SRM VALLIAMMAI ENGINEERING COLLEGE

(An Autonomous Institution)
SRM Nagar, Kattankulathur – 603 203

DEPARTMENT OF CHEMISTRY

GE3221-Engineering Sciences Laboratory
Chemistry Lab Manual
II SEMESTER (2024-25)
Regulations 2023

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GENERAL INSTRUCTIONAL POLICIES TO BE FOLLOWED IN THE CHEMISTRY LABORATORY

1. Students must wear a knee length (41-42 inch) laboratory coat while entering into the laboratory.
2. Students must wear closed leather shoe (black or brown colour) for safety purpose.
3. Students must always come to the chemistry lab with lab manual, observation note, and record note.
4. Students should bring waste cloth for cleaning their hands.
5. Students may wear chemical safety goggles, if it is required.
6. Students should report to the concerned chemistry lab as per the time-table schedule.
7. Students, who turn up late to the lab, will not be permitted to do the experiment.
8. Students are required to prepare thoroughly to perform the experiment before coming to chemistry laboratory
9. After completion of the experiment, students must get the signature of the concerned staff in-charge in the observation note.
10. The record note must be submitted to get signature of the previous week experiment to the concerned staff while coming to lab.
11. When the experiment is completed, students should clean the apparatus carefully and should return all the reagent bottles/components/instruments taken for the experiment.
12. Any damages to apparatus occurred during the experiment, should be brought to the notice oflab in-charge. The cost of the repair or new apparatus should be brought by the students.
13. Students should be present in the lab till the completion of allotted hours of lab.
14. Students should not sit on the work tables or keep hands for rest on tables for their own safety.
15. All doors and windows must be kept open while working in the laboratory. Fans and lights should be switched off, if they are not required.

16. Long hair should be tied when in the laboratory so that it will not catch on fire or come into contact with chemicals.
17. Do not enter the laboratory until your lab instructor is present.
18. While leaving the lab ensure that all gas tap connections, water taps, electrical switches such as and fans, lights shall be kept in off mode. All instruments must be properly shut down as per instructions of the teacher in charge.
19. Do not eat, drink and chew gum or smoke in the laboratory.
20. Do not perform unauthorized experiments. If you see someone else doing something you think may be dangerous, tell him or her to stop and/or report the incident to your lab instructor.
21. In case of emergency, do not get panicky and be brave enough to handle the situation. Medical attention should be requested immediately. If skin comes into contact with acid solutions, it should be washed with water and sodium bicarbonate solution alternately and continuously till skin is smoothened. Similarly, exposure to caustic alkalis may be treated with dilute acetic or boric acid solutions and water. Fire scar can be washed thoroughly with water in a tap in the sink and a little oil or pipette be applied. If eyes come into contact with chemicals, wash them with plenty of water and clean them with a dry fresh cloth.
22. Fire buckets containing sand and water or fire extinguishers should always be available in the lab in easily accessible places.

Vision of the Chemistry Department

Bridge the engineering professionals in recent advancements in chemistry for the developments of new devices and materials for the lives of individuals becoming different and complex global society.

Mission of the Chemistry Department

To draw up outstanding educators, employers and researchers and to make progress in the profession of education through teaching and learning and art of research on chemical science towards engineering & technology.

GE3221 ENGINEERING SCIENCES LABORATORY

(Second Semester- Common to all branches)

CHEMISTRY LABORATORY: (Any five experiments to be conducted)

OBJECTIVES

- To train the students in basic experimental skills in water contaminants such as copper and chromium.
- To familiarize the students with electroanalytical techniques such as pH metry, potentiometry, and conductometry to determine impurities in aqueous solutions.
- To familiarize the students with the determination of the molecular weight of a polymer by a viscometer.
- To make the student up-to-date with the properties and nature of alloys experimentally.
- To demonstrate the analysis of coal.

1. Estimation of copper content of the given solution by Iodometry.
2. Determination of strength and amount of acids in a mixture of acidsusing a conductivity meter.
3. Determination of strength and amount of HCl present in the whole of the given solution using a conductivity meter.
4. Estimation of the iron content of the given solution using a potentiometer.
5. Determination of chromium by EDTA titration.
6. Determination of strength of given hydrochloric acid using a pH meter.
7. Determine the molecular weight of the polyvinyl alcohol using an Ostwald viscometer.
8. Estimation of Nickel in steel.
9. Proximate Analysis of Coal.
10. Corrosion experiment-weight loss method.
11. Determination of COD value of industrial effluents.

TOTAL: 30 PERIODS

OUTCOMES

At the end of the course, the student should be able:

1. To find the quality of water samples for copper and chromium present in water.
2. To recognize the amount of various ions, present in the water sample through volumetric and instrumentation techniques.
3. To identify the molecular weight of the polymer using an Ostwald viscometer.
4. To recognize an environmental hazardous and threshold limit forindustrial effluents.
5. To recommend quality of coal and steel when it is exposed to various environment.

TEXTBOOKS

1. Vogel's Textbook of Quantitative Chemical Analysis (8th Edition, 2014).
2. Suchi Tiwari, Engineering Chemistry Lab Manual, Scitech Publications(India) Pvt. Ltd. (2nd Edition, 2013).
3. Pushpendra Kumar, Laboratory Manual for Engineering Chemistry,Reyansh Authortopic Pvt. Ltd., (1st Edition, 2022).

CONTENTS

Titration 1: Standardization of Sodium thiosulphate

Standard Potassium dichromate Vs Sodium thiosulphate

Sl.No	Volume of std. Potassium dichromate (mL)	Burette reading (mL)		Volume of sodium thiosulphate (mL)	Concordant value (mL)	Indicator
		Initial	Final			
1.	20	0				Starch
2.	20	0				

Calculation of the strength of Sodium thiosulphate

Volume of standard potassium dichromate $(V_1) = 20$ mL

Strength of standard potassium dichromate $(N_1) = 0.01$ N

Volume of unknown Sodium thiosulphate $(V_2) =$ _____ mL

Strength of unknown Sodium thiosulphate $(N_2) =$ _____ ?

According to volumetric formula, $V1 \times N1 = V2 \times N2$

$(N_2) = V_1 \times N_1 / V_2$

$(N_2) = 20 \times 0.01\ N / V_2$

Strength of sodium thiosulphate $(N_2) =$ _____ N

1. ESTIMATION OF COPPER CONTENT OF THE GIVEN SOLUTION BY IODOMETRY

Expt. No.	

Date			

AIM:

Estimate the amount of copper in the given solution by iodometric method. You are provided with a standard solution of potassium dichromate of strength 0.1 N and sodium thiosulphatesolution as link.

CHEMICALS REQUIRED:

1. Potassium dichromate
2. Sodium thiosulphate
3. Potassium iodide
4. Starch
5. Sulphuric acid
6. Ammonia
7. Acetic acid
8. Ammonium thiocyanate.

PRINCIPLE:

The strength of copper sulphate solution is determined by iodometric method. When potassium iodide is added to a solution of copper sulphate, a white cuprous iodide (Cu_2I_2) is precipitated and an equivalent amount of iodine is liberated. The free iodine is titrated against standard solution of sodium thiosulphate using starch as an indicator,

As soon as all the liberated iodine is reduced to iodide (NaI), the blue colour of the iodo-starch complex will disappear. The colour of precipitate in the conical flask will be white, Cu_2I_2. This indicates theend point. The reactions involved are:

$$2CuSO_4 + 4KI \longrightarrow Cu_2I_2 + K_2SO_4 + I_2$$

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

From the titre values, the strength of copper sulphate solution and the amount of copper are calculated.

$$K_2Cr_2O_7 + 7H_2SO_4 + 6KI \longrightarrow 4K_2SO_4 + Cr_2(SO_4)_3 + 3I_2 + 7H_2O$$

Titration 2: Estimation of Copper

Standardized sodium thiosulphateVs Copper sulphate

Sl.No	Volume of unknown $CuSO_4$ (mL)	Burette reading (mL)		Volume of sodium thiosulphate (mL)	Concordant value (mL)	Indicator
		Initial	Final			
1.	20	0				Starch
2.	20	0				

Calculation of the strength of Copper

Volume of standardized sodium thiosulphate (V_1) = _____ mL

Strength of standardized sodium thiosulphate (N_1) = 0.01 N

Volume of unknown Copper sulphate (V_2) = _____ mL

Strength of unknown Copper sulphate (N_2) =_____ ?

According to volumetric formula, $V1 \times N1 = V2 \times N2$

$(N_2) = V_1 \times N_1 / V_2$

$(N_2) =$

Strength of Copper sulphate (N_2) = _____ N

Calculation of amount of Copper

Amount of copper present in 1 L of the given solution

= Strength of Copper x Eq. wt. of Copper

= N_2 x 63.54 g

= _____ g

The liberated iodine is titrated against sodium thiosulphate using starch as indicator. The end point is the appearance of green colour. From the equations, it is clear that the equivalent weight of crystalline copper sulphate ($CuSO_4.5H_2O$) is its molecular weight. i.e., 249.54 and that of copper is its own atomic weight. i.e., 63.54.

PROCEDURE:

Titration 1: Standardization of sodium thiosulphate

The burette is washed and rinsed with sodium thiosulphate solution. Then the burette is filled with this sodium thiosulphate solution up to zero mark without any air bubbles. 20 mL of 0.1N potassium dichromate solution is pipetted out by means of clean and rinsed 20mL pipette into a clean conical flask. About 10mL 10% potassium iodide solution is added. The liberated iodine is titrated at once against sodium thiosulphate solution taken in the burette. When the solution in the conical flask becomes straw yellow colour, 1mL of freshly prepared starch solution is added as indicator and the titration is continued. The end point is the disappearance of blue colour and the appearance of pale green colour. Titre value is noted Titrations are repeated for concordant values.

Titration 2: Estimation of copper

20 mL of the given copper sulphate solution is pipetted out by means of a clean and rinsed 20 mL pipette into a clean conical flask. Ammonium hydroxide is added drop by drop till a permanent precipitate s formed. The precipitate is re dissolved by adding drop by drop acetic acid.

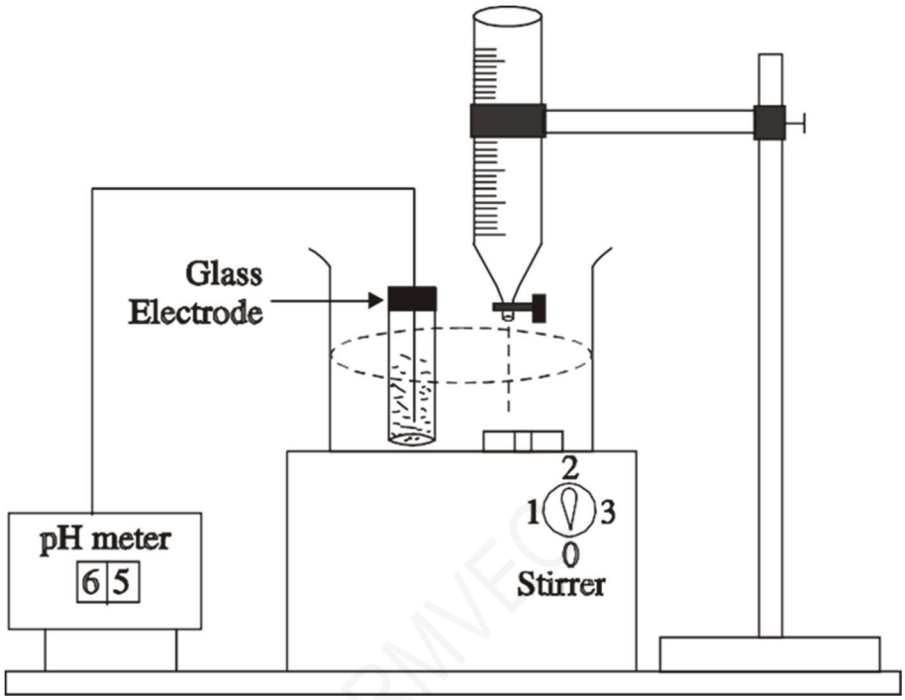
About 5 mL of 10% solution of potassium iodide is added. The liberated iodine is titrated against sodium thiosulphate solution taken in the burette. 1 mL of freshly prepared starch solution is added as indicator towards the end point. The end point is the disappearance of blue colour. The titration is repeated till concordant values are obtained.

From the titre values, the strength of copper sulphate and the weight of copper in the whole of thegiven solution can be calculated. The equivalent weight of copper is 63.54.

RESULT:

The strength of copper sulphate solution = _____ N

The amount of copper present in the given solution = _____ g



Determination of pH

Table 1
Titration of HCl vs NaOH

S. No	Volume of NaOH (mL)	pH

2. DETERMINATION OF STRENGTH OF GIVEN HYDROCHLORIC ACID USING A p^H METER

Expt. No.	

Date			

AIM:

To determine the strength of given hydrochloric acid by pH metry. You are providedwith a standard solution of 0.1N sodium hydroxide.

MATERIALS REQUIRED:

Hydrochloric acid, sodium hydroxide, pH meter, glass electrode

PRINCIPLE:

pH of the solution is related to the H_3O^+ ion concentration of the solution by theexpression.

$$pH = - \log [H_3O^+]$$

The concentration of H^+ ions in the solution is determined by measuring the pH of the solution.

When NaOH is added slowly from the burette to the solution of HCl, H^+ ions are neutralized by OH^- ions.

As a result, pH of the solution increases.

$$HCl + NaOH \longrightarrow NaCl + H_2O$$

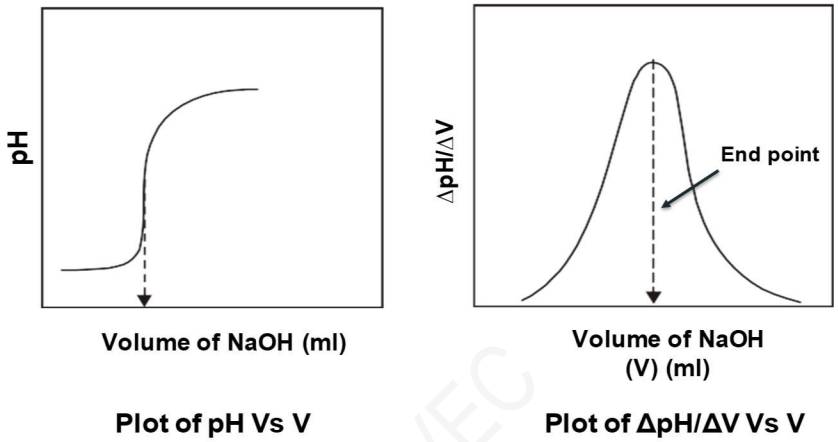
The increase in pH takes place until all the H^+ ions are completely neutralized. After the endpoint, further addition of NaOH increases the pH sharply as there is an excess of OH^- ions.

PROCEDURE:

The burette is filled with standard NaOH solution. 10 mL of HCl solution is pipetted out into a clean beaker. It is diluted to 100 mL with distilled water. The glass electrode is dipped into the solution and connected to the pH meter.

The NaOH solution is gradually added from the burette to the HCl solution in the beaker. pH is noted after each addition. The observed pH values are plotted against the volume of NaOH added. From the graph the end point is determined.

Model graph



Plot of pH Vs V

Plot of ΔpH/ΔV Vs V

Table 2
Titration of HCl vs NaOH

S. No	Volume of NaOH (mL)	pH	Δ pH	ΔV (mL)	Δ pH/ΔV	$V = (V_1+V_2)/2$ (mL)

Calculation of strength of HCl

Volume of mixture (HCl) $(V_1) = 10$ mL

Strength of mixture (HCl) $(N_1) =$ _____ ?

Volume of NaOH $(V_2) =$ _____ mL

Strength of NaOH $(N_2) = 0.1$ N

$(N1) = V2 \times N2/V1$

$= V2 \times 0.1/10$

Strength of HCl $(N_1) =$ _____ N

The amount of HCl present in

1 litre of the given solution = Strength of HCl × Eq.wt. of HCl

$= N \times 36.5$

= _____ g/L

The amount of HCl present in

100 mL of the given solution $= N \times 36.5/10$ g

= _____ g

RESULT:

The amount of HCl present in the given solution = _____ g/L

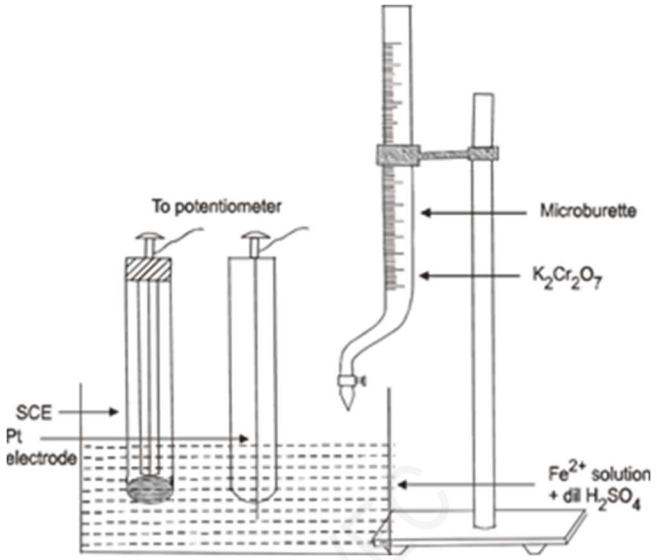


Table 1

$K_2Cr_2O_7$ vs Iron solution

S. No	Volume of $K_2Cr_2O_7$ added (mL)	EMF (mV)

3. ESTIMATION OF THE IRON CONTENT OF THE GIVEN SOLUTION USING A POTENTIOMETER

Expt. No.	

Date			

AIM:

To estimate the weight of Fe^{2+} ion present in the given solution. You are provided with 0.2 N solution of dichromate.

MATERIALS REQUIRED:

Potentiometer, platinum electrode, std. calomel electrode, std. $K_2Cr_2O_7$ solution, given ferrous solution.

PRINCIPLE:

Potassium dichromate oxidizes Fe^{2+}ion to Fe^{3+} ion and hence the following electrode is formed.

Pt/ Fe^{2+}, Fe^{3+}

When this is coupled with std. calomel electrode the following cell is obtained.

Pt/ Fe^{2+}, Fe^{3+}// SCE

The potential of the cell depends on the ratio (Fe^{3+}/ Fe^{2+}). Initially, during the addition of $K_2Cr_2O_7$ this ratio does not change appreciably. At the end point the ratio increases suddenly leading to a large increase in the potential measured. The addition of $K_2Cr_2O_7$ after the end point does not change this ratio and hence the potential of the cell remains almost constant.

PROCEDURE:

10mL of the given ferrous solution is pipetted out in a clean 250 mL beaker. To this, 10mL of H_2SO_4 and 80mL of conductivity water were added. Platinum electrode and saturated calomel electrode are dipped to form a cell. It is then connected to a digital potentiometer. The std. $K_2Cr_2O_7$ solution is taken in the burette.

Titration 1

A preliminary titration is carried out by adding std. $K_2Cr_2O_7$ solution in 1 mL portions and the emf of the cell is measured each time after the addition. The addition of $K_2Cr_2O_7$ is continued even after the end point.

Model graph

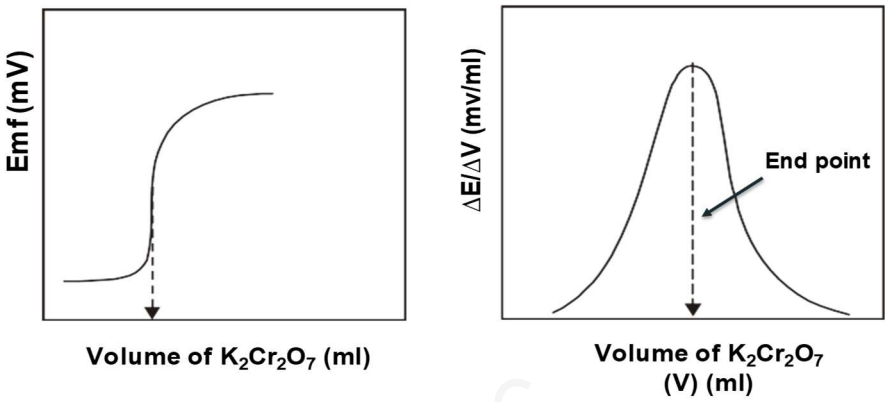


Table 2

$K_2Cr_2O_7$ vs Iron solution

S. No	Volume of $K_2Cr_2O_7$ (mL)	EMF (mV)	ΔE (mV)	ΔV (mL)	ΔE/ΔV (mV/mL)	$V = (V_1+V_2)/2$ (mL)

Calculation of strength of Fe^{2+} ion

Volume of ferrous ion solution $(V_1) = 10$ mL

Strength of ferrous ion solution $(N_1) =$ _____ ?

Volume of $K_2Cr_2O_7$ $(V2) =$ _____ mL (Titre value)

Strength of $K_2Cr_2O_7$ $(N_2) = 0.2$ N

$(N1) = V2 \times N2/V1$

$= V2 \times 0.2/10$

Strength of $K_2Cr_2O_7$ $(N_1) =$ _____ N

The amount of ferrous ion present in

1 litre of the given solution = Strength of ferrous ion solution × Eq.wt. of Fe^{2+}

$= N \times 55.85$

= _____ g/L

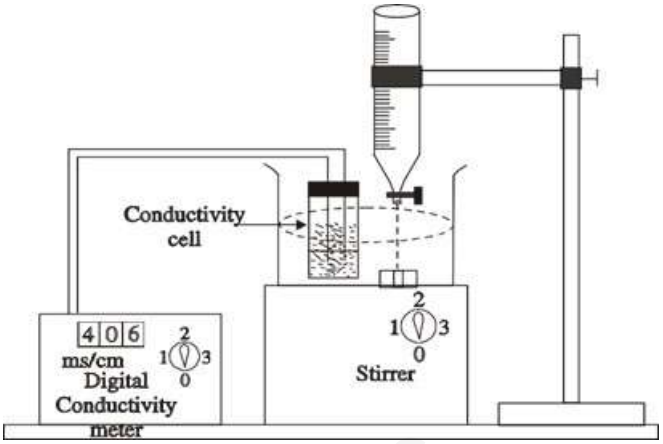
The volume at which there is a sharp increase in emf is the end point. The range at which the end point lies is found out by plotting volume of $K_2Cr_2O_7$ against emf (graph 1).

Titration 2

Titration is carried out by adding std. $K_2Cr_2O_7$ solution in portions of 0.2 mL near the end point and the emf of the cell is measured after each addition. The addition of $K_2Cr_2O_7$ is continued even after the end point for further 3-4 mL. The accurate end point is determined by plotting $\Delta E/\Delta V$ vs volume of $K_2Cr_2O_7$ added (graph 2). From the end point the strength of ferrous ion in the solution is calculated and the amount of Fe^{2+} ion is determined.

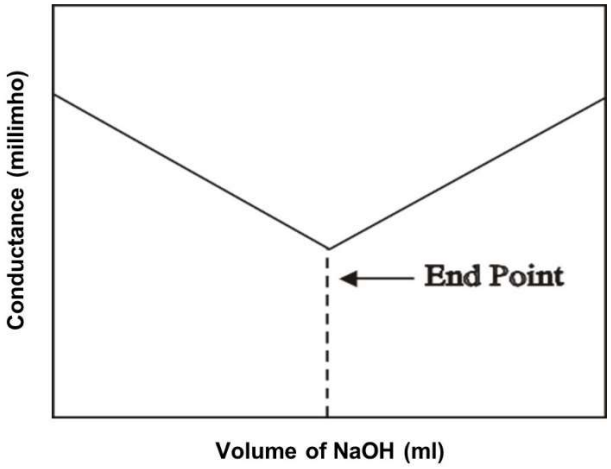
RESULT

The amount of ferrous ion present in the given solution = _____ g/L



Determination of Conductance

Model graph



4. DETERMINATION OF STRENGTH AND AMOUNT OF HCL PRESENT IN THE WHOLE OF THE GIVEN SOLUTION USING A CONDUCTIVITY METER

Expt. No.	

Date			

AIM

To determine the amount of strong acid (HCl) present in the given sample by Conductometric titration. You are provided with NaOH solution of strength 0.5N.

PRINCIPLE

A solution of an electrolyte conducts electricity due to its dissociation in to ions which depends on their number and mobility. Since specific conductance of a solution is proportional to the concentration of ions in it, conductance of the solution is measured during titration.

A reaction between strong acid and strong base, leading to the following neutralization.

$$HCl + NaOH \longrightarrow NaCl + H_2O$$

This reaction is followed conductometrically in a conductivity bridge using a conductivity cell. When a strong base of NaOH is added slowly from the burette to the solution of HCl, the fast moving H^+ ions are progressively replaced by slow moving Na^+ ions. As a result, conductance of the solution decreases. This decrease in conductance will take place until the end point is reached. Further addition of NaOH increases the conductance sharply as there is an excess of fast moving OH^- ion.

$$NaOH \longrightarrow Na^+ + OH^-$$

A plot is made between volume of NaOH added and the conductance of solutions. The end point is intersection of the two lines.

PROCEDURE

The micro burette is filled with standard NaOH solution. 20 mL of the given HCl is pipetted out in to a clean 100 mL beaker. The conductivity cell is placed in it and then diluted to 50 mL by adding conductivity water. The two terminals of the cell are connected to conductivity bridge. Initial conductance is read in the instrument for the acid alone without the addition of NaOH.

Now 0.05 mL of NaOH solution from the burette is added to the solution taken in the beaker, stirred and then conductivity is measured. The process is continued up to the end point. After the end point, further NaOH is gradually added and few more readings are noted.

Table

Sl. No.	Volume of NaOH added (mL)	Conductance (milli mho)
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		

Calculation of strength of HCl

Volume of NaOH (V_1) = _____ mL (Titre value)

Strength of NaOH (N_1) = _____ N

Volume of HCl (V_2) = 20 mL

Strength of HCl (N_2) = _____ ?

By volumetric principle $V1\ N1 = V2\ N2$

$(N_2) = V1 \times N1/V_2 = V_1 \times N_1/20$

Strength of HCl (N_2) = _____N

Calculation of amount of HCl

The amount of HCl present in

the whole of the given solution = Strength of HCl $(N_2) \times$ Eq.wt. of HCl

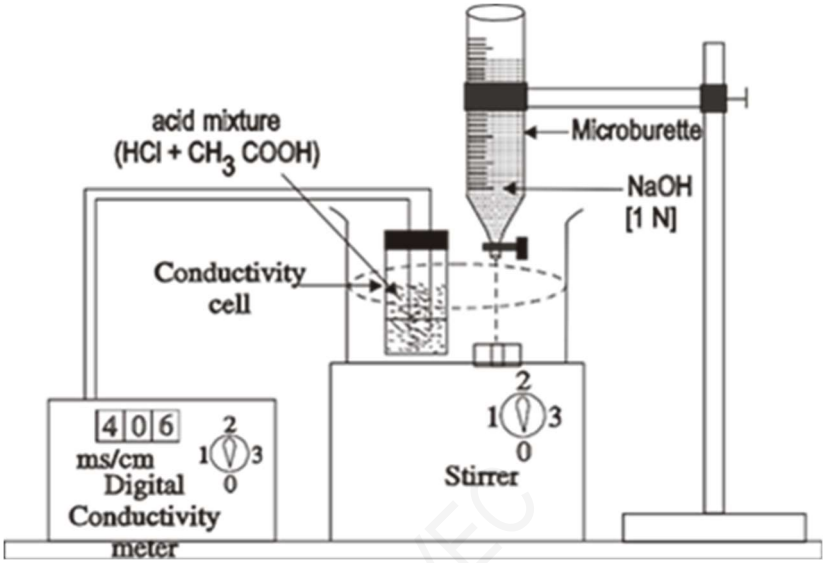
$= N \times 36.5$

= _____ g/L

A graph is plotted between the volume of NaOH and conductance and the end point is noted. It is the intersection of the two lines as in the figure. The amount of HCl present in the given solution is calculated.

RESULT:

The amount of HCl present in 1 litre of the given solution = _____ g/L



Model graph

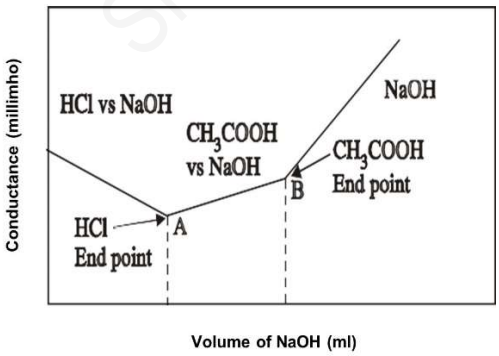


Table 1

Titration of mixture of acids vs NaOH

S. No.	Volume of NaOH added (mL)	Conductance (mho)

5. DETERMINATION OF STRENGTH AND AMOUNT OF ACIDS IN A MIXTURE OF ACIDS USING A CONDUCTIVITY METER

Expt. No.	

Date			

AIM:

To estimate the amount of hydrochloric (HCl) and acetic acids (CH_3COOH) present in the given mixture. You are provided with a standard solution of NaOH of strength 0.5 N.

MATERIALS REQUIRED:

Conductivity meter, conductivity cell, micro burette, pipette, Std. 0.5N NaOH.

PRINCIPLE:

Conductivity of a solution depends upon the number and nature of ions in solution. During the addition of base to acid solution, the H^+ ions are removed as water. Hence the conductance of the solution decreases.

$$H^+ + OH^- \longrightarrow H_2O$$

Acid Base

In a mixture of strong acid and weak acid, strong acid reacts with the base initially. The decrease in conductivity is sharp. After the neutralization of strong acid, weak acid reacts with the added base. Acetic acid being weakly ionized, the decrease in conductivity is slow. At the end point, when all the acids are neutralized, the addition of base increases the OH^- ion in solution. Hence there is a sharp increase in conductivity.

PROCEDURE:

The burette is filled with sodium hydroxide (NaOH) solution up to the zero mark.10 mLof HCl and 10mL of CH_3COOH are taken in a 250 mL beaker. The conductivity cell is placed in it and then diluted to 100 mL with distilled water. It is then connected with the conductivity meter. Then 0.5 mL of alkali is added from the burette to the solution. The solution is stirred carefully and then conductance is measured after each addition of alkali. A graph is drawn between conductance and volume of NaOH. From the graph, the first end point Aand the second end point B are noted. The amount of HCl and CH_3COOH present in 1 litre of the mixture is calculated from the end points A and B respectively.

Calculation of strength of HCl:

Volume of mixture (HCl) $(V_1) = 20$ mL

Strength of mixture (HCl) $(N_1) =$ _____ ?

Volume of NaOH $(V2) =$ ––––––(A) mL (1^{st} Titre value)

Strength of NaOH $(N_2) = 0.5$ N

$(N1) = V2 \times N2/V1$

$= V2 \times 0.5/20$

Strength of HCl $(N_1) =$ _____ N

The amount of HCl present in

1 litre of the given solution = Strength of HCl × Eq.wt. of HCl

$= N \times 36.5$

= _____ g/L

Calculation of strength of CH_3COOH:

Volume of mixture (CH_3COOH) $(V_1) = 20$ mL

Strength of mixture (CH_3COOH) $(N_1) =$ _____ ?

Volume of NaOH (B-A) $(V2) =$ _____ mL

Strength of NaOH $(N_2) = 0.5$ N

$(N1) = V2 \times N2/V1$

$= V2 \times 0.5/20$

Strength of CH_3COOH $(N_1) =$ _____ N

The amount of CH_3COOH present in

1 litre of the given solution = Strength of CH_3COOH × Eq.wt. of CH_3COOH

$= N \times 60$

= _____ g/L

RESULT:

The amount of HCl present in 1 litre of the given solution = _____ g/L

The amount of CH_3COOH present in 1 litre of the given solution = _____ g/L

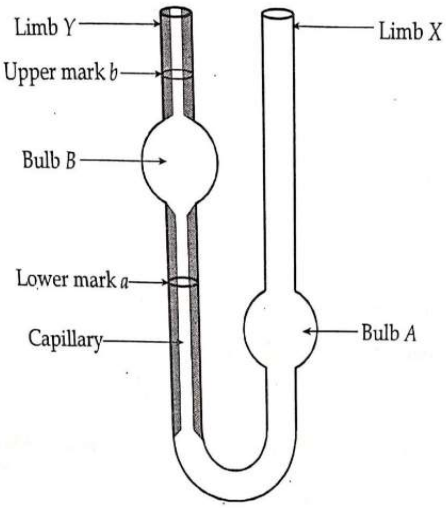


Fig: Ostwald's Viscometer

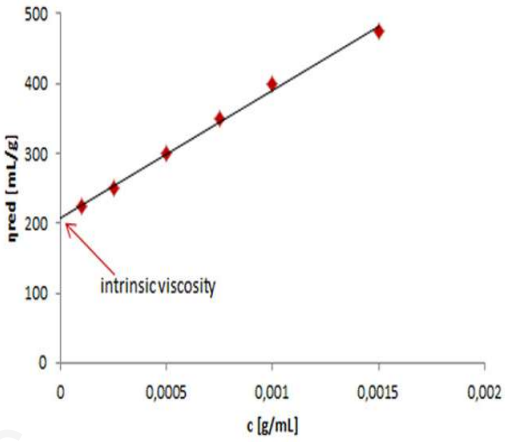


Table 1

Preparation of polymer solutions of various dilutions

Polymer solutions are prepared in a 50 mL standard flask

S.No	Volume of polymer solution (stock solution) (mL)	Volume of distilled water(mL)	Concentration (%)
1	The given 1% solution	-	1%
2	25	25	0.5
3	40	10	0.4
4	30	20	0.3
5	20	30	0.2
6	10	40	0.1

6. DETERMINE THE MOLECULAR WEIGHT OF THE POLYVINYL ALCOHOL USING AN OSTWALD VISCOMETER

Expt. No.		Date			

AIM

To determine the molecular weight of the polymer using Ostwald's viscometer. You are provided with 1% solution of the given polymer (polyvinyl alcohol).

MATERIALS REQUIRED

Polymer solution, Viscometer, solvent, stop clock, 50mL standard flask.

PRINCIPLE

Molecular weight of the polymer means average molecular weight of the polymer. This can be determined from intrinsic viscosity of a dilute polymer solution. The limiting value of reduced viscosity or inherent viscosity at infinite dilution of the polymer is known as intrinsic viscosity of polymer solution. When a very small amount of polymer is added to a solvent of low viscosity, the viscosity of the resulting solution increases sharply. This increase in viscosity depends upon the molecular weight of the polymer, concentration, size and shape of the solute molecules. The relative viscosity is the ratio of the viscosity of the solution to the viscosity of the solvent. This is given by

$$\eta_r = \eta_s / \eta_o$$

η_s is the coefficient of viscosity of the polymer solution

η_o is the coefficient of viscosity of pure solvent (water) at the same temperature.

For linear polymers, the intrinsic viscosity is related to the molecular weight of the polymer by Mark – Kuhn – Houwink equation.

$$[\eta] = k\, M^a$$

Where k and a are constant for a given polymer, solvent combination at a given temperature. The intrinsic viscosity is related to the specific viscosity of a polymer solution by the relation.

$$[\eta] = [\eta_{sp} / C]$$

η_{sp} – specific viscosity of the polymer. It is the relative viscosity of a polymer solution of known concentration minus 1.

C – concentration of the solution

η_{sp} is related to η_r by $[\eta_{sp}] = \eta_r - 1$

Table 2

Viscosity data of polymer solution/solvent

Flow time of pure solvent (t_0) = …….sec

S.No	Concentration of polymer (%)	Flow time t (sec)	$\eta_r = t/t_0$	$\eta_{sp} = \eta_r - 1$	$\eta_{red} = \eta_{sp}/c$
1	0.1				
2	0.2				
3	0.3				
4	0.4				
5	0.5				

Calculation

Mark - Kuhn - Houwink equation:

$[\eta_i] = kM^a$

$\text{Log } \eta_i = \log K + a \log M$

$\text{Log } M = (\log \eta_i - \log K)/a$

$M = \text{antilog } (\log \eta_i - \log K)/a$

Where M = Molecular weight of the polymer

For polyvinyl alcohol,

$K=45.3x10^{-5}$ gm/dl

a = 0.64

η_{sp}/C = Viscosity/concentration in % weight= viscosity x 100/concentration

=

Where $\eta_r = t/t_0$

t and t_0 are flow times for solution and solvent respectively

[Η] can be known by drawing a graph between Ηsp/C vs C. The intercept of Ηsp/C at 0 concentration is known as intrinsic viscosity.

PROCEDURE

Step 1: Polymer solutions of different concentrations 1%, 0.5%, 0.4%, 0.3%, 0.2%, 0.1% are prepared form the given polymer stock solution as in table 1.

Step 2: The solvent is taken in the viscometer. It is sucked through the capillary tube up to the upper mark, without any air bubbles. The flow time of the solvent is noted as it flows from the upper mark to the lower mark. The flow time measurements are repeated twice and the average of the two readings are taken.

Step 3: The viscometer is first filled with the 1% polymer solution. The flow time is measured as before. Similarly, the flow times of other dilutions are also determined and tabulated.

For each polymer solution, the viscometer should be thoroughly washed and rinsed with the solvent.

From the flow times, reduced viscosity (Ηsp/C) can be calculated. Graph is plotted between Ηsp/C vs C. A straight line is obtained. The intercept is called intrinsic viscosity.

Degree of polymerization:

It is the number of repeating units in a polymer molecule. The repeating unit in PVA is corresponding to unit weight of 44.

The degree of polymerization = (Calculated M.wt of polymer/44) per molecule

RESULT

The molecular weight of the given polymer = _____

The degree of polymerization = _____

Table
Water sample Vs Ferrous ammoniumsulphate

S. No.	Volume of water sample (mL)	Burette readings (mL)		Volume of Ferrous ammonium sulphate (mL)	Concordant value (mL)
		Initial	Final		

Calculation:

Quantity of Fe $(NH_4)_2(SO_4)_2$ added for blank (A) = mL

Quantity of Fe $(NH_4)_2(SO_4)_2$ added for sample (B) = mL

Dilution Factor = $\frac{\text{Volume of dilute sample}}{\text{Volume of waste water sample added}}$

$$\text{Dilution Factor} = \frac{40}{10}$$

Chemical Oxygen Demand (COD) = $\frac{\text{(A-B) X N X 8 X 1000 X Dilution Factor}}{\text{Volume of Sample}}$

=

COD = _____ ppm

7. DETERMINATION OF COD VALUE OF INDUSTRIAL EFFLUENTS

Expt. No.		Date			

AIM:

To determine the chemical oxygen demand (COD) exerted by the given waste water sample.

APPARATUS REQUIRED:

Reflux Apparatus, Burette, Hot plate/ Heating mantel.

PRINCIPLE:

The organic matter present in sample gets oxidized completely by $K_2Cr_2O_7$ in the presence of H_2SO_4 to produce CO_2 and H_2O. The excess of $K_2Cr_2O_7$ remaining after the reaction is titrated with $Fe(NH_4)_2(SO_4)_2$. The dichromate consumed gives the oxygen required for the oxidation of organic matter.

REAGENTS:

1. Standard Potassium dichromate 0.2 N
2. Sulphuric acid with reagent (Conc. Ag_2SO_4+ H_2SO_4)
3. Std. Ferrous ammonium sulphate (0.1 N)
4. Ferroin indicator
5. Mercuric sulphate
6. Silver sulphate

PROCEDURE:

1. Take 10 mL of water sample.
2. 5 mL of more conc. Dichromate solution are placed in a flask together withglass beads.
3. Add slowly 15 mL of H_2SO_4 containing Ag_2SO_4 and mix thoroughly.
4. Add pinch of mercurous sulphate (Hg_2SO_4) and silver sulphate (Ag_2SO_4)

Connect the flask to condenser. Mix the contents thoroughly before heating.Improper mixing may result in bumping and the sample may be blown out.

5. Reflux for a minimum period of 2 hours. Cool and wash down the condenserwith distilled water.

6. Dilute the sample to make up to 40 mL with distilled and cool.
7. Add 2-3 drops of Ferroin indicator. Mix thoroughly and titrate it against 0.1 N $Fe(NH_4)_2(SO_4)_2$. Sharp colour change from blue- green to wine red indicatethe end point.
8. Reflux the blank solution in the same manner using distilled water instead of sample.

RESULT:

The COD for the given water sample = _____ ppm