C H A P T E R- II

Experimental Techniques :

The oxidation of Anilines viz Aniline ($C_6H_5NH_2$),O. Chloro Aniline ($C_6H_4NH_2Cl$) and P-Chloro Aniline ($C_6H_4NH_2Cl$) was studied here. The potassium bromate was used as an oxidant The reaction or the oxidation was studied in strong acidic medium.

C H E M I C A L S :

Chemicals use were AR grade BDH.

The source of Br (V) was $KBrO_3$ (BDH, AR). Sodium Thiosulphate (BDH), All anilines (BDH, AR), Sulphuric Acid (A.R.) Na_2SO_4 ,K_2SO_4 and $MgSO_4$ (BDH AR.) Distilled water.

Standard solutions for various experiments were prepared as follows :

1. Preparation of Anilines :

All the anilines were first purified by the double distillation. Then these double distilled colourless anilines were used to prepare the solutions.

Aniline B.P = $130^{O}C$

O-Chloro Aniline B.P. = $208^{O}C$

P-Chloro Aniline M.P. = $70^{O}C$

The solution of Anilines was prepared in 2N sulphuric Acid.

The accurately, weighed amount of Anilines was dissolved in 2 N sulphuric Acid to prepare the desired concentration.

2. Potassium Bromate ($KBrO_3$):

To prepare (0.1 M) stock solution 16.7 gms. of potassium bromate (BDH) was accurately weighed on chemical balance and then dissolved in one litre distilled water. For the various experiements the desired amount of this stock solution was taken and was diluted to the required concentration.

3; Sodium Thiosulphate ($Na_2S_2O_3$)Solution :

To prepare (0.01M) this stock solution 2.480 gms of sodium thiosulphate (BDH)was dissolved in one litre distilled water. This stock solution was diluted to the required concentration and then used.

4 Sulphuric Acid (H_2SO_4)

The (1ON) sulphuric Acid (AR) was prepared by the standardization method. Then this stock solution was used for the various experiments by dilution to the desired concentration.

5. Potassium Iodide (KI) :

The 10% fresh solution of potassium iodide (BDH) was prepared for every experiment .

B) Indicator :- A fresh starch solution.

P r o c e d u r e

Two standard flasksof 150 ml are taken. In one flask 20 ml of potassium bromate and 20ml of sulphuric acid solution were taken. In the another flask 20 ml of Aniline solution and 20 ml of sulphuric Acid solution was taken, and these two flasks was kept in thermostat till the reaction mixture attains the temperature of thermostat . Then the solution from the second flask, containing aniline solution and H_2SO_4 solution was added in the first flask. After addition immediately 5 ml aliquot of this reaction mixture was pipetted out, in 10 ml of 10% potassium iodide solution which were already taken in iodometric flask. Then the liberated

iodine was titrated with 0.002 M sodium thiosulphate solution, using starch as indicator. The end point is blue to colourless. This is an initial reacting 'a'. Then after every Five minutes 5 ml aliquots of reaction mixture were titrated addopting above procedure. From these titration readings the rate constant (k) was calculated.

$$K = \frac{2.303}{t} \log_{10} \frac{a}{a-x}$$

Where a = Initial concentration of potassium bromate in terms of 0.002 M sodium Thiosulphate.

x = The amount of potassium bromate consumed in time interval (t) interms of sodium Thiosulphate.

t = Time in minutes.

The slopes and intercepts of the graphs were evaluated by least square method using PB100 personal computer. The program was prepared in basic language.

Ammonia was detected by adding Nessler's reagent in alkaline solution of reaction mixture which has given buff coloured precipitate.