Exp. 2

AIM: Estimation of Iodine in common salt using Iodometry.

Apparatus Required: Pipette, burette, measuring cylinder, distilled water, droppers, conical flask

Chemicals Required: Iodised common salt, 10% KI, 5N HCl, 0.5% w/v starch solution, 1% sodium nitrate, 20% v/v H_2SO_4, $Na_2S_2O_3$, $CuSO_4.5H_2O$, KSCN

Principle: Iodometry i.e. dealing with the titration of Iodine liberated in chemical reactions, is one of very effective, easiest and accurate method to estimate the amount of oxidising agent present, or to estimate the amount of iodine in iodised salt.
Because of thiosulphate is not a primary standard (it is a secondary standard) so it has to be standardized against primary standards.

Thiosulphate is oxidised by Iodine / tri-Iodide to tetrathionate ion and this reaction is finally used for estimation of Iodine.

The reaction proceeds forward quantatively in slightly acidic medium. In strongly acidic / alkaline medium it leads to errors much due to side reactions.

Also some countries has Iodide and some has Iodate in their salt, so we've also to check that which is present.

For standardization Sodium Thiosulphate is titrated against solution of $CuSO_4$. In this $CuSO_4$ is first titrated with Iodide and then thiosulphate is added dropwise and end pt. can be known with use of starch solution indicator. Also KSCN is added 5-6 drops so that I_3^- is not much complexed with starch so that it gets completely reacted.

During this titration as the solution becomes colourless end point is supposed to be attained but sometimes if we wait for 10 (say) seconds colour comes back so then 2-3 drops extra thiosulphate so it gets fully titrated.

Now, to identify Iodate or Iodide is present a spot test is to be done initially, so that we can proceed further accordingly.

Reactions:

1.) $I^- + I_2 \longrightarrow I_3^-$ (highly soluble in water)

2.) $IO_3^- + 5I^- + 6H^+ \longrightarrow 3I_2 + 3H_2O$

3.) $2NaNO_2 + H_2SO_4 \longrightarrow 2HNO_2 + Na_2SO_4$

$2HNO_2 + 2I^- \longrightarrow 2NO + H_2O$

4.) $2Cu^{+2} + 4I^- \longrightarrow Cu_2I_2 + I_2$

5.) $I_2 + S_2O_3^{2-} \longrightarrow S_4O_6^{2-} + 2I^-$

Procedure: A) Rapid Test for Iodide / Iodate

1.) Take a pinch of salt on watch glass and divide it into 2 parts.

a) Test for Iodide:

Add 2-3 drops of mixture of:

→ 0.25 ml 1% $NaNO_2$.
→ 0.25 ml 20% V/V H_2SO_4
→ 25 ml 0.5% w/v starch solution

if iodide is present the salt will immediately turn dark blue. ~~purple~~

$2NaNO_2 + H_2SO_4 \longrightarrow 2HNO_2 + Na_2SO_4$

$2HNO_2 + 2I^- \longrightarrow 2NO + H_2O$

b) Test for Iodate :

Add 2-3 drops of mixture of

→ 25ml 10% KI
→ 0.6ml 5N HCl
→ 25ml 0.5% w/v starch solution

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

if Iodate is present the salt will immediately turn dark blue.

B) Standardization of Sodium Thiosulphate

1) Pipette out 10ml of copper sulphate solution (0.005M) in a conical flask and then add 5ml of 5% KI solution.

2) Titrate it with sodium thiosulphate solution until the solution turns pale yellow from yellow, add 7-8 drops of starch indicator and keep adding thiosulphate.

3) Continue titration until violet coloration fades, add 5-6 drops of KSCN solution, and titrate till colourless end pt.

4) Now as said earlier, if we wait for few seconds it may generate coloration back so continue titration until a final end pt.

5.) Note the reading, calculate molarity of thiosulphate & repeat to get consistent values.

C) Determination of Iodine content by Iodometry

(If test 'b' was +ve)

1. Weigh 1.3 g of the salt and make 50ml of solution.
2. Add 1 ml 2N H_2SO_4 (for acidic medium)
3. Add 5ml 10% KI, solution turns yellow
4. Cover the flask immediately and place in dark for 10 mins (as reaction is slow, & light accelerates side reaction)
5. Wash the sides of flask with distilled water
6. Use a rinsed burette and fill it with standardized $Na_2S_2O_3$ and adjust level to zero.
7. Add 2ml of starch indicator, solution turns dark purple (to be added before the eq. point) (use freshly prepared starch indicator)
8. Continue titration until the solution becomes colourless
9. Repeat the experiment 3 times.

Observation & Calculations:

Standardization of Sodium Thiosulphate

	Vol. of $CuSO_4$ sol: (0.005M) taken (in ml)	Vol. of $Na_2S_2O_3$ (in ml)	Concordant value
1.	10ml	9.8ml	
2.	10ml	9.8ml	9.8ml
3.	10ml	9.8ml	

$$2Cu^{+2} + 2I^- + 2S_2O_3^{2-} \longrightarrow Cu_2I_2 + S_4O_6^{2-}$$

Moles of $S_2O_3^{2-}$ = Moles of Cu^{+2}

$$M_1V_1 = M_2V_2$$

$$M_1(9.8) = (0.005)(10)$$

$$\boxed{M_1 = 0.0051\ M}$$

Titration against salt sample

	Vol. of salt sol. (in ml)	Volume of $S_2O_3^{2-}$ used (in ml)	Concordant value
1	50ml	3.5ml	
2	50ml	3.5ml	3.5ml
3	50ml	3.5ml	

$$IO_3^- + 6S_2O_3^{2-} + 6H^+ \longrightarrow 3S_4O_6^{2-} + 3H_2O + I^-$$

$$6 \times M_1V_1 \text{ (Iodate)} = M_2V_2 \text{ (Thiosulphate)}$$

$$6 \times M_1 \times 50 = 0.0051 \times 3.5$$

$$M_1 = \frac{0.0051 \times 3.5}{50 \times 6} = 5.95 \times 10^{-5}$$

Molarity of Iodate solution = 5.95×10^{-5}

$\Rightarrow$ At. wt. of Iodine = 126.90 g

$\Rightarrow$ 1 litre solution contains $5.95 \times 10^{-5} \times 126.90$ g of Iodine

= 7.55 mg Iodine per litre solution

Concentration in ppm –:

we started with 13g of salt dissolved in 50ml of water

so, 50ml solution contains,

$$7.55 \times 10^{-3} \times \frac{50}{1000} g = 3.77 \times 10^{-4} g \text{ of Iodine}$$

Thus, 13g of salt contains 3.77×10^{-4} g of Iodine

10^6 g contains $\frac{3.77 \times 10^{-4}}{13} \times 10^6 = 29$ g of Iodine

$\Rightarrow$ 29ppm

Result:

1.) Iodate ions were identified in the given salt

2.) Concentration of Iodate ions was determined using iodometric titrations.

3.) Concentration of Iodine (in ppm) in given salt sample = 29ppm

Precaution:

1.) Burette, pipette, conical flasks should be correctly washed/rinsed

2.) Check no air bubble in burette.

3) Use funnel for filling and remove it after filling also.

4) Iodine, H_2SO_4, KI solutions must be kept away from light.