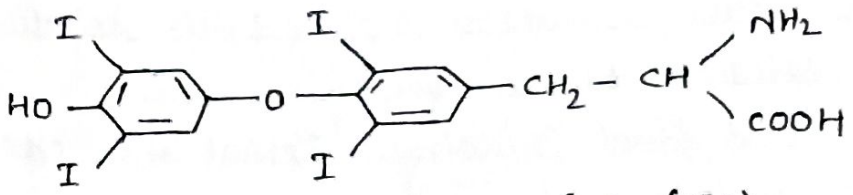
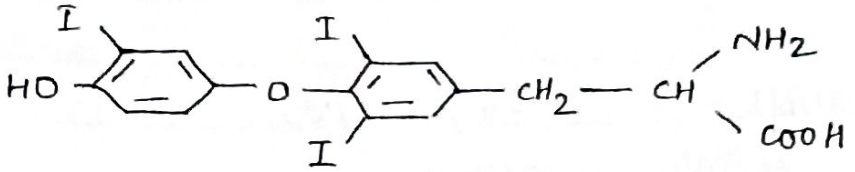
Structures:



Thyroxine (T4)



Triiodothyronine (T3).

Reactions Involved:

Rapid Tests:

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

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

$$2HNO_2 + 2I^- \longrightarrow I_2 + 2NO + H_2O.$$

Standardisation:

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

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

Net Rxn:

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

Estimation:

$$2Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2NaI$$

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

Date 18-12-2021

Expt. No. 2 Page No. 4.

Aim: Estimation of Iodine in iodised common salt using iodometric titration.

Apparatus Required: Burette, pipette, standard flasks.

Chemicals Required: Iodised soluble salt (10% KI), $NaNO_2$ (1%), HCl (5N), 20% w/v H_2SO_4, 0.5% w/v starch solution, distilled water, $Na_2S_2O_3$, $CuSO_4$ solution (0.005M), distilled water, iodised common salt.

Principle: Iodometry is used in determination of lipid hydroperoxide in serum, estimation of peroxide, etc. It is an excellent tool to estimate the amount of iodine present in common salt. The most common and universal titrate for iodine is Sodium Thiosulphate ($Na_2S_2O_3$) which is not a primary standard. So, A solution of sodium thiosulphate needs to be standardised which we do by using $CuSO_4$ solution. The reaction proceeds quantitatively in acidic or neutral medium. In strongly alkaline medium, oxidation of thiosulphate doesn't proceed irreversibly which leads to errors. In the solution with no other coloured species, it is possible to see the colour of 5×10^{-6} M $I_3^{\ominus}$. Starch shouldn't be added until after first equivalence point, otherwise some starch remains bounded to iodine. In titration involving Cu^{+2}, the precipitate Cu_2I_2 tends to bind to iodine. Thiocyanate is added to prevent this. The actual structure of the starch-iodine complex involves $I_5^{\ominus}$ and amylose. The procedure defined below has two iodometric reactions but before them, a rapid test to determine the form in which iodine is present.

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Observations and calculations:

Standardisation of $Na_2S_2O_3$:

S.No	Volume of Sodium Thiosulphate used
1	9.8 ml
2.	9.8 ml
3.	9.8 ml

Concentration of $CuSO_4$ = 0.005 M

Molarity x Volume of $CuSO_4$ = molarity x volume of $Na_2S_2O_3$

(Normality = molarity here as n factor = 1)

$0.005 \times 10 = M_1 \times 9.8$

$M_1 = 0.0051$ Molar.

Estimation of Iodine:

S.No.	Volume of Sodium Thiosulphate used.
1.	3.5 ml
2.	3.5 ml
3.	3.5 ml

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

6 x Molarity x Volume of Iodate = Molarity x volume of $Na_2S_2O_3$

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

$M = 0.0000595 = 5.95 \times 10^{-5}$

Expressing in Parts per Million:

5.95×10^{-5} mole Iodine is present in 1L of solution.

Atomic weight of Iodine = 126.90 μ

So, weight of Iodine per litre solution

$= 5.95 \times 10^{-5} \times 126.90 =$ 0.755 mg

weight of Iodine in 50 ml solution

$= \frac{0.755 \times 500}{1000} =$ 0.3775 mg.

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Procedure:

(A) Rapid Test

(1) Take a pinch of common salt and divide into two parts.

(2) To the first part, add 10% KI solution (25 ml), 0.6 ml 5N HCl and 25 ml 0.5% w/v starch solution. If blue-violet colouration is observed, the salt contains KIO_3^- ion.

(3) To the second part, add $NaNO_2$, H_2SO_4 and starch solution. If blue violet colouration is observed, the salt contains $I^{\ominus}$ ions.

(B) Standardisation of Sodium Thiosulphate.

(1) Pipette out 10 ml of $CuSO_4$ of 0.005 M concentration in a conical flask and add 5 ml of 5% KI solution. The solution will turn yellow.

(2) Titrate with $Na_2S_2O_3$ solution until solution turns ~~purple~~ pale yellow. Add 7-8 drops of starch at this stage.

(3) Continue titration until the purple colour fades. then add 5-6 drops of KSCN solution and titrate again. The endpoint gives a colourless solution.

(4) Note the burette reading and find the molarity of $Na_2S_2O_3$.

(C) Estimation of Iodine.

(1) Weight X g (=10) of salt and make 50 ml solution. To this, add 2N H_2SO_4 (1 ml).

(2) Add 5 ml 10% KI solution, the solution turns yellow.

(3) Stopper the flask immediately and keep in dark for 10 mins.

(4) Fill burette with standardised $Na_2S_2O_3$ and keep the level to zero.

(5) Wash the sides of the flask with distilled water.

(6) Titrate until the solution turns yellow.

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In 13 g salt, 0.3775 mg of Iodine is present.

$\therefore$ parts per million of Iodine $= \frac{0.3775}{13} \times 1000$

$= 29.03$ ppm.

(7) Add 2 ml starch solution, the solution turns dark purple.
(8) Continue titration until the solution becomes colourless.
(9) Note the burette reading and repeat the experiment three times.

Result.

(1) Positive for Iodate solution.
(2) Molarity of $Na_2S_2O_3$ solution = 0.0051M
(3) Amount of Iodine = 29.03 ppm.

Precautions.

(1) Burette, pipette and flask should be rinsed properly.
(2) Check there is no air bubble in burette.
(3) Swirl flask during titration.
(4) Read the lower meniscus while taking the readings.
(5) Freshly prepared starch be used as indicator.

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