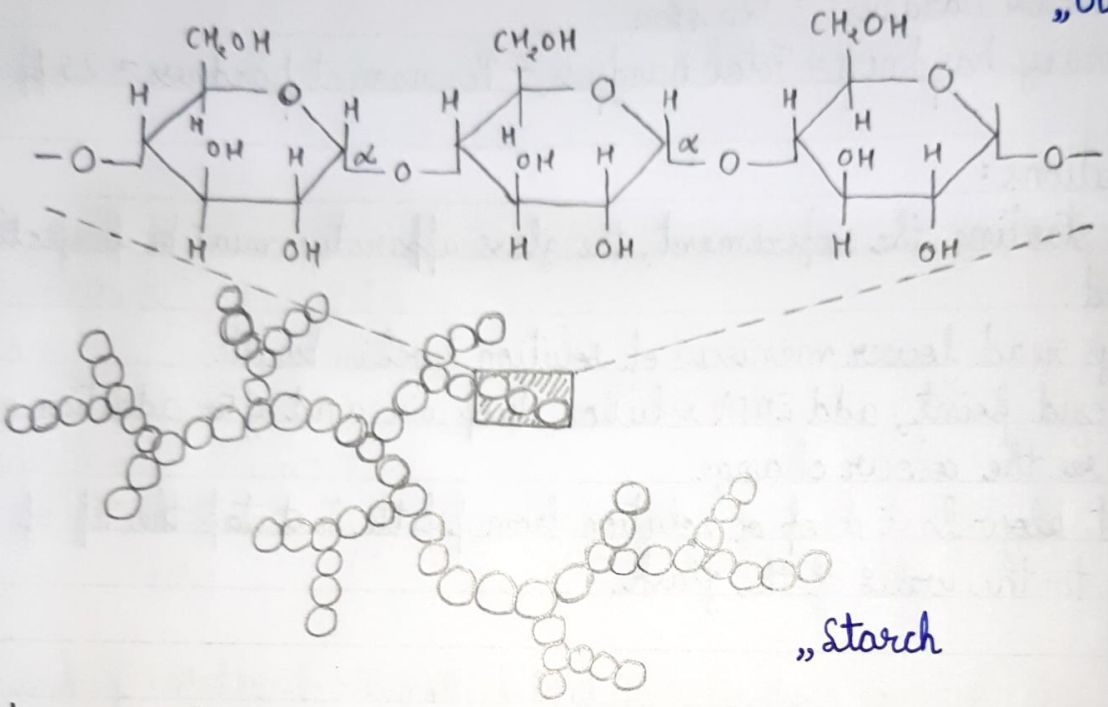
Indicator used:

Starch is used as an indicator in iodometric titration.



Reactions involved:

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

$$2Mn(OH)_2 + O_2 \longrightarrow 2MnO(OH)_2 \text{ (Basic manganese oxide)}$$

$$2MnO(OH)_2 + H_2SO_4 \longrightarrow MnSO_4 + H_2O + [O]$$

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

$$I_2 + Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6 \text{ (sodium tetrathionate)}$$

$$\text{Starch} + I_2 \longrightarrow \text{Blue colour complex}$$

Interference of nitrite is overcome by adding NaN_3 & H_2SO_4

$$NaN_3 + H^+ \longrightarrow NH_3 + Na^+$$

$$NH_3 + NO_2 + H^+ \longrightarrow N_2 + 2H_2O$$

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Experiment 5.

Aim:

To determine the concentration of dissolved oxygen from the given water sample using standard N/40 $Na_2S_2O_3$ solution.

Reagents required:

Potassium iodide solution (10%), N/40 Hypo solution ($Na_2S_2O_3$), Starch sol^n (freshly prepared), manganese sulphate.

Apparatus required:

Burette, Pipette, Measuring flask, Glass rod, iodometric flask.

Theory:

The experiment is based on the oxidation of potassium iodide by 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 hydro-xide is produced in it by the action of potassium hydroxide & manganous sulphate.

Starch is used as an indicator in iodometric titration. Starch is a viable indicator in the titration process because it turns deep dark blue when iodine is present in a solution. When starch is heated in water, decom-position occurs and beta-amylose is produced. Beta-amylose with iodine, resulting in a dark blue colour change.

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Observations:

Volume of water for each titration = 50 ml

Volume of $\frac{N}{40}$ thiosulphate solution = 44.5 (V) ml

Calculations:

50 ml of water sample × V ml of N/40 $Na_2S_2O_3$ solution

$$\text{Normality} = \frac{V}{50 \times 40} = \frac{44.5}{50 \times 40} = 0.02225\,N$$

$$\text{Strength} = \frac{8V}{50 \times 40} = 0.178 \text{ gm of } O_2/\text{litre}$$

$$= 178 \text{ ppm}$$

Amount of dissolved oxygen = 178 ppm

Azide modification is used to remove substances like nitrites, sulphides etc. which liberate iodine from potassium iodide to dissolve oxygen.

Procedure:

(i) Took 50 ml water sample in iodometric flask; avoiding as far as possible, contact with air.
(ii) Immediately added 1 ml of manganese sulphate solution from the burette & added 1 ml of alkaline iodide from the other burette.
(iii) Inserted the stopper & shake several times.
(iv) Allow the ppt to settle half way & mixed again. Repeated this process of shaking & settling at least 3 times.
(v) Added 1 ml of conc. H_2SO_4. Inserted the stopper & shake again.
(vi) Allowed the yellow solution to stand for 5 mins.
(vii) Withdrew 50 ml of solution. Titrated it against standard N/40 $Na_2S_2O_3$ sol^n till the colour of the solution becomes faint yellow.
(viii) At that stage added few drops of starch solution. The colour turned to the deep blue due to formation of starch iodide.
(ix) Continued the titration till the solution becomes colourless.

Result:

Dissolved oxygen = 178 ppm.

Precautions:

(i) Sol^n in iodide flask should be shaken carefully keeping thumb on lid.
(ii) Sol^n should be well shaken before each aliquot is taken for titration.
(iii) Sol^n in iodide flask should not be sucked into pipette with mouth.

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