

27/04/23

## COMPLEXOMETRY

### 1. Estimation of Total hardness of water by using Standard EDTA Solution

Aim: To estimate total hardness of water by using Ethylene Diamine Tetra-acetic Acid (EDTA) solution.

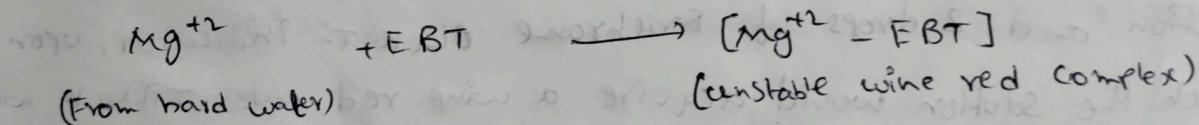
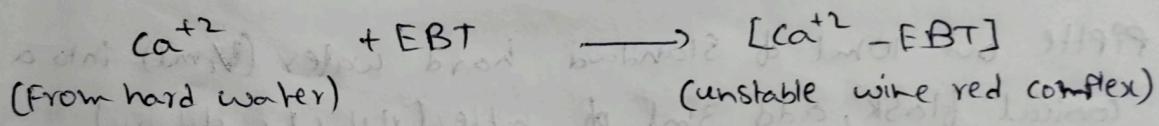
Apparatus: Pipette, burette, standard volumetric flask, conical flask, beaker, dropper.

Chemicals required: Disodium salt of EDTA, Eriochrome Black-T (EBT) indicator, Calcium carbonate, dil. HCl.

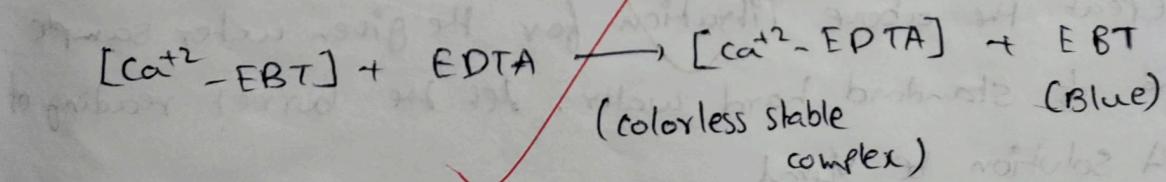
Indicator: Eriochrome Black-T (EBT)

End point: Change of color from wine red to blue.

Principle: This method is based on the following facts; Eriochrome Black-T (a blue coloured dye) when added to a simple hard water buffered to pH value of about 9-10, combine with  $\text{Ca}^{+2}$  and  $\text{Mg}^{+2}$  ions of hard water to form an unstable wine red complex.



EDTA solution when added to hard water abstracts calcium and magnesium ion from the above unstable complex and forms a colorless stable complex with regeneration of blue colored dye (EBT).



Thus, the amount of EDTA used corresponds to hardness of water. The end point of titration will be change of color from wine red to blue

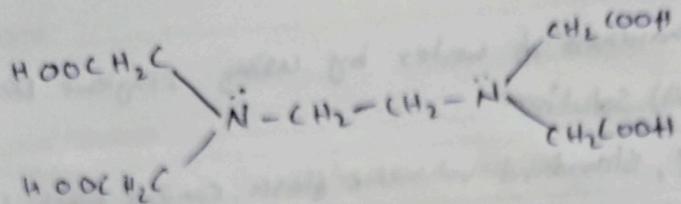


Fig. Structure of Ethylenediamine Tetra-acetic acid (EDTA)

Procedure: The experimental procedure involves the following procedure steps.

Step 1:- Preparation of standard Hard water:

mg of pure, dry  $\text{CaCO}_3$  is weighed and dissolved in minimum quantity of dil. HCl to get clear solution and the volume is made up to the mark with distilled water in a 100ml volumetric flask.

Step 2:- Standardization of disodium salt of EDTA

Pipette out 10ml of Standard hard water ( $V_1$  ml) into a clean conical flask, add 3ml of buffer ( $\text{NH}_4\text{Cl} + \text{NH}_4\text{OH}$ ) solution and 2 drops of Erichrome Black T indicator, upon which the solution would acquire a wine red color. Titrate with the solution of disodium salt of EDTA taken in the burette, till the wine red colour changes to blue which is the end point. Let the burette reading of EDTA be  $V_2$  ml

Step 3:- Determination of total hardness

Repeat the above titration for the given water sample instead of standard hard water. Let the burette reading of EDTA solution be  $V_3$  ml

Observation  
Step 1:-  
weight of  
Molarity  
molecular  
Step 2:- St

Soln.

1

2

3

by law

where M

Molarity

## Observation & calculations :-

Step 1:- Molarity of standard hard water

weight of  $\text{CaCO}_3$  = 0.1 gm

Molarity of  $\text{CaCO}_3$  = 0.01 M

$$M = \frac{g}{M.W} \times \frac{1000}{1000} = \frac{0.1}{100} \times 100 = 0.001 \text{ M}$$

Molecular weight of  $\text{CaCO}_3$  = 100

Step 2:- Standardization of disodium salt of EDTA :-

| S.No. | Vol. of Standard Hard water taken<br>$V_1$ (ml) | Burette reading (ml) |       | Vol. of EDTA solution consumed<br>$V_2$ (ml) |
|-------|---|----------------------|-------|--|
|       |   | Initial              | Final |  |
| 1     | 10  | 0                    | 11.6  | 11.6   |
| 2     | 10  | 11.6                 | 22.6  | $22.6 - 11.6 = 11.0$                         |
| 3     | 10  | 22.6                 | 33.6  | $33.6 - 22.6 = 11.0$                         |

By law of equivalence,  $\frac{M_1 V_1}{m_1} = \frac{M_2 V_2}{m_2}$  ( $m_1 = m_2 = n$ )

where  $M_1$  = Molarity of standard hard water = 0.01 M

$V_1$  = vol. of standard hard water in conical flask = 10 ml

$M_2$  = Molarity of EDTA =

$V_2$  = Volume of EDTA consumed = 11.0 ml

$$M_2 = \frac{M_1 V_1}{V_2} = \frac{0.01 \times 10}{11} = \frac{0.1}{11} = 0.009 \text{ M}$$

Molarity of disodium salt of EDTA solution = 0.009 M.

Step 3:-

| S.No. | vol. of water sample taken, $V_3$ (ml) | Burette reading |       | vol. of EDTA Soln consumed $V_4$ (ml) |
|-------|--|-----------------|-------|---------------------------------------|
|       |  | Initial         | Final |                                       |
| 1.    | 10                                     | 0               | 4.3   | $4.3 - 0 = 4.3$                       |
| 2.    | 10                                     | 4.3             | 8.6   | $8.6 - 4.3 = 4.3$                     |
| 3.    | 10                                     | 8.6             | 11.9  | $11.9 - 8.6 = 4.3$                    |

by the law of equivalence  $\frac{M_3 V_3}{n_3} = \frac{M_4 V_4}{n_4}$  ( $n_3 = n_4 = n$ )

where  $M_4 = M_2 = \text{molarity of EDTA} = 0.009 \text{ M}$

$V_4 = \text{volume of EDTA consumed} = 4.3 \text{ ml}$

$M_3 = \text{molarity of water sample} = M_3$

$V_3 = \text{volume of water sample taken} = 10 \text{ ml}$

$$M_3 = \frac{M_4 V_4}{V_3} = \frac{M_2 V_4}{V_3} = \frac{0.009 \times 4.3}{10} = 0.00387 \text{ M}$$

Molarity of given water sample =  $0.00387 \text{ M}$

Total hardness =  $M_3 \times \text{molecular weight of } \text{CaCO}_3 (100) \times 1000$

~~=  $(\text{CaCO}_3 (100)) \times 1000$~~

$$= 0.00387 \times 100 \times 1000 = 387 \text{ ppm}$$

Total hardness of water is 387 ppm

G  
27/09/22

2. Estimate  
Aim:- To  
in  
me

Apparatus  
chemicals

Indicate

End point

Theory

in which  
and the  
oxidatio  
react i  
equati

Adding

2KMnO<sub>4</sub>

Acco

Perm

exp@

color

the

acti

4/10/23

## PERMANOANALYSIS

### 2. Estimation of Iron(II) Using Potassium Permanganate

Aim: To estimate the amount of Ferrous iron present in the given iron sample solution by permanganometric method.

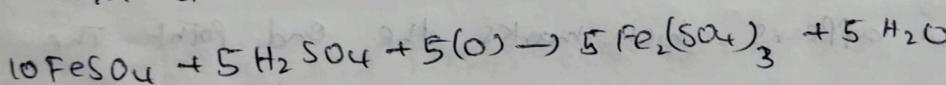
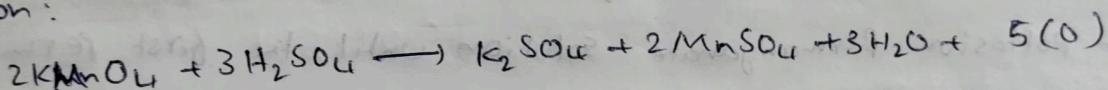
Apparatus: Burette, Pipette, conical flask, beaker.

Chemicals: Potassium Permanganate, Ferrous ammonium Sulphate hexahydrate,  $H_2SO_4$

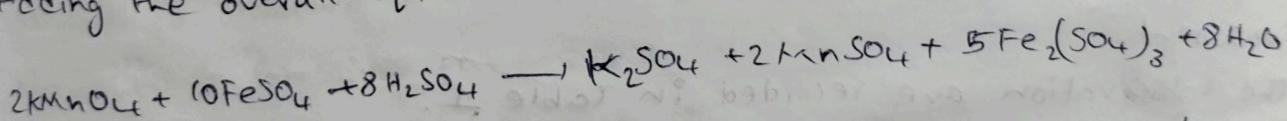
Indicator: Potassium Permanganate acts as a self-indicator

End point: colorless to pale pink

Theory: Potassium Permanganate is a powerful oxidizing agent in which manganese is present in its +7 oxidation state and Mohr's salt ( $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ ) contains iron in its +2 oxidation state which acts as a reducing agent. These two react in dilute sulphuric acid medium according to the partial equation:



$\times 1000$  Adding the overall eq. is



According to this overall balanced equation, one mole of permanganate reacts with five moles  $Fe^{+2}$ . In this reaction, except permanganate, all other reactants and products are colourless. When all the  $Fe^{+2}$  ions are completely reacted, the excess drop of permanganate imparts pink color to the solution which marks the end point of titration. i.e., permanganate acts as self-indicator in this titration.

Procedure: The experiment involves these steps

Step 1: Preparation of standard Mohr's salt solution:

About 0.8g of Mohr's salt is carefully weighed and transferred into a 100ml volumetric flask through the funnel, dissolve the salt in small amount of distilled water and add 2ml conc. sulphuric acid and the solution is made up to the mark with distilled water.

Step 2: Standardization of potassium permanganate solution with standard ferrous ammonium sulphate solution:

The burette is rinsed with the permanganate solution and filled with the same solution up to the mark. The initial reading of burette is noted.

1ml of the Mohr's salt solution is pipetted out carefully into a clean 250ml of conical flask, followed by addition 5ml of 1:1 sulphuric acid by using a measuring jar and titrate with potassium permanganate solution until the first permanent pale pink colour appears which marks the end point. The titration is repeated until two successive titre values are concurrent.

The observations are recorded in Table 1.

Step 3: Titration of Iron(II) sample with standard  $K_2Cr_2O_7$ :

1ml of the Mohr's salt solution of unknown concentration is pipetted out carefully into a clean 250ml conical flask, followed by 5ml of 1:1 sulphuric acid by using a measuring jar. After gently swirling the contents of the conical flask to homogenize the solution, it is titrated with the permanganate solution drop-wise until a drop in excess infests a permanent pale pink color, which marks the end point. The titration is repeated

until two successive concurrent litre values. The observation are recorded in Table 2.

### Observation and Calculation:

Step 1: Normality of standard Ferrous ammonium sulphate

weight of the salt = 8.03 gms

$$\text{Normality (N)} = \frac{\text{Weight of the salt}}{\text{Equivalent weight of Mohr salt}} \times \frac{1000}{100} = \frac{0.8 \times 1000}{392.13} = \frac{8}{392.13}$$

Equivalent weight of Mohr's salt is 392.13  $\therefore N = 0.0204 N$

Table 1: Standardization of Potassium permanganate solution with standard Mohr's salt solution

| S.No. | Volume of Standard<br>Mohr's Salt solution<br>taken, $V_1$ (ml) | Burette reading (ml) |       | vol. of Pot.<br>permanganate<br>solution run down<br>$V_2$ (ml) |
|-------|---|----------------------|-------|---|
|       |   | Initial              | Final |   |
| 1.    | 10 ml   | 0.2 ml               | 8 ml  | 8 ml  |
| 2.    | 10 ml   | 8 ml                 | 16 ml | $16 - 8 = 8$ ml   |
| 3.    | 10 ml   | 8 ml                 | 16 ml | $16 - 8 = 8$ ml   |

Normality of standard Mohr's salt solution taken =  $N_1 = 0.0204 N$

volume of standard Mohr's Salt solution taken =  $V_1 = 10 \text{ ml}$

Normality of potassium permanganate solution consumed =  $N_2 = ?$

Volume of potassium permanganate solution consumed =  $V_2 = 8 \text{ ml}$

By the law of equivalence:  $N_1 V_1 = N_2 V_2$  (or)

$$N_2 = \frac{N_1 V_1}{V_2} = \frac{0.02 \times 10}{8} = \frac{0.1}{4} = \frac{1}{4} \times 10^{-1} = 0.25 \times 10^{-1} = 0.025 N$$

Normality of potassium permanganate solution ( $N_2$ ) =  $0.025 N$

Table 2: Titration of Unknown Iron(II) Sample solution with Standard Potassium permanganate solution

| S.No. | volume of Iron(II) solution taken: $V_3$ (ml) | Burette reading (ml) |         | volume of Pt. permanganate solution run down, $V_4$ (ml) |
|-------|---|----------------------|---------|--|
|       |   | Initial              | final   |  |
| 1)    | 10 ml   | 16 ml                | 20.8 ml | 4.8 ml   |
| 2)    | 10 ml   | 20.8                 | 25.3 ml | 4.5 ml   |
| 3)    | 10 ml   | 25.3 ml              | 29.8 ml | 4.5 ml   |

Normality of Iron(II) solution taken =  $N_3 = ?$

volume of Iron(II) solution taken =  $V_3 = 10 \text{ ml}$

Normality of Potassium permanganate solution consumed =  $N_4 = ?$   
 $= 0.025 \text{ N}$

volume of potassium permanganate solution consumed =  $V_4 = 4.5 \text{ ml}$

By the law of equivalence:  $N_3 V_3 = N_4 V_4$

$$(i) N_3 = \frac{N_4 V_4}{V_3} = \frac{0.025 \times 4.5}{10} = \frac{25 \times 4.5 \times 10^{-4}}{10} = 1125 \times 10^{-5} \\ \approx 0.01125 \text{ N}$$

Normality of Iron (II) solution =  $N_3 = 0.01125 \text{ N}$

Amount of ferrous iron present in the given iron sample:-

$$\text{Equivalent weight of ferrous Iron} = \frac{\text{Atomic weight}}{1} \\ = \frac{55.85}{1} = 55.85$$

Amount of  $\text{Fe}^{+2}$  present in 100 ml of iron solution (X) g =  $N_3 \times \text{Eq. wt}$   
 $\text{of } \text{Fe}^{+2} (55.85)$

$$0.01125 \times 55.85 \\ = 0.62831 \text{ g}$$

Amount of  $\text{Fe}^{+2}$  present in the given 10 ml of an Iron

$$\text{Solution} = X/10 = \frac{0.62831}{10} = 0.062831 \text{ g}$$

Result: ~~4/10/23~~

Amount of  $\text{Fe}^{+2}$  present in the given 100 ml of Iron solution = 0.062831 g

4/10/23  
3. Determination

Aim: Determ.

Mohr's

Apparatus: Con-

chemicals: Sta-

Indicator: P-

End point:-

Theory: Th-

The presence

determination

Ranges 7-8

indicator

Silver nit-

precipitated

results in

precipitated

method, H-

formation

slightly

and in the

(in, v.)

Procedure

Estimati

1. Wash

solution with

volume of Pt.  
permanganate solution  
run down, until the

4.8 ml

4.5 ml

4.5 ml

$$\text{summed} = N_4 = N_2 = 0.025 \text{ N}$$

$$V = V_4 = 4.5 \text{ ml}$$

$$0.01125 \times 10^{-5}$$

$$0.01125 \text{ M}$$

iron sample:

eight

$$n(X)g = N_3 \times \text{Eq. wt of Fe}^{+2} (55.85)$$

$$0.01125 \times 55.85$$

$$6.2831 \text{ g}$$

of an Iron

$$\text{solution} = 0.062831 \text{ g}$$

01/12/23 Argentometry

### 3. Determination of chloride content of water

Aim: Determination of chloride content of water by Mohr's method or (Argentometric titration).

Apparatus: conical flask, burette, pipette, volumetric flask, beaker.

Chemicals: Standard Silver nitrate, Potassium chromate

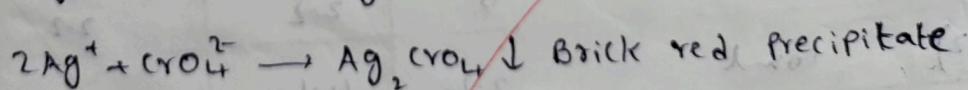
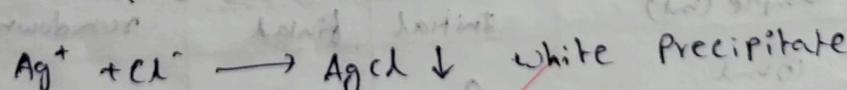
Indicator: Potassium chromate

End point: light yellow to brick red colour

Theory: The source of chloride in natural water is due to

the presence of NaCl, KCl, MgCl<sub>2</sub> and CaCl<sub>2</sub>. This method for determination of chloride in water is performed in the pH ranges 7-8. The Mohr method uses chromate ions as an indicator in the titration of chloride ions with a standard silver nitrate solution. After all the chloride has been precipitated as white silver chloride, the first excess of titrant results in the formation of brick red silver chromate

precipitate, which signals the end point. In the Mohr's method, the determination of endpoint is based on the formation of a second precipitate should have solubility slightly greater than the precipitate between the analyte and the titrant (silver chloride precipitate).



Procedure:

Estimation of chloride by using stand silver nitrate solution

1. wash the burette with distilled water and rinse with

- Standardised solution of silver nitrate ( $\text{AgNO}_3$ ) and  
the fill the burette with the same.
2. Pipette out 25 ml of given water sample and transfer into a conical flask. Add 1 ml of the potassium chromate indicator solution (advisable to use a pipette for this purpose).
  3. Titrate the resultant solution with silver nitrate solution, and ensure that the flask is constantly swirled.
  4. As the titration proceeds slowly a red colour is formed and disappear slowly after addition of each drop of silver nitrate.
  5. At this stage slow down the rate of addition of silver nitrate. The end point is indicated by sudden development of a faint but distinct change in colour to reddish-brown indicates the precipitation of silver chromate which does not disappear on swirling the conical flask.
  6. Repeat titration for concurrent readings.

#### Observation and Calculation:

Estimation of chloride by using standard silver nitrate solution.

Table:-

#### Sample Drinking Water

| S.No. | Vol. of water sample (ml) | Burette Reading |       | Vol. of silver nitrate rundown (ml) |
|-------|---------------------------|-----------------|-------|-------------------------------------|
|       |                           | Initial         | Final |                                     |
| 1)    | 10 ml                     | 0               | 2.2   | 2.2                                 |
| 2)    | 10 ml                     | 2.2             | 4.4   | 2.2                                 |

Normality  
vol. of S  
vol. of  
Amount  
Normal

Result:- The

Q/11/

KATI MEDIYA

Normality of Standard silver nitrate solution ( $N_1$ ) = ~~0.02~~  $\times 10^3$  N

vol. of Silver nitrate ( $V_1$ ) = ~~2.2 ml~~

vol. of water ( $V_2$ ) = 10 ml

Amount of chloride ions in mg/litre =

$$\frac{\text{Normality of } \text{AgNO}_3 \times \text{volume of } \text{AgNO}_3 \times \text{equivalent weight of Cl}}{\text{volume of water sample}} \times 10^3$$

$$= \frac{0.02 \times 2.2 \times 35.5}{10} \times 10^3$$

$$= 78.1 \times 10^3 \times 10^{-3} \text{ ppm} = 78.1 \text{ ppm}$$

Result :- The Amount of chloride in water sample is

78.1 ppm (or) mg/L

Q/11/23

22/11/23

## Conductometry

### Conductometric Titration of Strong Acid with Strong Base

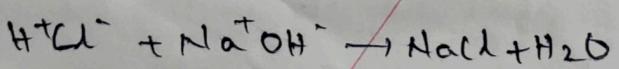
Aim: To determine strength HCl solution by titrating it against standard NaOH solution conductometrically.

Apparatus: Conductivity meter, burette, beaker.

Chemicals: HCl, NaOH

#### Principle:

Conductometric titration is based upon measurement of the conductance during course of titration. The number of free ions and mobility of the ions affect the conductivity of an aqueous solution. When one electrolyte is added to another electrolyte, the change in number of free ions causes a change in the conductance. For example, when a strong acid (HCl) is titrated against a strong base (NaOH), the acid solution has high conductivity due to highly mobile  $H^+$  ions just before NaOH solution is added from the burette. The conductivity of acid solution decreases while adding the NaOH, due to neutralization of highly mobile  $H^+$  ions of the acid with  $OH^-$  ions of the base.



Thus, the conductance of the solution continues to decrease until the equilibrium point is reached.

Further addition of NaOH solution will increase the conductance of solution depends upon the no. of ions and

their mobilities  
drawing a graph  
depends upon  
extrapolation  
gives the end  
for neutralization  
Procedure:  
measurement

Pipette out 4  
beaker. Dip the  
rinsing the  
and HCl solution  
solution. Add  
burette to HCl  
conductivity of  
solution. Repeat

NaOH Solution  
reading of

and note the  
Observation &

Observed: co  
Table-1. A g  
(Y-axis) and  
Point (Neutrali  
graph.

their mobilities. Two straight lines are obtained on drawing a graph between conductance ~~of solution~~ and volume of NaOH. On extrapolation, these lines intersect at a point that gives the end point and volume of titrant required for neutralization.

### Procedure:

#### Measurement of the conductivity of the solution:

Pipette out 40 ml of the HCl solution into a 100 ml beaker. Dip the conductivity cell in HCl solution after rinsing the conductivity cell with distilled water and HCl solution. Note the conductance value of HCl solution. Add 1 ml of NaOH solution taken from the burette to HCl solution and stir well. Note the conductance of the solution after the addition of NaOH solution. Repeat the procedure by addition of 1 ml NaOH solution every time and the conductivity reading of the remaining solution. Take 15-20 readings and note the readings in the given table.

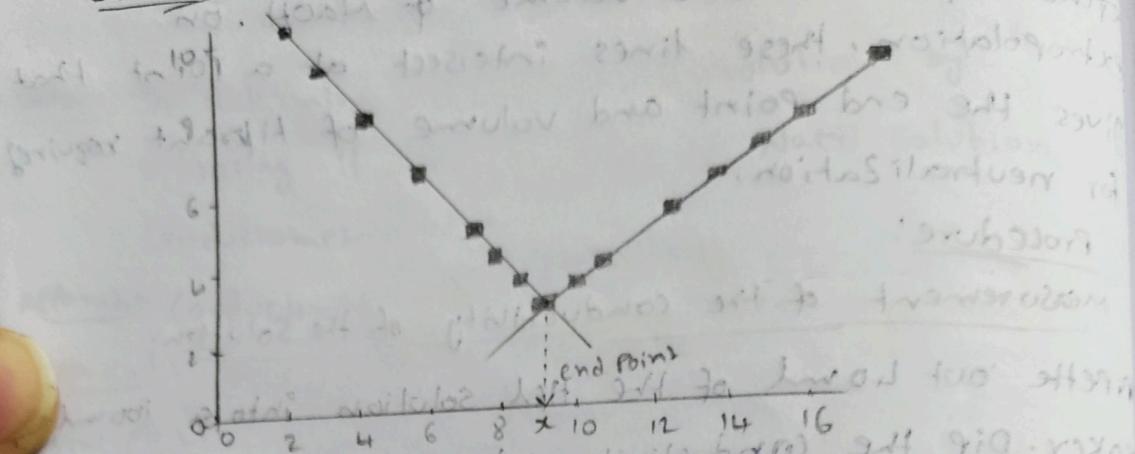
#### Observation & calculation:

Observed conductance readings are noted from the Table-1. A graph is plotted b/w observed conductance (Y-axis) and volume of NaOH added (X-axis). Equivalence point (neutralization point) is noted (down) from the graph.

Table 1

Fig. Graph of Conductance vs Volume of NaOH

Solution



Operating Instructions:

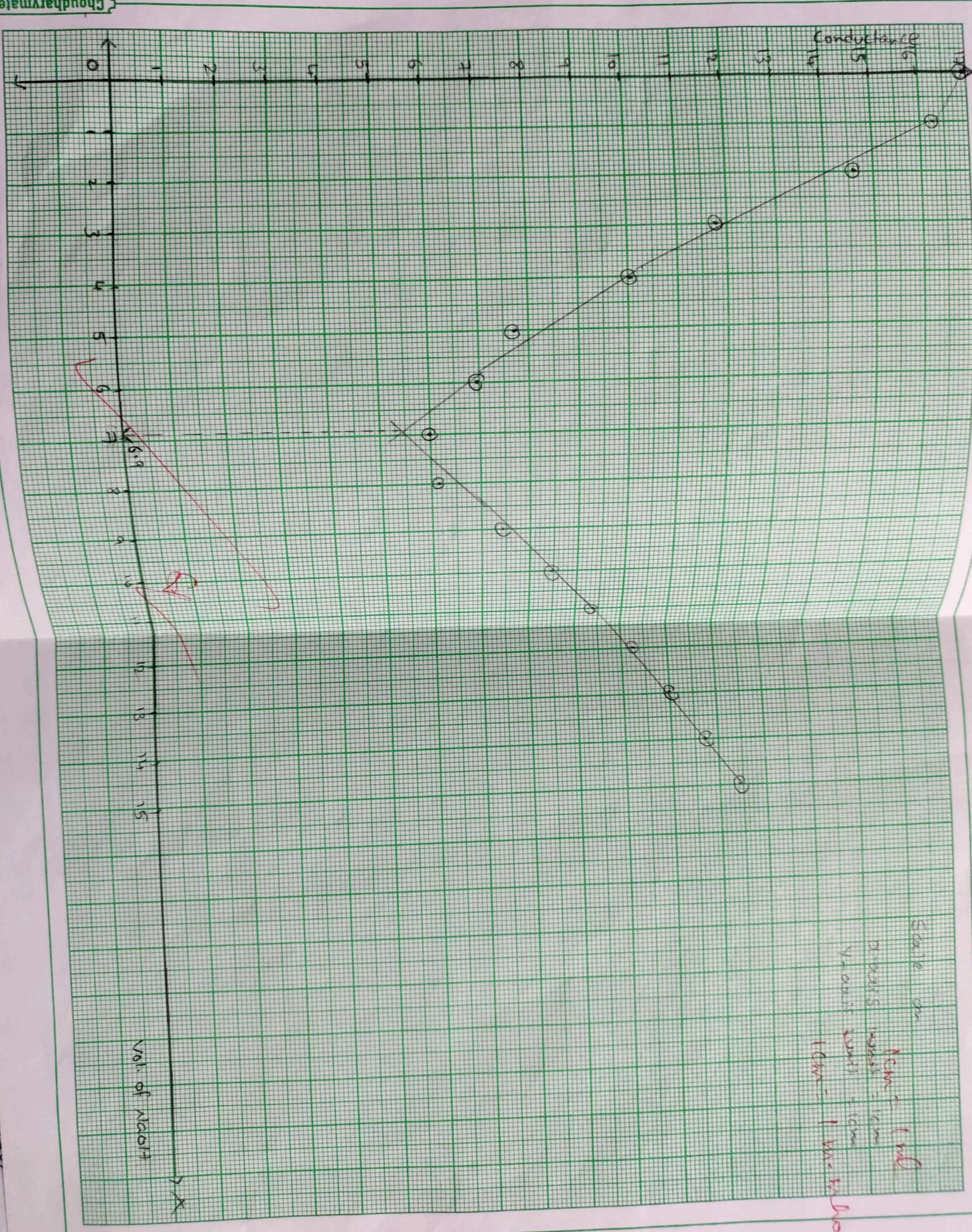
- Digital Conductivity meter:
  1. Switch on the instrument and allow it to warm up for a minute or two.
  2. Keep check/read switch at check position and selectors switch in 10K range and calibrate to 10.00.
  3. Dip the conductivity cell in HCl solution.
  4. Bring the switch to read position and note down HCl titration values.
- EI-611 Conductivity Meter:
  1. Switch on the instrument and allow it to warm up for a minute or two.
  2. Set the temperature component to room temperature (25/30°C).
  3. Select the range to 200mS.
  4. Keep the conductivity cell in the distilled water and the function knob in the check position and adjust the cell constant value to 1.000.
  5. Keep the conductivity cell in the test solution (HCl solution) and note the reading on the display.

The s  
determin  
Volume  
Norma  
Volum  
Norma  
Amount

# Conductometry

C.R.No. 23244 A12A7

P.S.N.V.S. Nikhil



Digital  
1. Switch  
for a  
2. keep  
selectors  
3. Dip H  
4. Bring  
methanol  
EI - 611  
5. Switch  
6. Add  
set pH  
(25/30°C)  
Select  
keep  
and the  
just  
the  
keep -

in the given solution

Table 1 Determination of the strength of the given HCl.

| S.No. | Volume of NaOH (ml) | observe conductance (Millimhos) |
|-------|---------------------|---------------------------------|
| 1     | 0                   | 17.75                           |
| 2     | 1                   | 16.38                           |
| 3     | 2                   | 14.51                           |
| 4     | 3                   | 11.95                           |
| 5     | 4                   | 10.03                           |
| 6     | 5                   | 7.75                            |
| 7     | 6                   | 7.0                             |
| 8     | 7                   | 6.0                             |
| 9     | 8                   | 6.1                             |
| 10    | 9                   | 6.21                            |
| 11    | 10                  | 7.66                            |
| 12    | 11                  | 9.45                            |
| 13    | 12                  | 10.31                           |
| 14    | 13                  | 11.09                           |
| 15    | 14                  | 11.7                            |
| 16    | 15                  | 12.42                           |

The strength of the given acid (HCl) solution is determined in the following way

Volume of NaOH solution ( $V_1$ ) : 6.9 ml

Normality of NaOH solution ( $N_1$ ) : 0.2 N

Volume of HCl solution ( $V_2$ ) : 40 ml

Normality of HCl ( $N_2$ ) :  $\frac{V_1 \times N_1}{V_2} = \frac{6.9 \times 0.2}{40} = 0.0345 \text{ N}$

Amount of HCl in the given solution :  $N_2 \times \text{Eq.wt of HCl}$

67/12/23

## 5. Esti

Aim:- To es  
given

### Apparatus:-

### chemicals:-

### Principle:-

an indica  
of PH of  
of consta  
will be r

Esti  
chromate  
reduct  
yes du  
. From  
given

### oxidis

SSium  
ivm , if  
vol -

$$= 0.0345 \times 36.55 \text{ g}$$

$$= 1.26 \text{ g}$$

### Result:-

By this experiment "Conductometric titration of strong Acid with Strong base" is hence done.

Amount of HCl present in  
the given Solution = 1.26 g

8  
67/12/23

6/12/23

## Potentiometry

### 5. Estimation of Fe(II) using Potentiometer

Aim: To estimate the amount of Fe(II) present in the given solution Potentiometer.

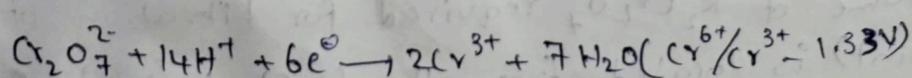
Apparatus: Pipette, burette, beakers, calomel and platinum electrodes, glass rod.

chemicals: Standard Potassium dichromate, ferrous electrodes, ammonium sulphate and  $H_2SO_4$

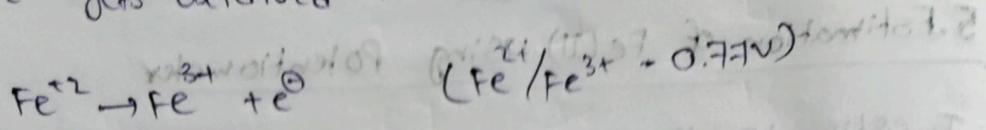
Principle: Potentiometric titration involves the measurement of Potential between two electrodes, one is an indicator electrode whose potential changes with that of PH of the solution and the other a reference electrode of constant potential. The equivalence point of the reaction will be revealed by sudden change in the potential.

Estimation of ferrous iron by using potassium dichromate is a redox titration which involves oxidation and reduction. During their titration, the solution potential changes due to the change in the conc. of oxidised/reduced form. From the Nernst equation, we know that the potential of a given reaction will depend on the relative concentration of oxidised/reduced form.

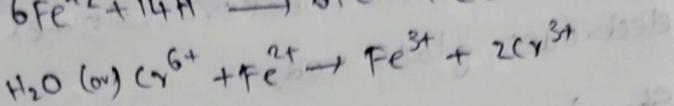
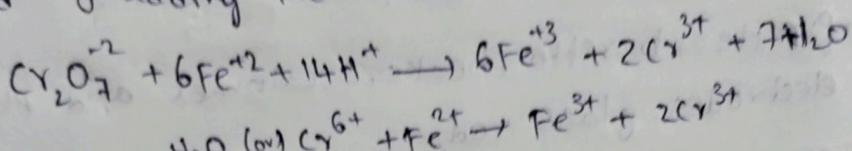
Potassium dichromate is an oxidising agent and in acid medium, it follows the half reaction to give Cr(III) as the reduction product.



while  $\text{Fe}^{+2}$  gets oxidised to  $\text{Fe}^{+3}$  as per the reaction



The overall ionic equation of this titration can be obtained by adding the above two:



The EMF of cell is given by  $E = E_{\text{cathode}} - E_{\text{anode}}$

cell representation:  $\text{SCE} | \text{Fe}^{+2} || \text{Cr}^{+6} | \text{Cr}^{+3} | \text{Pt}$

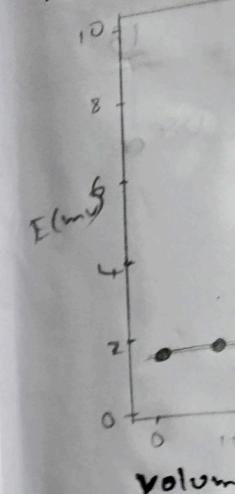
procedure:

1. Pilot Titration: Take 40ml of ammonium ferrous sulphate in a 100ml beaker, add 10ml of dilute Sulphuric acid and 5ml of distilled water. SCE is used as a reference electrode and platinum foil which acts as an indicator electrode are dipped in  $\text{Fe}^{+2}$  solution. Carry out necessary connections. Add 1 ml of 0.04N  $\text{K}_2\text{Cr}_2\text{O}_7$  from the burette and stir the solution, wait for a minute and note the EMF. After every addition of 1ml titrant, stir the solution and note the potential reading. An abrupt change in the potential gives a rough estimate of equivalence point.

Graphs:

1. Potentiometric titration curve (S-shaped curve) is obtained by plotting a graph between volume of  $\text{K}_2\text{Cr}_2\text{O}_7(\text{V})$  and potential ( $E$ )
2. The volume of  $\text{K}_2\text{Cr}_2\text{O}_7$  required for titration of  $\text{Fe}^{+2}$  (end point) is located from the first derivative curve

(v and  
Model g)

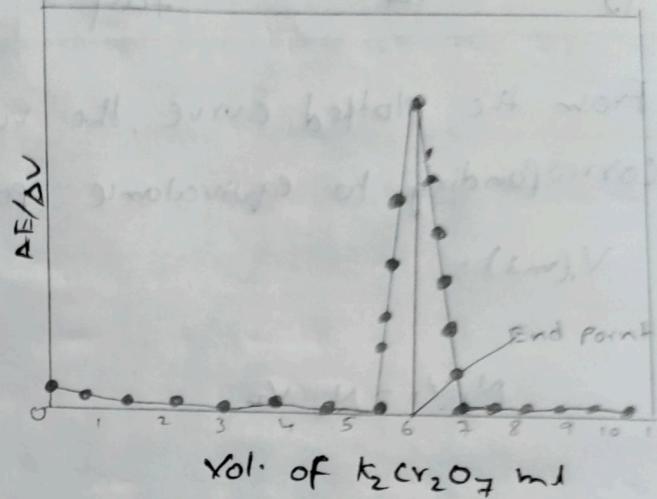
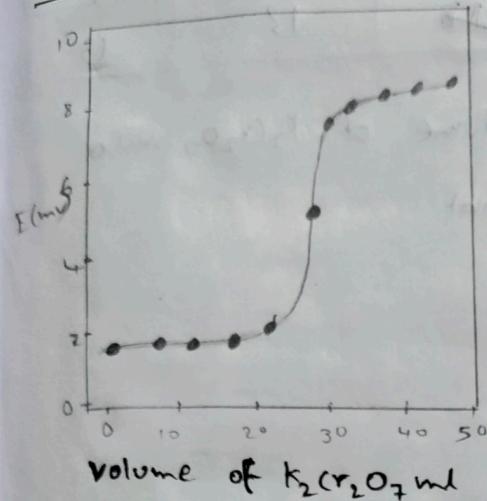


observation

1. Pilot Tit

| S.N.O. | Vol.<br>$\text{K}_2\text{Cr}_2\text{O}_7$ |
|--------|---|
| 1.     |   |
| 2.     |   |
| 3.     |   |
| 4.     |   |
| 5.     |   |
| 6.     |   |
| 7.     |   |
| 8.     |   |
| 9.     |   |
| 10.    |   |
| 11.    |   |
| 12.    |   |

(v and  $\Delta E/\Delta V$ ).  
Model graphs:



### Observation and Calculations:

#### 1. Plot Titration:

| S.No. | Vol. of $K_2Cr_2O_7$ ml | Potential (E) (mV) | $\Delta V$ | $\Delta E$ | $\Delta E/\Delta V$ |
|-------|-------------------------|--------------------|------------|------------|---------------------|
| 1.    | 0                       | 230                | -          | -          | -                   |
| 2.    | 1                       | 288                | 1          | 58         | 58                  |
| 3.    | 2                       | 297                | 1          | 9          | 9                   |
| 4.    | 3                       | 320                | 1          | 23         | 23                  |
| 5.    | 4                       | 339                | 1          | 19         | 19                  |
| 6.    | 5                       | 339.5              | 1          | 13         | 13                  |
| 7.    | 6                       | 368                | 1          | 16         | 16                  |
| 8.    | 7                       | 389                | 1          | 21         | 21                  |
| 9.    | 8                       | 418                | 1          | 29         | 29                  |
| 10.   | 9                       | 569.3              | 1          | 27.5       | 27.5                |
| 11.   | 10                      | 715                | 1          | 22         | 22                  |
| 12.   | 11                      | 723                | 1          | 8          | 8                   |

|     |    |     |   |   |   |
|-----|----|-----|---|---|---|
| 13  | 12 | 732 | 1 | 9 | 9 |
| 14  | 13 | 736 | 1 | 4 | 4 |
| 15. | 14 | 749 | 1 | 3 | 3 |

From the plotted curve, the volume of  $K_2Cr_2O_7$  used corresponding to equivalence point

$$V_1(\text{ml}) =$$

$$N_1 V_1 = N_2 V_2$$

$$\text{Normality of } K_2Cr_2O_7 (N_1) = 0.4 \text{ N}$$

$$\text{Volume of } K_2Cr_2O_7 (\text{V}_1) \text{ ml}$$

$$\text{Volume of Fe(II) solution (V}_2\text{) = 40 ml}$$

$$\text{Normality of Fe(II) solution (N}_2\text{) = 0.09 N}$$

$$\text{Normality of Fe(II) solution (N}_2\text{) = } \frac{N_1 \times V_1}{V_2} = \frac{0.4 \times 40}{40} = 0.09 \text{ N}$$

Amount of ferrous ions present in the given iron sample:

$$\text{Equivalent weight of ferrous iron: Atomic weight / 2 = } \frac{55.85}{2} = 27.9 \text{ g/mol}$$

$$x = N_2 \times \text{Eqwt of Fe}^{+2} (27.9) = 0.09 \times 27.9 = 2.5026 \text{ g}$$

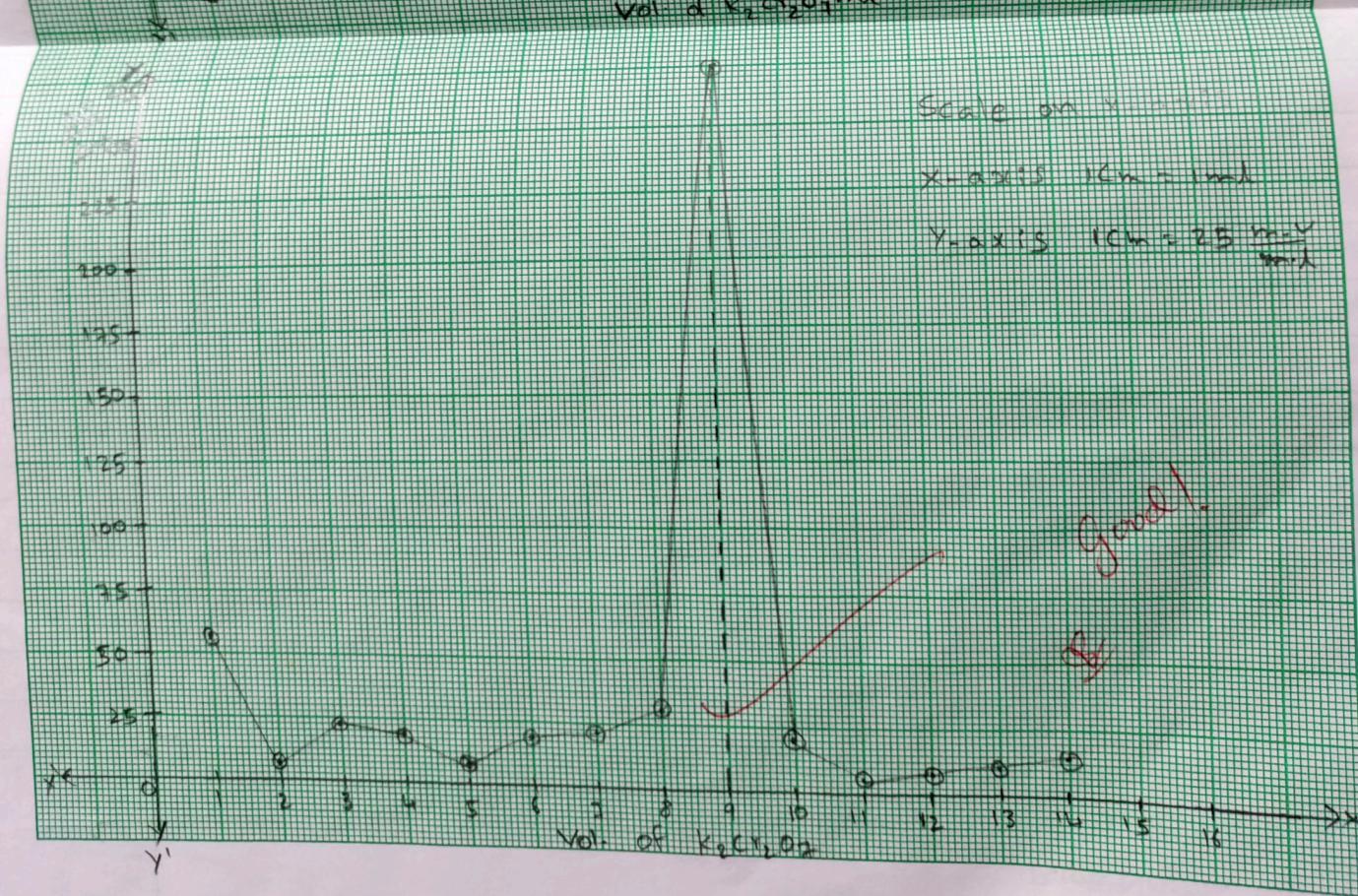
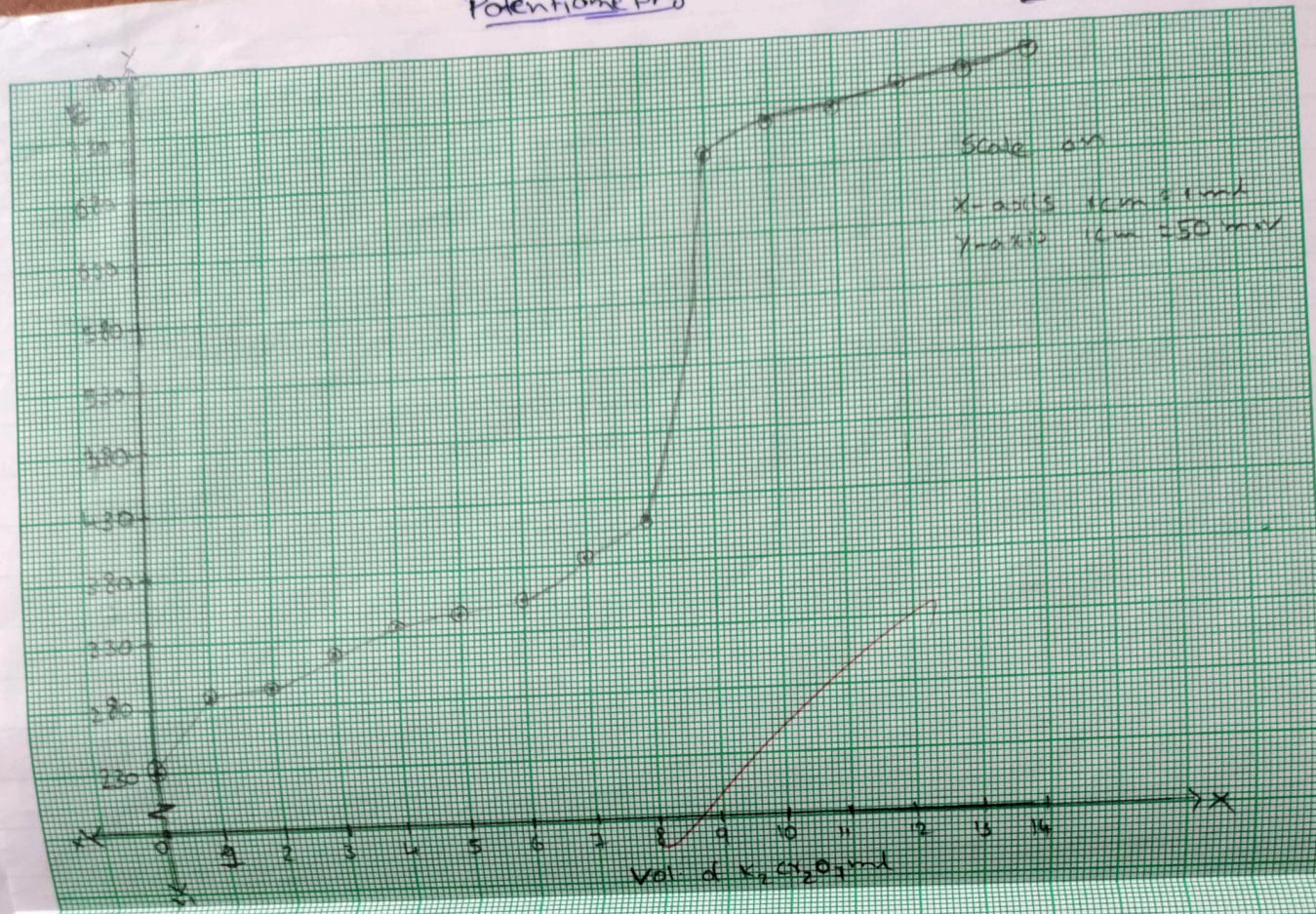
$$\text{Amount of Fe}^{+2} \text{ present in the given 100 ml of an iron ore solution: } x/10 = \frac{2.5026}{10} = 0.25026 \text{ g}$$

Result: Amount of  $Fe^{+2}$  present in the given 100 ml of an iron sample solution is 0.25026 g

OB/12/23

Potentiometry

P.S.N.V.S. Nikhil  
23241A12A7



From the corresponding values of  $V_1$  (ml)

Normality =  $\frac{\text{Volume}}{\text{Normality}}$

Normality =  $\frac{\text{Volume}}{\text{Normality}}$

Amount of sample =  $\frac{\text{Normality}}{\text{Normality}}$

Equivalent on one round or on one

soft: 1

Iron Sample Solution is 0.5026 g

18/12/23

## VISCOMETRY

q. Determination of viscosity of given liquid by Ostwald viscometer

Aim: To determine the viscosity of given liquid by using ostwald viscometer.

Apparatus: ostwald viscometer, stop watch, Pipette

chemicals: distilled water, unknown liquids.

Theory: when a liquid flows, it has an internal resistance or an internal friction to flow. viscosity is the property of the fluid which offers resistance to its own flow. Honey flows less readily than water because honey has higher viscosity than water.

In the lab for comparing the viscosities of diff. liquids, the commonly used instrument is ostwald's viscometer. In this operation, different liquids are taken exactly in same volume. ostwald's viscometer has two bulbs, one at upper end (A) and other at lower end (B). The liquids are sucked up in the upper bulb A initially, enough liquid volume is taken in bulb B such that when the liquid stands above for each liquid for its flow from X to Y. The driving pressure ( $P$ ) at all stages of flow in the liquid is given by ' $hdg$ ', where  $h$  is difference in heights of liquid in upper and lower ends and  $d$  is density of liquid and  $g$  is acceleration due to gravity.

- viscosity is procedure:
1. Take a capillary and make it vertical.
  2. Introduce the bulb of the viscometer.
  3. Suck the rubber bulb above the capillary and allow the liquid to flow from the capillary.
  4. Repeat the steps for unknown liquid.
  5. Calculate the viscosity.

due to gravity.

viscosity is measured by using the formula  $\frac{\eta_1}{\eta_{\text{water}}} = \frac{t \times d_1}{t_{\text{water}} \times d_1}$

procedures

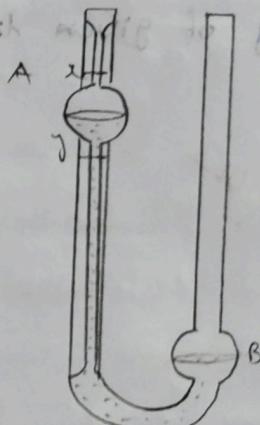
1. Take a clean and dry Ostwald viscometer and set it vertically on a stand.
2. Introduce 10 ml of distilled water into the larger bulb A.
3. Suck the liquid up into the bulb A through a rubber tubing attached to the end of the delivery tube above the mark 'X'.
4. Allow the liquid to flow freely through the capillary and note the time for the liquid to flow from mark (X) to (Y).
5. Repeat the procedure 2 to 3 times and get an avg value of t<sub>w</sub>.
6. Dry the viscometer in an oven and then repeat steps 2 and 5 to obtain avg time for the unknown liquid to flow from mark X to Y.

$$\frac{\eta_1}{\eta_{\text{water}}} = \frac{t \times d_1}{t_{\text{water}} \times d_1}$$

$\eta$  = viscosity

t = time to flow

d = density



| S.No. | wt | Liquid         | Time to flow in seconds |                |                | Time (Avg) | Viscosity (millipoise) |
|-------|----|----------------|-------------------------|----------------|----------------|------------|------------------------|
|       |    |                | t <sub>1</sub>          | t <sub>2</sub> | t <sub>3</sub> |            |                        |
| 1.    |    | water          | 33                      | 34             | 34             | 34         | 0.7975                 |
| 2.    |    | unknown liquid | 46                      | 46             | 47             | 46         |                        |

ded. vspinal off  $\frac{n_1}{n_w} = \frac{t_w d_1}{t_1 d_w}$  for liquid subscript

$n_1$  = viscosity of given liquid = ? in millipoise off 46 sec

$n_w$  = viscosity of distilled water = 0.7975 millipoise addit

$d_1$  = density of liquid = 0.786 g/cc from off 46 sec

$d_w$  = density of distilled water = 1g/cc

$t_{1f}$  = avg time of flow of liquid = 46 sec no prob

$t_{wf}$  = avg time of flow of distilled water = 34 sec off

$n_1 = \frac{t_w d_1}{t_1 d_w}$  result of substitution off 46 sec off wt to supv

$n_1 = \frac{46 \times 0.786}{34 \times 1} \times 0.7975 = 0.847$  millipoise

$n_1 = 0.847$  millipoise

result

viscosity of given liquid = 0.847 millipoise

S  
12/12/2023

## SURFACE TENSION

10. Determination of surface tension of a lubricant by using stalagmometer (principle)

Aim: To determine the surface tension of given liquid by using a stalagmometer. It is based on zero

Apparatus: Stalagmometer, beaker etc.

Chemicals: Known liquids, distilled water, oil etc.

Theory: Surface tension is defined as the force in dynes acting on a surface at right angles to any line of unit length or per centimeter. There are two methods of determining surface tension

1. Drop weight method and 2. Drop number

The liquids with large intermolecular forces would have higher surface tension ( $72.8 \text{ dyne/cm}$  or  $0.0728 \text{ Newton/m}$ ).

Surface tension of a liquid is measured by using an apparatus called stalagmometer.

Principle:

The size of the drop falling down at the end of the tube depends on surface tension of the liquid and size of capillary tube. Thus, when a liquid is allowed to flow through a capillary tube, a drop will increase in size to a certain extent and then fall off.

→ The total surface tension of supporting the drop is

$2\pi r \gamma$ , where  $r$  = radius of outer circumference of dropping end of capillary tube.

→ For two liquids,  $w_1 = 2\pi r_1 \gamma_1$ ,  $w_2 = 2\pi r_2 \gamma_2$

$$\frac{w_1}{w_2} = \frac{\gamma_1}{\gamma_2}$$

→ This drop weight method can be employed for comparing surface tension of 2 diff liquids. However, it is easily to count the no. of drops by equal volumes of 2 liquids than finding the weight of single drop.

→ for two diff. liquids, the weight of equal volumes are proportional to their density. Let  $n_1, n_2$  be the no. of drops of 2 liquids.

$$\text{volume of single drop} = \frac{V}{n_1}$$

$$\text{weight of single drop} = V \frac{d_1}{N_1}$$

$$2\pi r \gamma_1 = V \frac{d_1}{N_1} \rightarrow ①$$

Similarly for 2nd liquid 2<sup>nd</sup> liquid is bi-pipit acts along

$$2\pi r \gamma_2 = V \frac{d_2}{N_2} \rightarrow ②$$

Dividing these two equations i.e  $1/2$  we get,

$$\frac{\gamma_1}{\gamma_2} = \frac{N_1 d_1}{N_2 d_2} \rightarrow ③$$

Procedure: In work a, add gradles a divergent well of

Take a clean standard Stalagmometer (no. 0 of 3512) &

• Suck the distilled water to the level above the mark in Stalagmometer.

• Now allow the liquid to flow and count the no. of drops falling from upper mark to the lower mark.

• Repeat H  
observation

S.No.

1. Dist

2. unk

calculate

Surface

Density

Density

No. of

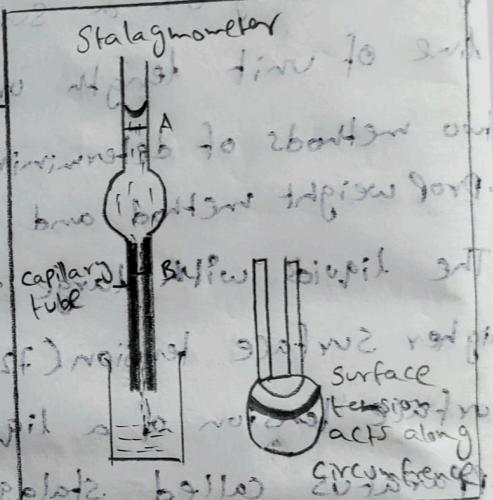
No. of

Surface

Result:

Surface

b.p.



for  
However,  
by equal  
weight  
will volume  
be the no.

- Repeat the above procedure with the given liquid.
- Observations & Calculations

| S.No. | Liquid                 | No. of drops   |                |         | Surface tension<br>(dynes/cm) |
|-------|------------------------|----------------|----------------|---------|-------------------------------|
|       |                        | N <sub>1</sub> | N <sub>2</sub> | Average |                               |
| 1.    | Distilled water        | 155            | 141            | 148     | 72.8                          |
| 2.    | Unknown organic liquid | 315            | 327            | 321     | 26.38                         |

Calculate the surface tension by using the formula.

$$\frac{Y_1}{Y_w} = \frac{N_w d_1}{N_1 d_w}$$

Surface tension of water ( $Y_w$ ) = 72.8 dyne/cm

Density of water ( $d_w$ ) = 1 g/cc

Density of unknown liquid ( $d_1$ ) = 0.786 g/cc

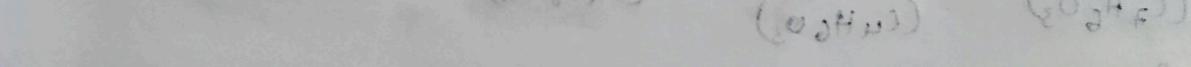
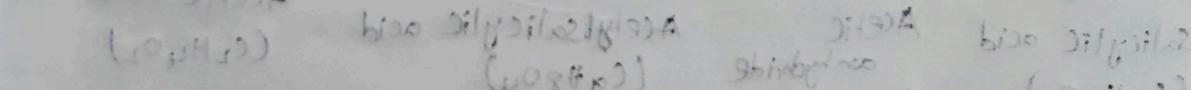
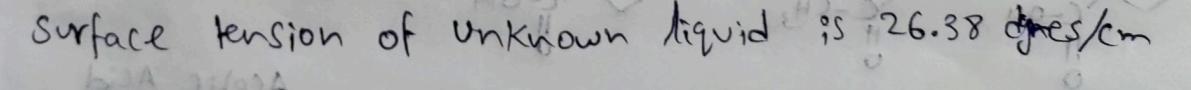
No. of drops of water ( $N_w$ ) = 148

No. of drops of liquid ( $N_1$ ) = 321

Surface tension of the liquid ( $Y_1$ ) =

$$\text{Now } Y_1 = \frac{N_w d_1}{N_1 d_w} Y_w = \frac{148 \times 0.786 \times 72.8}{321 \times 1} = 26.38 \text{ dyne/cm}$$

Result: Surface tension of Unknown liquid is 26.38 dyne/cm



## DRUG SYNTHESIS-I

### II. PREPARATION OF ASPIRIN

Aim: To prepare a sample drug of aspirin from

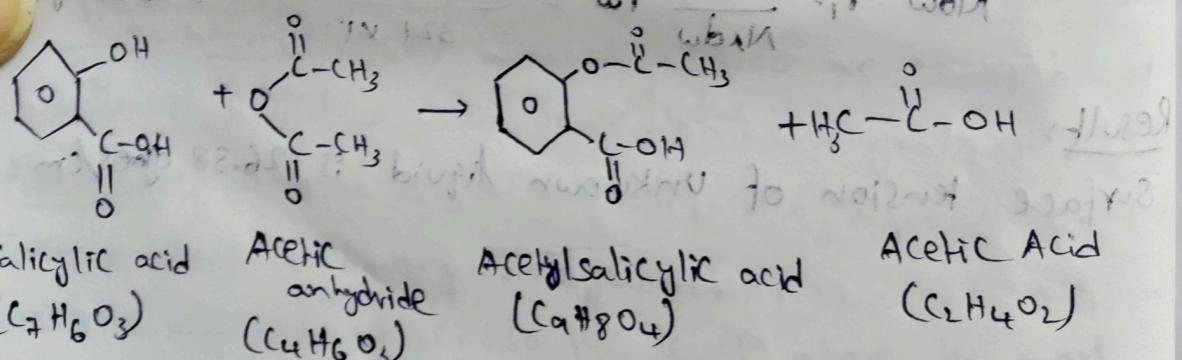
Salicylic acid.

Chemicals: Salicylic acid, conc.  $H_2SO_4$ , acetic anhydride.

Apparatus: Beakers, funnel, conical flask, glass rod, filter papers, filtration unit etc.

Theory: The chemical name of aspirin is acetyl salicylic acid. Aspirin is extensively used as an anti-pyretic, analgesic, anti-inflammatory drug, as a pain killer in treatment (of) rheumatism, arthritis, as a medicine which prevents heart attack by checking clotting in arteries.  $\therefore$  (ab) bispid reaction to ptional

Principle: Salicylic acid undergoes  $O$ -acetylation with acetic anhydride in the presence of conc.  $H_2SO_4$  to give  $O$ -acetyl salicylic acid



#### Procedure:

Take 2gm of salicylic acid, 4 ml of acetylating mixture (Acetic anhydride and glacial acetic acid) and 1-2 drops

of conc. sulphuric acid in a round bottomed flask/ conical flask. The mixture is warmed on water bath at  $50-60^{\circ}\text{C}$  with occasional shaking for 30 min. Allow the solution to cool to room temp. and pour the solution into 100 ml of cold water taken in 250 ml beaker with stirring (to destroy the excess of reagents). Scratch the sides of beaker with a glass rod to induce crystallization and stir the solution till complete precipitation. Filter the colourless crystalline solid and wash it with cold water. Spread the solid the aspirin on a filter paper and dry the solid.

The crude aspirin so obtained is recrystallised by dissolving it in alcohol and pouring it into 400 ml of warm water. The solution which then becomes turbid is warmed to give a clear solution and then allowed to cool slowly. The needles which separate out are filtered. The melting point of the sample of aspirin is  $130^{\circ}\text{C} - 135^{\circ}\text{C}$ .

#### Precautions:

- Take acetyl chloride in excess as it can act as acetylating reagent and solvent. Make sure that all the salicylic acid is dissolved in acetylating mixture.
- The presence of un-reacted salicylic acid is checked by adding a drop of 1% of  $\text{FeCl}_3$  solution to reaction mixture and observing the color.

## Calculations:

Result: The yield of prepared Aspirin is 150.800 g.

Melting point of Aspirin is 132 °C