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EXPERIMENT NO. 3

AIM:

To determine the amount of dissolved oxygen in given water sample.

APPARATUS REQUIRED:

Burette, pipette, retort stand, funnel, beakers, iodometric flask and measuring cylinders.

CHEMICALS REQUIRED:

Manganese sulphate (Alkaline), Alkaline iodide azide, sulphuric acid, freshly prepared starch solution, N/40 $Na_2S_2O_3$ i.e. Hypo solution.

THEORY:

Dissolved oxygen in boiler feed water causes corrosion of boiler plates. Its determination if present in water, is therefore essential.

The mechanism of corrosion is as follows:

Iron in contact with water exerts a certain solution pressure. It, thus, passes from the metallic state to that of mobile ions and gains positive charge, loosing its electrons. Thus an oxidation or anodic half reaction is set up. On the other hand, dissolved oxygen reacts with water to give hydroxyl ions at cathode areas. The hydroxyl ions now combine with the ferrous ions present in water, to form ferrous hydroxide ($Fe(OH)_2$), the electrons neutralise each other with a flow of current between the adjacent anode and cathode areas.

The reactions of this electrochemical corrosion may be illustrated as:

At anode areas,

$$Fe_{(s)} = Fe^{+2}_{(s)} + 2e^-$$

Teacher's Signature :

Chemical Reactions:

$$KOH + MnSO_4 \rightarrow Mn(OH)_2 + K_2SO_4$$

$$Mn(OH)_2 + O_2 \rightarrow MnO(OH)_2$$

(Brown ppt.)

$$MnO(OH)_2 + H_2SO_4 \rightarrow MnSO_4 + H_2O + [O]$$

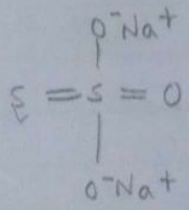
Nascent oxygen

$$KI + H_2SO_4 + [O] \rightarrow K_2SO_4 + I_2$$

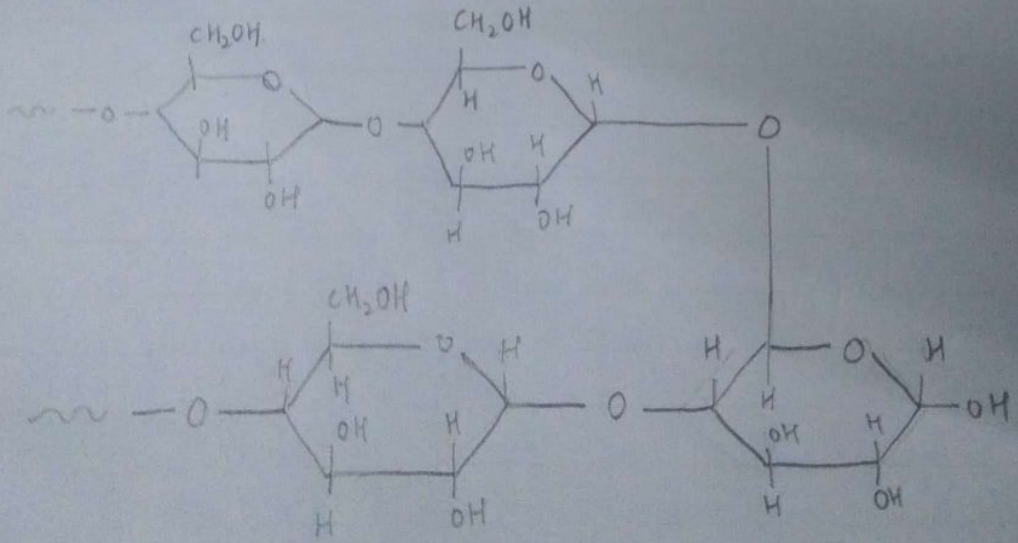
$$I_2 + Na_2S_2O_3 \rightarrow Na_2S_4O_6 + NaI$$

$$HNO_2 + HN_3 \rightarrow N_2O + N_2 + H_2O$$

Hypo Sola.:



Starch Indicator: $(C_6H_{10}O_5)_n$



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$$Fe^{+2} + 2OH^- \longrightarrow Fe(OH)_2$$

$$Fe(s) + 2OH^- \longrightarrow Fe(OH)_2 + 2e^-$$

At cathode areas,

$$O_2(g) + 2H_2O(l) + 4e^- \longrightarrow 4OH^-(aq.)$$

Total corrosion reaction is:

$$2Fe(s) + O_2(g) + 2H_2O(l) \longrightarrow 2Fe(OH)_2$$

The above reactions - reveal the dissolved oxygen in water accelerates the iron - water corrosion.

The experiment is based on the oxidation of potassium iodide by the dissolved oxygen. The liberated iodine is titrated against a standard sodium thiosulphate solution using starch as final indicator. Since, oxygen dissolved in water remains present in molecular state; it is not capable of reacting with potassium iodide. An oxygen carrier such as manganese hydroxide is, therefore, used to bring about the reaction. Manganese hydroxide is produced in situ by the action of potassium hydroxide and manganous sulphate. The chemical reactions taking place as:

$$MnSO_4 + 2KOH \rightarrow Mn(OH)_2 + K_2SO_4$$

$$2Mn(OH)_2 + O_2 \rightarrow 2MnO(OH)_2$$

[Basic manganese oxide]

$$MnO(OH)_2 + H_2SO_4 \rightarrow MnSO_4 + 2H_2O + O$$

$$2KI + H_2SO_4 + [O] \rightarrow K_2SO_4 + H_2O + I_2$$

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

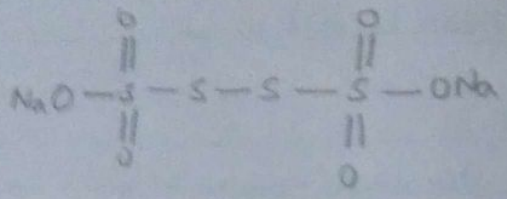
(Sodium tetrathionate)

PROCEDURE:

1. Wash all the apparatus with water and fill in burette with hypo solution.

Teacher's Signature :

Sodium Tetra thionate :



OBSERVATION TABLE :

S.No. and colour change	Initial Reading (mL)	Final Reading (mL)	Volume used (mL)
1. Brown to light yellow	0.0	62.6	62.7
2. Blue to colourless	62.7	63.6	0.9

Total volume of N/40 $Na_2S_2O_3$ soln. used = 63.6mL

Vol. of water used = 50mL

CALCULATIONS :

$$N_1V_1 = N_2V_2$$

(Hypo soln.) (Given water sample)

$$\frac{N}{40} \times 63.6 = N_2 (50)$$

$$N_2 = \frac{63.6}{2000} N = 0.0318N$$

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2. Take 50mL water sample iodometric flask and close it to avoid contact with air.
3. Immediately add 0.5 mL of manganous sulphate solution from the burette and 0.5 mL of alkaline iodide from the other burette.
4. Insert the stopper and shake several times for at least 20 minutes.
5. Allow the precipitate to settle half way and mix again.
6. Repeat this process of shaking. Add 1mL of conc. sulphuric acid and insert the stopper and shake again.
7. Allow the yellow solution to stand for 5 minutes.
8. Titrate it against the standard N/40 sodium thiosulphate solution till the colour of the solution becomes faint yellow. At this stage, add a few drops of starch solution. The colour turns to deep blue due to formation of starch iodide.
9. Continue titration till the solution becomes colourless.

RESULT :

Amount of dissolved oxygen present in given water sample = 254.4 ppm.

PRECAUTIONS :

1. The apparatus was rinsed with the solution to be taken in it.
2. The lower meniscus of burette solution was read.
3. Azide soln. was added to remove the interference of the nitrites present.
4. The stopper flask was not opened again and again, else the error would have been introduced.

Teacher's Signature :

Strength of dissolved oxygen,

$= 0.0318 \times 8$ g/L

$= \underline{0.2544}$ g/L

$= 254.4$ mg/L

$= \underline{\underline{254.4 \text{ppm}}}$