thus it is an ideal vehicle for supply of constant amount of iodine to everybody daily. It is also most economical way of supplying iodine. Government of India had taken a decision of supply of iodine via iodised salt from 1962 under the National Goitre Control Programme.

The iodised salt may lose iodine due to moisture and heat during transportation. To avoid this the PFA (Prevention of Food Adulteration) Act states that iodine content of salt should not be less than 30 ppm at production and 15 ppm at consumer level. It is essential to monitor the amount of iodine in salt by quantitative method (Iodometry). The details of procedure are given.

5.2) Principle and Laboratory Procedures for Iodine Estimation in Salt

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I) Principle

The iodine content in iodated salt is estimated by a process called iodometric titration. Free iodine reacts with sodium thiosulphate solution as follows:

$2Na_2S_2O_3$	+	I_2	=	2NaI	+	$Na_2S_4O_6$
Sodium thiosulphate		Iodine		Sodium iodide		Sodium tetrathionate

II) Equipment and Chemicals

a) Equipment

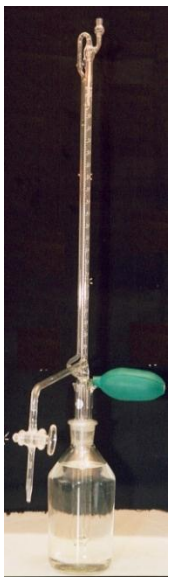
1. Laboratory balance for preparing reagents
2. Beakers – 100, 200, 500 ml
3. Glass bottles with stoppers for reagents:

   1,000 ml

   250 ml
4. Open pan balance for weighing salt samples

5. Measuring cylinders with stopper 50 ml
6. Wash bottle 500 ml
7. Conical flasks with stopper 200 ml
8. Glass or plastic funnel
9. Auto dispensers

10. Burette 10 ml auto zeros



b) Chemicals

1. Sodium thiosulphate - $Na_2S_2O_3$, Analytical Regent Grade (AR)
2. Concentrated sulphuric acid – H2SO4, (AR)
3. Potassium iodide – KI, (AR)
4. Soluble chemical starch
5. Boiled double-distilled water, pharmaceutical grade

The approximate cost of reagents would be Rs. 1200, which would analyze 100 salt samples.

III) Preparation of Reagents

a) Sodium thiosulphate ($Na_2S_2O_3$): Dissolve 1.24 grams in 1 litre double-distilled water. Store the solution in a cool and dark place. Normality may change as time progresses. It is advisable to prepare small quantity of 0.005 N $Na_2S_2O_3$ each day as required to avoid change of normality and storage problems.

b) 2.N Sulphuric acid ($2H_2SO_4$): To 90 ml double-distilled water, add 5.56 ml concentrated H_2SO_4 slowly. Add double-distilled water to make 100 ml. Store in a cool dark place. The solution may be kept indefinitely.

Caution: To avoid violent and dangerous reaction always add acid to water, never water to acid.

c) Potassium iodide (KI, AR): Dissolve 10 grams KI in 100 ml double-distilled water. Store in a cool, dark place. Properly stored, the solution may be kept for 6 months.

d) Saturated Sodium Chloride (NaCl): To boiling double distilled water go on adding sodium chloride while stirring until no more of it dissolves. Cool the solution

e) **Soluble Chemical Starch**: Weigh 1 gram of soluble starch and dissolve in 10 ml double distilled water. Add 90 ml of saturated sodium chloride solution to make it up to 100 ml. Add a pinch of sodium benzoate as a preservative.

IV) Procedure

Weigh 10 grams of salt and put it in a stoppered conical flask. Add 50 ml of distilled water and dissolve the salt. Add 1 ml of 2N H_2SO_4 followed by 1 ml of 10% KI solution with the help of an automatic dispenser and close the flask with a stopper. If the iodine is present, the solution will turn yellow. Keep the flask in the dark (for e.g. in a closed cupboard) for 10 minutes to avoid exposure to light. Remove the flask and titrate against 0.005N $Na_2S_20_3$. During the titration, when the yellow colour becomes pale, add 2 drops of the starch solution as an external indicator. The solution will become purple. Continue titration till the solution becomes colourless. Note the burette reading. To calculate the iodine content in parts per million (PPM), refer to the **Table – 8**.

Table 8: Iodine Content in Parts Per Million (PPM)

Burette reading	Parts Per Million (PPM)	Burette reading	Parts Per Million (PPM)
0.0	0.0	2.6	27.5
0.1	1.1	2.7	28.6
0.2	2.1	2.8	29.6
0.3	3.2	2.9	30.7
0.4	4.2	3.0	31.7
0.5	5.3	3.1	32.8
0.6	6.3	3.2	33.9
0.7	7.4	3.3	34.9
0.8	8.5	3.4	36.0
0.9	9.5	3.5	37.0
1.0	10.6	3.6	38.1
1.1	11.6	3.7	39.1
1.2	12.7	3.8	40.2
1.3	13.8	3.9	41.3
1.4	14.8	4.0	42.3
1.5	15.9	4.1	43.4
1.6	16.9	4.2	44.4
1.7	18.0	4.3	45.5
1.8	19.0	4.4	46.6
1.9	20.1	4.5	47.6
2.0	21.2	4.6	48.7
2.1	22.2	4.7	49.7
2.2	23.3	4.8	50.8
2.3	24.3	4.9	51.9
2.4	25.4	5.0	52.9
2.5	26.5	5.1	54.0

Burette reading	Parts Per Million (PPM)	Burette reading	Parts Per Million (PPM)
5.2	55.0	7.7	81.5
5.3	56.1	7.8	82.5
5.4	57.1	7.9	83.6
5.5	58.2	8.0	84.6
5.6	59.2	8.1	85.7
5.7	60.3	8.2	86.8
5.8	61.4	8.3	87.8
5.9	62.4	8.4	88.9
6.0	63.5	8.5	89.9
6.1	64.5	8.6	91.0
6.2	65.6	8.7	92.0
6.3	66.7	8.8	93.1
6.4	67.7	8.9	94.2
6.5	68.8	9.0	95.2
6.6	69.8	9.1	96.3
6.7	70.9	9.2	97.3
6.8	71.9	9.3	98.4
6.9	73.0	9.4	99.5
7.0	74.1	9.5	100.5
7.1	75.1	9.6	101.6
7.2	76.2	9.7	102.6
7.3	77.2	9.8	103.7
7.4	78.3	9.9	104.7
7.5	79.4	10.0	105.8
7.6	80.4		

5.3) Calculations

The table of iodine content as determined by the burette reading has been prepared based on the following:

1) 1 ml of 0.005N $Na_2S_2O_3$ = 0.1058 mg of iodine

2) Thus, the burette reading X 0.1058 will give the amount of iodine in 10 gm of salt

3) To get the iodine value in parts per million, one has to multiply by 1,00,000 to either sides

Gms of salt	I_2 in mg
10 X 1,00,000	0.1058 X 1,00,000

4) To convert mg in to gm, divide by 1000

Equation becomes:

Gms of salt (I0,00,000) = Burette Reading X 0.1058X 100

= Burette Reading X 10.58

Thus Burette reading X 10.58 will give the iodine content in parts per million.

5.4) Precautions

Adding sulphuric acid to a solution of iodated salt liberates iodine, which is titrated with sodium thiosulphate. Starch is used as an external indicator. Potassium iodide solution is added to keep the iodine in the dissolved state.

1. The starch solution must be added near the end of the titration, when very little iodine is left and the solution has a faint-yellow colour. If starch is added earlier, the iodine starch complex becomes very strong and reacts too slowly with sodium thiosulphate, resulting in false high readings.

2. The titration should be done in a comfortably cool room because iodine is volatile and the sensitivity of the starch indicator diminishes as the temperature rises.

3. Potassium iodide (KI) is used because of the low solubility of iodine. The liberated iodine forms an unstable complex KI_3 with KI:

   $KI + I_2 = KI_{-3}$ and $I^- + I_2 = I_3$

   As free iodine is used up in the reaction with thiosulphate, the equilibrium between I_2 and I_3 ions is disturbed and more iodine is dissolve in order to maintain the equilibrium.

4. A few minutes should be allowed before titration, since the rate of reaction between I^- ions and the oxidant is slow.

5. The reaction mixture should be kept in the dark before titration because light accelerates a side reaction in which iodide ions are oxidized to iodine by atmospheric oxygen.