National Iodine Deficiency Disorders Control Program (NIDDCP)

Estimation of Iodine Content in Iodised Salt by Iodometric Titration

Principle

The principle is that iodine is liberated by adding sulphuric acid to a solution of iodised salt. Potassium iodide solution is added to keep the iodine in the dissolved state. Iodine liberated is titrated with sodium thiosulphate solution to form sodium iodide and sodium tetrathionate. Starch is used as an external indicator.

$$KIO_3 + 5KI + 3H_2SO_4 \text{ -----> } 3K_2So_4 + 3I_2 + 3H_2O$$

$$KI + I_2 \text{ ------> } KI_3$$

$$2Na_2S_2O_3 + I_2 \text{ -------> } 2NaI + Na_2S_4O_6$$

Equipment and Chemicals

Equipment

Chemical balance/Electronic balance/Electrical balance
Gas burner
Reagent bottles with stoppers à 250 ml, 500 ml & 1000 ml
Measuring cylinder with stopper, 50 ml
Wash bottle, 500 ml
Glass stirring rod
Conical flask with stopper, 100 ml
Glass or plastic funnel
Burette, 50 ml
Burette, stand
Pipette – 25 ml
A closed box, cupboard or drawer to keep the conical flask
Physical balance (capacity 50 – 100 gms)

Chemicals

Sodium thiosulphate, ($2Na_2\ S_2\ 0_3\ 5H_2O$)
Concentrated sulfuric acid, (H_2SO_4)
Potassium iodide (KI)
Soluble chemical starch
Potassium Iodate

All the chemicals should be analytical grade and double distilled water to be used which should be free of iodine and other contaminants.

Preparation of Standard solutions and reagentsSodium thiosulphate (0.005 M)

Dissolve 1.24 g sodium thiosulphate in 1 litre hot double distilled water. This volume will be sufficient for testing 200 salt samples. The solution should be stored in a cool, dark place.

Sulfuric acid (2N H_2SO_4)

Add 5.56 ml concentrated sulfuric acid drop wise into a 90 ml of chilled double distilled water and make the final volume upto 100 ml with double distilled water. This volume will be sufficient for testing 50 salt samples. **CAUTION:** To avoid violent and dangerous reaction, always add acid to water, never water to acid.

Potassium iodide (KI)

Dissolve 100 g potassium iodide in 1000 ml double distilled water. This volume will be sufficient for testing 200 salt samples. This should be stored in a refrigerator.

Saturated Salt Solution

Take 100 ml double distilled water in a conical flask and add sodium chloride until the salt is insoluble. Heat the solution till the NaCI crystals are formed on the sides of the vessels. After cooling down the saturated salt solution at room temperature transfer the supernatant to a clean bottle.

Starch(1%)

Take 1 g of starch and prepare a slurry in 50 ml water. Add this slurry slowly to 50ml of boiling water.

Standardistaion of Sodium Thiosulphate solution

The sodium Thiosulphate solution is to be standardized with Standard Potassium iodate solution to determine its exact normality. Take 0.005 N thiosulphate solution in a burette. Pipette out 25 ml of 0.005 N standard potassium iodate solution in a conical flask. Add 2 ml of 2N $H_2 So_4$ and 5 ml of 10% KI solution. Titrate the solution against sodium thiosulphate till the solution becomes pale yellow in colour. To this, add 1 ml starch. The solution turns deep purple. Add thiosulphate sulphate solution drop by drop from the burette till the purple colour completely disappears.

Normality of Thiosulphate is calculated as follows :

Volume of KIO_3 **(V_1)** x Normality of KIO_3 **(N_1)** **=** Volume of $Na_2S_2O_3$ **(V_2)** x Normality of $Na_2S_2O_3$ **(N2)**

$$\text{Normality of Sodium Thiosulphate} = \frac{V_1 \times N_1}{V_2}$$

Procedure

Dissolve 10 g of salt in 50 ml double distilled water. Then add 1 to 2 ml of 2 N sulfuric acid and 5 ml of 10% potassium iodide to it. On shaking, the solution will turn to a yellow colour. Close the flask with stopper and keep the flask in the dark for about 10 minutes. Remove the samples from the dark and titrate against the sodium thiosulphate solution until it turns into a very light yellow colour (pale yellow). Subsequently, add a few drops (1-5 ml) of 1 % starch solution. The solution will turn into a deep purple colour. Add thiosulphate drop by drop from the burette until the solution becomes colourless and note the final reading.

The iodine content of the sample in parts per million is calculated by the formula:-

$$\text{Iodine Content (ppm)} = \frac{R \times 100 \times 10^3 \times 0.127 \times N}{6}$$

where R - The Volume of Thiosulphate used (burette reading)

100 - For converting burette reading for 1000gm of salt

10^3 - For converting gms of iodine to milligrams

0.127 - The weight of Iodine equivalent to 1ml of normal sodium thiosulphate solution

N - Normality of sodium thiosulphate solution

6 - To arrive at the value of 1 atom of iodine liberated

Precautions

The reaction mixture should be kept in the dark for 10 minutes before titration because light accelerates a side reaction in which iodide ionsare oxidized to iodine by atmospheric oxygen.

Starch solution must be prepared fresh everyday because it cannot be stored

The starch solution must be added near the end of the titration, when very little amount of 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.

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

Sodium thiosulphate if stored for more than a week should be standardized using potassium iodate

Potassium iodide is used because of low solubility of iodine. The liberated iodine forms an unstable complex KI_3 with KI. A few minutes should be allowed before titration, since the rate of reaction between I ions and the oxidant is slow.

USE OF SPOT TESTING KIT

A simple kit has been developed for on the spot estimation of iodine content. In the spot testing method, estimation of iodine content in salt is done with the help of a standard starch solution provided in the Spot Testing Kit (STK). The STK has been advocated as a method for semi-quantitative estimation of iodine in salt. The STK consists of a test solution (A), and a recheck solution (B). One drop of test solution (A) will be added to a pinch of salt sample. The change in colour of salt sample will range from white to dark violet depending on the iodine content of salt, *i.e.* Nil, less than 15ppm, and 15 ppm and above, which will be compared to the standard colour chart provided with the kit. If on addition of test solution (A), no change in colour of salt is observed, the recheck solution (B) will be added. This will be done to make the salt medium acidic, in case the salt has alkaline constituents, then the test solution (A) will be added again. The intensity of the blue colour will be directly proportionate to the iodine content of salt

Procedure for use of Spot Testing Kit

Take a spoonful of iodated salt to be checked and spread it flat.

Open the seal of the ampule (white cap) by making a pin hole.

Discharge a drop of the test solution on the surface of the salt by gently pressing the ampule.

The salt will turn light blue to dark violet depending on the iodine content of the salt.

Use the colour chart given on plastic box to compare and determine the iodine range in the iodated salt.

IF THE SALT IS ALKALINE OR MIXED WITH ALKALINE FREE FLOW AGENTS, A DROP OF TEST SOLUTION WILL NOT DEVELOP ANY COLOUR EVEN IF IODINE IS PRESENT, WHENEVER ONE GETS NO COLOUR INDICATION PLEASE ADOPT PROCEDURE GIVEN BELOW:

Take a spoon of salt on plate and level it

Add one drop of Recheck solution (Red cap)

Over the same spot, put one drop of test solution (white cap)

If iodine is present colour will develop. If Iodine is not present no colour indication will be there.

Note: Shelf life of spot testing kit is 18 months.

EXTERNAL EVALUATION OF NIDDCP:

An Evaluation of the status of NIDDCP in India was carried out during 1996 by the Canadian International Development Agency (CIDA) which felt that the story of USI in India was one of the remarkable achievements as it has made spectacular progress towards the goal of USI. The study revealed that the Salt Department has been successful in ensuring the supply of good quality iodised salt for human consumption and cited that the Role of the Salt Department is one of the seven issues that has an important bearing on the future sustainability of Universal Salt Iodisation. The study also commended India for offering two technologies : iodizing machinery and the spot testing kit which are important tools for producing good quality iodised salt.

INTERNAL EVALUATION OF NIDDCP:

The Government of India, Ministry of Industry with support of UNICEF carried out an Internal evaluation of the USI component of the programme during 1997-98. The study carried out by the Indian Institute of Health Management & Research, Jaipur, concluded that not only has the capacity and production of iodised salt in the country increased considerably, but the movement and distribution also have been streamlined. Further, the consumption of iodised salt at the household levels in urban and rural areas has also showed Improvement. Nevertheless, to maintain the significant achievements in universal accessibility and availability of iodised salt, the study concluded that efforts are needed to consolidate the gains, bridge the existing gaps and deficiencies, and strengthen the programme efforts to sustain the production and consumption of iodised salt. It also commended that the Salt Department has made a significant contribution towards the universal iodisation of salt and has played a crucial role in increasing the production of iodised salt and its distribution throughout the country.

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