

SATHYABAMA



Regulation: 2019

(DEEMED TO BE UNIVERSITY)

Accredited with Grade 'A' by NAAC

DEPARTMENT OF CHEMISTRY CHEMISTRY LAB MANUAL

I Semester and II Semester B.E. / B.Tech (Common to All Branches of Engineering and Technology)

COURSE NAME: CHEMISTRY LAB

COURSE CODE: SCYA 2101

Name	:
Year	: ——— Branch and Section:
Roll No.	·

GENERAL INSTRUCTIONS

- 100% attendance is compulsory for all the laboratory classes.
- Students must bring their lab coat, lab manual, observation book and record note book for their chemistry practical classes.
- Students should maintain their observation note book and record note book neatly.
- Students should write the date, name of the expt., diagram, tabulation, procedure, etc. in their observation note book before entering the lab.
- Students should write the completed experiment in the record note book only after getting it corrected in the observation note.
- In the record, LHS should be used for diagrams, tabular column, calculation and model graph (if any) and RHS should be used for writing aim, formula, apparatus required, procedure and the result.
- Diagrams, tabular column and graph should be drawn with pencil and procedure in blue / black ink and students should paste the graph along with the corresponding experiments neatly.
- Students who are late for the practical class will not be allowed to do their experiment.
- A place and number will be allotted to each student. Students have to follow this seat number for regular practical classes for the whole semester.
- Do not place any glass apparatus on the working table. They should be kept only in the plastic tray. Do not keep the glass ware at the edge or corner of the table so that breakage of glass ware can be avoided. Always keep your place neat, clean and dry.
- Numbered solution bottles are kept on the side tables in the lab. Students should take the bottles according to their seat number (for some solutions need not follow the seat number, consult with staff for more information). After the completion of experiment, these bottles should be replaced in the same place.
- Each concordant reading should be attested by the staff members before proceeding to the next part of the experiment. Without getting the readings attested, students should not proceed with the experiment.
- Students should wash all the glass wares after completing the experiment and should be replaced in the plastic tray.
- The instruments kept in the lab are already calibrated. These instruments should not be disturbed or tampered. All the instruments and electrodes should be handled carefully.

- Any breakage or damage to glass ware or equipment should be informed to the staff members (do not touch or clean the broken glass wares) immediately. Students should sign in the breakage register and the breakage amount will be collected.
- Materials like filter paper, glass beads etc. should not be thrown in the sink after their usage.
- Students are requested not to leave the lab without the permission of the staff in-charge.
- Your place will be checked by the assistants for cleanliness and for the replacement of the glass ware. Students have to leave the lab only after the period gets over.

Digital Conductivity Meter



Digital Potentiometer



Digital pH Meter



Digital Photocolorimeter



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Edited by: Dr. A. Sheik Mideen

Ex. No. 1 Date:

ESTIMATION OF MIXTURE OF ACIDS BY CONDUCTOMETRY

AIM:

To estimate the amount of acids present in the whole of the given solution. You are provided with standard 0.2 N sodium hydroxide solution.

PRINCIPLE:

Initial conductance of acid mixture is due to the presence of fast moving H⁺ ions. Upon titration with NaOH, the fast moving H⁺ ions are replaced by slow moving Na⁺ ions. This results in decrease of conductance till the entire HCl is neutralized.

$$H^+Cl^- + Na^+OH^- \rightarrow NaCl + H_2O$$

Further addition of NaOH increases the conductance value due to the formation of sodium acetate.

$$CH_3COO^-H^+ + Na^+OH^- \rightarrow CH_3COONa + H_2O$$

After the complete neutralization of acetic acid, any further addition of NaOH increases the conductance value rapidly.

A plot between volumes of NaOH added versus the conductance of the solution will give two intersection points. The first point corresponds to the titration between strong acid vs strong base. The second point corresponds to the titration between weak acid vs strong base.

APPARATUS & REAGENTS:

Conductivity meter, beaker, burette, pipette, glass rod, sodium hydroxide, hydrochloric acid etc.

PROCEDURE:

Makeup the given acid solution into a clean 100 ml standard flask with distilled water. Pipette out 20 ml of the made up acid solution into a clean 100 ml beaker. Pipette out 20ml of distilled water into the beaker. Rinse the conductivity cell thoroughly with distilled water and immerse into the beaker. Stir the solution gently and carefully with a glass rod. Measure the initial conductance of acid solution.

Now add 1 ml of NaOH from the burette. Stir the solution carefully with the glass rod and note the conductance.

Continue the titration by adding NaOH (in an aliquot of 1 ml) from the burette. When we add NaOH the conductance initially decreases to certain volume of NaOH. conductance starts increasing slightly for the addition of NaOH. After a particular volume the conductance increases rapidly. Plot a graph between the conductance values and volume of NaOH added as shown in the model graph. The first point of intersection of the two lines in the graph gives the first end point of the titration. Consider this value as V1. Consider the second point of intersection of the two lines in the graph as V_3 . V_3 — V_1 gives the second end point of the titration.

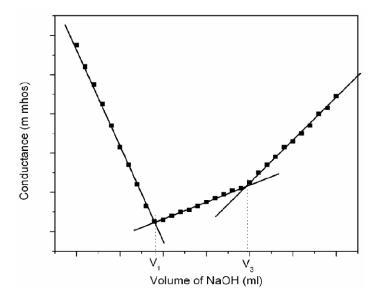
RESULT:

1.	Amount of HCl present in the	whole of the given solution	=	g
2.	Amount of CH ₃ COOH presen	it in the whole of the given solution	=	g

OBSERVATION:

S. No.	Volume of NaOH added (ml)	Conductance (mMho)
1	1	
2	2	
3	3	
4	4	
5	5	
6	6	
7	7	
8	8	
9	9	
10	10	
11	11	
12	12	
13	13	
14	14	
15	15	
16	16	
17	17	
18	18	
19	19	
20	20	
21	21	
22	22	
23	23	
24	24	
25	25	
26	26	
27	27	
28	28	
29	29	
30	30	

MODEL GRAPH:



CALCULATION

1. Volume of NaOH (V_1) ml Normality of NaOH (N_1) = 0.2 NVolume of Mixture of acids (V₂) 20 ml Normality of HCl = $\frac{V_1 \times 0.2}{20}$ [V₁ - Refer graph]

The amount of HCl present in one litre = Normality of $HCl \times Equivalent$ weight of HCl (36.5)

The amount of HCl present in the whole of the given solution

$$= \frac{Normality \ of \ HCl \times 36.5 \times 100}{1000} \ grams$$

2. Volume of NaOH (V₄) =
$$(V_3-V_1)$$
 ml
Normality of NaOH (N₁) = 0.2 N
Volume of mixture of acids (V₂) = 20 ml
Normality of CH₃COOH = $\frac{(V_3-V_1)\times 0.2}{20}$ [V₁ and V₃ - Refer graph]

The amount of CH₃COOH present in one litre

= Normality of $CH_3COOH \times Equivalent$ weight of CH_3COOH (60)

The amount of CH₃COOH present in the whole of the given solution

$$= \frac{Normality\ of\ CH_{3}COOH \times 60 \times 100}{1000}\ grams$$

Ex. No. 2 Date:

ESTIMATION OF FERROUS ION BY POTENTIOMETRY

AIM:

To estimate the amount of ferrous ions present in the whole of the given solution. You are provided with s standard 0.1 N potassium dichromate solution.

PRINCIPLE:

For a ferrous – ferric system: $Fe^{2+} \leftrightarrow Fe^{3+} + e^{-}$

The electrode potential is determined by combining platinum electrode immersed in F^{2+} - Fe^{3+} system with saturated calomel electrode to form a cell and it is measured as emf. The cell is represented by:

$$Hg/Hg_2Cl_2$$
, $KCl(satd)$ // Fe^{3+} , Fe^{2+}/Pt

and E_{cell} is given by:

$$E_{cell} = E_{RHS} - E_{LHS}$$

by applying the Nernst equation,

$$E_{cell} = \left[E_{Fe^{3+}/Fe^{2+}}^{o} + \frac{0.0591}{n} \log \frac{Fe^{3+}}{Fe^{2+}} \right] - 0.2422$$

Where E is the emf of the electrode, $E^o_{Fe^{3+}/Fe^{2+}}$ is the standard electrode potential for ferrous-ferric system, n is the number of electrons involved in the reaction, F is the Faraday's constant, R is the gas constant and T is the temperature.

Ferrous ions are oxidized to ferric ions by adding potassium dichromate which simultaneously increases the emf of the cell with a sharp increase in the emf. at the end point which is due to complete conversion of ferrous to ferric ions.

APPARATUS & REAGENTS:

Potentiometer, standard calomel electrode, platinum electrode (indicator electrode), beaker, burette, pipette, glass rod, standard flask, potassium dichromate, ferrous solution and sulphuric acid

PROCEDURE:

Transfer the given ferrous ion solution into a clean 100 ml standard flask. Make up the solution with distilled water. Pipette out 20 ml of the made up solution into a 100 ml beaker and add 20 ml of dil. H₂SO₄. Immerse the platinum and calomel electrodes in the solution and connect them to the potentiometer.

Stir the solution and note the initial constant emf of the solution. Then add 0.5 ml aliquots of K₂Cr₂O₇ solution from the burette and note the emf. Initially the difference in emf will be small followed by a large difference in the emf values near the end point. After the end point again the difference in emf values will be small. By calculating ΔE , ΔV and $\Delta E / \Delta V$, a graph is plotted by taking volume of $K_2Cr_2O_7$ solution (V) on the X-axis and $\Delta E / \Delta V$ on the Y-axis. From the graph, the volume of K₂Cr₂O₇ solution consumed is determined.

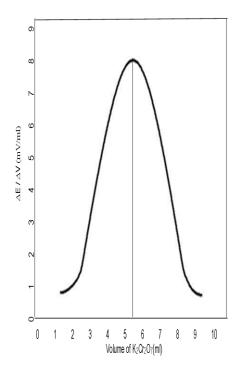
RESULT:

Amount of ferrous ion present in the whole of the given solution = _____ g.

OBSERVAION:

S. No.	Volume of	Observed emf,	ΔΕ	ΔV	$\Delta E / \Delta V$
5. 110.	$K_2Cr_2O_7$, V (ml)	E (mV)		ΔV	(mV/ml)
1	0				
2	0.5				
3 4	1				
4	1.5				
5	2				
6	2.5				
7	3				
8	3.5				
9	4				
10	4.5				
11	5				
12	5.5				
13	6				
14	6.5				
15	7				
16	7.5				
17	8				
18	8.5				
19	9				
20	9.5				
21	10				
22	10.5				
23	11				
24	11.5				
25	12				
26	12.5				
27	13				
28	13.5				
29	14				
30	14.5				
31	15				

MODEL GRAPH:



CALCULATION

Normality of Fe²⁺ ion solution =
$$\frac{V_1 \times 0.1}{20}$$

[V₁ - Refer graph]

The amount of Fe²⁺ ion present in one litre

= Normality of
$$Fe^{2+}$$
 ion × Equivalent weight of Fe^{2+} ion (55.85)

The amount of Fe²⁺ ion present in the whole of the given solution

$$= \frac{Normality\ of\ Fe^{2+}\ ion\ \times 55.85\times 100}{1000}\ grams$$

Ex. No. 3 Date:

DETERMINATION OF pKa VALUE OF GLYCINE BY pH METRY

AIM:

To determine the titration curve of the given amino acid, glycine and to use this curve to estimate its pK_a values.

PRINCIPLE:

Amino acids have the general formula NH₂-CHR-COOH. They can act as both acids and bases. In water, most amino acids exist as zwitter ions, i.e., a dipolar ion. Thus, they are amphoteric molecules that can be titrated with both acid and alkali. Glycine, the simplest amino acid, has one acid and one amine group and is diprotic in nature. Isoelectric point, pI, is the pH of an amino acid at which the net charge on the molecule is zero. When such an amino acid is titrated against a base, it acts as an acid, and with an acid, it acts as a base.

The pH of such a weak acid or base can be calculated from the Henderson-Hasselbalch equation

$$pH = pK_a + \log \frac{[basic\ species]}{[acidic\ species]}$$

APPARATUS AND REAGENTS:

0.1 N glycine, 0.1 N HCl, 0.1 N NaOH, pH meter, pH glass electrode, burettes, beaker and buffer solutions (4, 7 and 9) for calibrating pH meter.

PROCEDURE:

TITRATION I: Glycine vs HCl

10 ml of the given 0.1 N amino acid, glycine is pipetted out into a clean 100 ml beaker followed by the addition of 20 ml of distilled water. A pH glass electrode is rinsed in de-ionized water and immersed in the glycine solution. The pH of the mixture is recorded using a calibrated pH meter. A clean burette is taken and filled with 0.1 N HCl.

Initially, 1 ml of HCl is added to the mixture. The solution is stirred well using a glass rod and the change in pH is recorded. The acid is added in increments of 1 ml until the pH falls to 3. Between pH 3 and 2, HCl is added in increments of 0.5 mL. Beyond pH 2, HCl is added in increments of 1 ml until the pH reaches 1.5. The readings are recorded in a tabular column as shown in Table 1.

TITRATION II: Glycine vs NaOH

10 ml of the given 0.1 N amino acid, glycine is pipetted out into a clean 100 mL beaker followed by the addition of 20 mL of distilled water. A pH glass electrode is rinsed in de-ionized water and immersed in the glycine solution. The pH of the mixture is recorded using a calibrated pH meter. A clean burette is taken and filled with 0.1 N NaOH.

Initially, 1 ml of NaOH is added to the mixture. The solution is stirred well using a glass rod and the change in pH is recorded. The base is added in increments of 1 mL until the pH increases to 9. Between pH 9 and 10, NaOH is added in increments of 0.5 mL. Beyond pH 10, NaOH is added in increments of 1 mL until the pH reaches 11.5. The readings are recorded in a tabular column as shown in Table 2.

The titration curve is plotted with Volume of HCl and NaOH on the x axis and pH on the y axis. The two plateaus on the curve obtained is noted where despite the addition of the acid or base, the pH of the solution does not vary significantly. The pKa values of glycine are estimated from the graph. These values are then compared with that reported in literature.

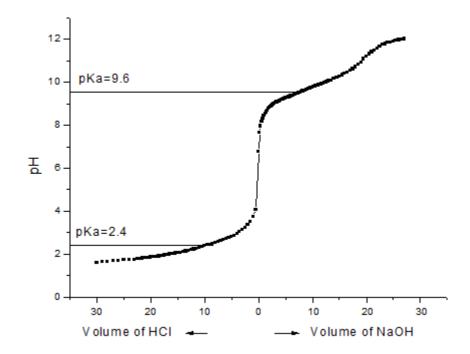
RESULT:	The two pl	ζ, values of	glycine are _	and	
	v ,, o p-	- a , - a -	<u> </u>		

OBSERVATION:

TAB	TABLE 1: Glycine vs. HCl						
Sl.							
No.	0.1 N HCl	Observed pH					
1	1						
2	2						
2 3 4 5 6	3						
4	4						
5	5						
	6						
7	7 8						
8							
9	9						
10	10						
11	11						
12	12						
13	13						
14	14						
15	15						
16	16						
17	17						
18	18						
19	19						
20	20						
21	21						
22	22						
23	23						
24	24						
25	25						
26	26						
27	27						
28	28						
26	26						
30	30						

TABI	TABLE 2: Glycine Vs. NaOH					
Sl.	Volume of	Observed				
No.	0.1 N NaOH	pН				
1	1					
2	2					
3	3					
4	4					
5	5					
6	6					
7	7					
8	8					
9	9					
10	10					
11	11					
12	12					
13	13					
14	14					
15	15					
16	16					
17	17					
18	18					
19	19					
20	20					
21	21					
22	22					
23	23					
24	24					
25	25					
26	26					
27	27					
28	28					
26	26					
30	30					

MODEL GRAPH:



Ex. No. 4 Date:

ESTIMATION OF IRON BY PHOTOCOLORIMETRY

AIM:

To estimate the amount of Fe³⁺ ion present in the given water sample using spectrophotometer.

PRINCIPLE:

When a monochromatic light passes through a homogeneous coloured solution, a portion of incident light is reflected, a portion is absorbed and the remaining is transmitted.

$$I_0 = I_r + I_a + I_t$$

Where

 I_0 = Intensity of light incident before entering the solution

 I_r = Intensity of incident light reflected I_a = Intensity of incident light absorbed Intensity of incident light transmitted

 I_r is usually eliminated and hence $I_0 = I_a + I_t$. The mathematical statement of Lambert- Beer's Law is given by

$$T = I/I_0 = 10^{-kcl}$$

Where

T = Transmittance of solution

I = Intensity of light leaving the solution

 I_0 = Intensity of light incident on the solution

k = Molar absorption coefficient

c = Concentration of the solution in moles/ litre

l = Length (path thickness) of the absorbing solution

(or)
$$A = \log (I_0/I) = k c l$$

where A is the absorbance or optical density of solution i.e when a ray of monochromatic light passes through an absorbing medium, its density decreases exponentially as the concentration of absorbing substance and the width or thickness increases independently. Keeping the path length constant (say l = 1 cm), the variation is only due to concentration, c.

Fe³⁺ ion does not give any colour in solution. However, it develops a red colour when it reacts with potassium thiocyanate solution.

$$Fe^{3+} + 6KSCN \rightarrow [Fe(SCN)_6]^{3-} + 6K^+$$

Red colour complex

Further, this colour is in the blue region, (λ =480 nm). Spectrophotometer has a wide range of adaptability that allows selection of monochromatic light of any wavelength in the visible spectrum.

INSTRUMENTATION:

The light source is an ordinary light bulb and monochromatic light is obtained by using either a glass prism or a diffraction grating. The monochromatic light then passes through the filter and is directed through a cell containing the sample. The incident light passes through the solution and hits the photoelectric cell and the output of this can be seen in the display.

PROCEDURE:

Prepare a series of standard solution containing 2-10 ppm of iron by adding 1 ml of 1:1 HCl and 1 ml of 10% potassium thiocyanate solution. The red solution is made up to 100 ml in a standard flask. Switch on the photocolorimeter and warm it up for about 10 minutes. Adjust the monochromator at the wavelength $\lambda = 480$ nm. The blank is distilled water with the reagents used for the experiment. Keep the blank solution in the cuvette and adjust the instrument to transmittance corresponding to 100 or absorbance is zero. Similarly, place the various standard concentrations of nickel solutions in the cuvette one by one and measure the corresponding absorbance. Also, measure the absorbance of the unknown solution. Draw the calibration graph to determine the concentration of the unknown solution.

RESULT:

Amount of iron present in the given solution = _____ ppm.

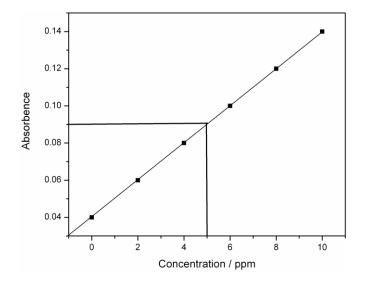
OBSEERVATION:

Wavelength, $\lambda = 480 \text{ nm}$

Distilled water (Blank) = zero absorbance

Sl. No.	Concentration (ppm)	Absorbance
1	Blank	0
2	2	
3	4	
4	6	
5	8	
6	10	
7	Unknown	

MODEL GRAPH:



Ex. No. 5 Date:

ESTIMATION OF TOTAL HARDNESS OF WATER

AIM:

To estimate the total hardness of the given sample of water by EDTA method

PRINCIPLE:

This estimation is based on complexometric titration. Ethylene diamine tetra acetic acid (EDTA) forms stable complexes with calcium and magnesium ions present in the hard water.

$$\begin{array}{c} \text{HOOC-CH}_{\underline{2}} \\ \text{:N-CH}_{\underline{2}}\text{-CH}_{\underline{2}} \\ \text{N:} \\ \text{HOOC-CH}_{\underline{2}} \end{array}$$

The hard water is buffered to a pH of about 9-10 and Eriochrome black T indicator is added. The indicator combines with some of Ca^{2+} and Mg^{2+} ions in the hard water to form an unstable complex of wine red in colour.

$$\begin{bmatrix} Ca^{2+} \\ Mg^{2+} \end{bmatrix} + EBT \longrightarrow \begin{bmatrix} Ca^{2+} \\ Mg^{2+}EBT \end{bmatrix} Complex$$
Hard water indicator (Weak) (Wine red colour)

When the EDTA is added, the free Ca²⁺and Mg²⁺ ions form complex with EDTA. These complexes are more stable than the indicator metal ion complex. When all the hardness causing ions are complexed by EDTA the indicator is set free which gives steel blue colour

APPARATUS & REAGENTS:

Burette, pipette, Conical flask, EDTA, Standard hard water, NH₄OH-NH₄Cl buffer solution, EBT indicator

PROCEDURE:

TITRATION I: Standardization of EDTA solution

Pipette out 20 ml of the standard hard water into a clean conical flask. Add about 5ml of (NH₄Cl + NH₄OH) buffer solution and a small quantity of Eriochrome Black T indicator. The solution turns to wine red in colour. Titrate against EDTA solution taken in the burette until the solution turns to steel blue colour. Repeat the titration for concordant values. Let V1 be the volume of EDTA consumed by standard hard water.

TITRATION II: Estimation of total hardness of given water sample

Pipette out 20 ml of sample hard water into a clean conical flask. Add about 5 ml of (NH₄Cl + NH₄OH) buffer solution and a small quantity of Eriochrome Black T indicator. The solution turns to wine red in colour. Titrate against EDTA solution taken in the burette until the solution turns to steel blue colour. Repeat the titration for concordant values. Let V2 be the volume of EDTA consumed by the sample hard water.

RESULT: Total hardness of the given sample of water = ppm (parts of CaCO₃ per million parts of water).

OBSERVATION AND CALCULATION:

Titration I: Standardization of EDTA solution

Burette : EDTA solution

Pipette solution : 20 ml of std. hard water + 5ml buffer solution

Indicator : Eriochrome Black T End point : Wine red to steel blue

Sl. No.	Volume of Std.	Burette Reading		Volume of	Concordant
	hard water	Initial	Final	EDTA	Value

1 ml of standard hard water = 1 mg of $CaCO_3$ equivalent hardness(1) 20 ml of standard hard water = 20 mg of $CaCO_3$ equivalent hardness(2) 20 ml of standard hard water consumes = V_1 ml of EDTA(3)

From equation (1) and (2):

 V_1 ml of EDTA = 20 mg of CaCO₃ equivalent hardness

1 ml of EDTA = $\frac{20}{V_1}$ mg of CaCO₃ equivalent hardness

Titration II: Estimation of total hardness of water

Burette : Standardised EDTA solution

Pipette solution : 20ml water sample + 5ml buffer solution

Indicator : Eriochrome Black T End point : Wine red to steel blue

S. No.	Volume of Given	Burette Reading		Volume of	Concordant
	Water Sample	Initial	Final	EDTA	Value

20 ml of sample hard water consumes $= V_2$ ml of EDTA $= V_2 \times 1$ ml of EDTA

20 ml of sample hard water $= V_2 \times \frac{20}{V_1}$ mg of CaCO₃ equivalent hardness

1000 ml of sample hard water $= V_2 \times \frac{20}{V_1} \times \frac{1000}{20}$ mg of CaCO₃ equivalent hardness

Total hardness of given water sample = $1000 \times \frac{V_2}{V_1}$ ppm.

Ex. No. 6 Date:

ESTIMATION OF PERCENTAGE OF COPPER IN AN ORE

AIM:

To estimate the percentage of copper present in the given ore. You are supplied with an accurately 0.5 N potassium dichromate solution and an approximately 0.05 N sodium thio sulphate solution.

PRINCIPLE:

The estimation is based on iodometry titration. In iodometry, a strong oxidizing agent such as potassium dichromate is treated in neutral or acid solution with a large excess or iodide ions the latter reacts as a reducing agent and the oxidant will be quantitatively reduced .in such cases an equivalent amount of iodine is liberated and the titrated with a solution of a reducing agent, which is usually sodium thiosulphate freshly prepared starch solution is used as an indicator which given intense blue colour with iodine due to the formation of an adsorption complex. The end point is the disappearance of the blue colour.

A solution of potassium dichromate acidified with dilute sulphuric acid is treated with excess of 10% KI solution. The liberated iodine is treated against sodium thiosulphate using starch indicator until the blue colour disappears.

On adding potassium iodine to a finely ground ore dissolved in perchloric acid, copper reacts to liberate iodine

$$2Cu^{2+} + 4 KI \longrightarrow 4K^{+} + Cu_{2}I_{2} + I_{2}$$

The liberated Iodine is titrated against sodium thiosulphate using starch indicator. The disappearance of blue colour is the end point.

PROCEDURE:

The given copper ion solution is made upto 100ml in a standard flask and is prepared in the following manner.0.5gm of the ore is accurately weighed and dissolved in 15ml of 75% perchloric acid in an iodine flask. The mixture is refluxed without allowing the various to escape. The contents are cooled and 15ml of water is added and again boiled for 5 mintues. After cooling 1:1 ammonium hydroxide solution is added to precipitate ion hydroxide.

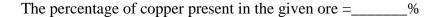
TITRATION I: Standardization of sodium thiosulphate

Pipette out 20ml of standard potassium dichromate into clean conical flask. Add one test tube of dilute sulphuric acid and 10ml of 10% KI solution. Titrate the liberated iodine immediately against sodium thiosulphate taken in the burette. When the solution turns pale yellow, add 1ml of starch indicator and continue the titration. The end point is the disappearance of blue colour. Repeat the titration for concordant titre value and calculate the strength the sodium thiosulphate solution.

TITRATION II: Estimation of copper

Pipette out 20ml of the made up copper solution into a clean conical flask. Add liquor ammonia drop by drop until a deep blue precipitate is formed. Dissolve the precipitate with slight excess of dilute acetic acid and add 10ml of 10% KI solution. Titrate the liberated iodine against sodium thiosulphate taken in the burette. When the solution becomes straw yellow, add 1ml starch indicator and continue the titration. The end point is the disappearance of blue color. Repeat the titration for concordant titre value and calculate the strength of copper in the solution. From this, the amount of copper is estimated.

RESULT:



OBSERVATION AND CALCULATION:

TITRATION I: Standardization of sodium thiosulphate

Burette Solution Sodium thiosulphate

Std. potassium dichromate + 20ml dil.H₂SO₄ +10ml of 10% KI Pipette solution

Indicator Starch

Disappearance of blue colour (Appearance of pale green colour) **End Point**

S. No.	Volume of K ₂ Cr ₂ O ₇ (ml)	Burette Reading		Volume of	Concordant
		Initial	Final	thiosulphate (ml)	value

Volume of potassium dichromate, V_1 ml Normality of potassium dichromate, N₁ N = Volume of Sodium thiosulphate, V_2 = ml Normality of sodium thiosulphate, N_2 ? =

$$V_1N_1 = V_2N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

TITRATION II: Estimation of copper

Burette Solution : Std. sodium thiosulphate

Pipette solution : 20ml of copper ion solution +10ml of 10% KI

Indicator : Starch

End Point : Disappearance of blue color (Appearance of white percipitate)

S. No	Volume of	Burette Reading		Volume of	Concordant
	copper ion solution (ml)	Initial	Final	thiosulphate (ml)	value

CALCULATION:

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

The amount of copper present in one liter of the solution = Normality \times 63.54 = x g/lit.

The amount of copper present in the whole of the given solution $= \frac{x \times 100}{1000}$ = y g

The percentage of copper in the given ore $= \frac{y \times 100}{0.5}$

= _____%

Ex. No. 7 Date:

DETERMINATION OF ALKALINITY OF WATER

AIM:

To determine the types and amount of alkalinity present in the given sample of water. You are provided with a solution of Na₂CO₃ of strength 0.01N and a link solution of HCl solution are provided.

PRINCIPLE:

Alkalinity in water is due to the presence of soluble hydroxides, bicarbonates and carbonates. Determination of the various types and amounts of alkalinity is easily carried out by titration with standard HCl employing the indicators like phenolphthalein and methyl orange independently or successively.

1)
$$OH^- + H^+ \longrightarrow H_2O$$

2) $CO_3^{2^-} + H^+ \longrightarrow HCO_3^-$ Phenolphthalein
3) $HCO_3^- + H^+ \longrightarrow H_2O + CO_2$ Methyl orange

The titration of water sample against a standard acid up to phenolphthalein end point shows the completion of hydroxide alkalinity and one half of carbonate alkalinity present.

On the other hand titration of the sample against a standard acid using methyl orange end point makes the completion of reactions 1 to 3. Total amount of the acid consumed is used to measure the total alkalinity.

The possible combination of ions is OH⁻ only,HCO₃ only,CO₃ ²-only, OH⁻ andCO₃ ²- together and CO₃ ²-and HCO₃ together

OH and HCO₃ ions cannot exist together since both the ions combine instantaneously to form CO_3^{2-} .

$$OH^- + HCO_3^- \longrightarrow CO_3^{2-} + H_2O$$

Similarly CO₃²⁻ OH and HCO₃ cannot exist together.

PROCEDURE

TITRATION I: Standardization of HCl

Pipette out 20 ml of standard sodium carbonate solution into a clean conical flask. Add 3 drops of methyl orange indicator. The solution turns yellow in colour. Titrate the solution against the HCl solution taken in the burette. The end point is the colour change from **vellow to pale pink**. Repeat the titration for concordant titre value.

TITRATION II: Estimation of alkalinity of water sample

Pipette out exactly 20 ml of the given water sample into a conical flask. Add few drops of phenolphthalein indicator and titrated against the standard hydrochloric acid taken in the burette. The end point is the **disappearance of pink colour** (P ml). Into the same solution add few drops of methyl orange indicator. The solution changes to yellow in colour. Continue the titration till **pale pink colour reappears** by adding the same HCl without break (M ml). Titration is repeated till you get concordant titre values.

RESULT:

The individual amounts of alkalinity in the water sample

	= ppm
(b) Carbonate (CO ₃ ² -) alkalinity	= ppm
(c) Bicarbonate alkalinity (HCO ₃ ⁻)	= ppm
(d) Total Alkalinity	= ppm

OBSERVATION AND CALCULATION:

TITRATION I: Standardization of HCl

Burette solution: 20 ml of std. Na₂CO₃

Pipette solution: HCl

Indicator: 2-3 drops of methyl orange

End point: Yellow to pale pink

S. No	Volume of	Burette Reading (ml)		Volume of	Concordant value (ml)
	Na ₂ CO ₃ (ml)	Initial	Final	HCl (ml)	,,

Volume of Na₂CO₃ solution (V_1) = 20 ml= 0.01 NNormality of Na₂CO₃ solution (N₁) Volume of HCl solution (V_2) = ml Normality of HCl solution (N_2) = ?

$$V_1 \times N_1 = V_2 \times N_2$$

$$N_2 = \frac{20 \times 0.1}{V}$$

$$= \dots N$$

TITRATION II: Estimation of alkalinity of water sample

Burette solution: 20 ml of water sample

Pipette solution: HCl

Indicator 1: 2-3 drops of phenolphthalein

End point 1: Disappearance of pale pink colour (Phenolphthalein End Point: P)

Indicator 2: 2-3 drops of methyl orange

End point 2: Reapperance of pale pink colour (Methyl orange End Point: M)

S. No.	Volume of water sample (ml)		Buretto		Volun Std. HO		Concor Valu	rdant 1e (ml)
	(IIII)	Initial	Fin [P]	al [M]	[P]	[M]	[P]	[M]

S.No	Case	OH-	CO3 ²	HCO ₃
1	P=0	Nil	Nil	M
2	P=M	P=M	Nil	Nil
3	P = ½ M	Nil	2P	Nil
4	P > ½ M	2P – M	2(M-P)	Nil
5	P < ½ M	Nil	2P	(M – 2P)

(i) If P > 1/2 M, Water sample contains $OH^+ + CO_3^{-2}$ alkalinity.

$$OH^{-} = \frac{(2P - M) \times Normality of \ HCl \times 50 \times 1000}{20}$$
$$= -----ppm$$

$$CO_3^{2-} = \frac{2(M-P) \times Normality of \ HCl \times 50 \times 1000}{20}$$

$$= -----ppm$$

(i) If P < 1/2 M, Water sample contains $CO_3^{2-} + HCO_3^{-}$ alkalinity.

$$CO_3^{2-} = \frac{2P \times Normality \ of \ HCl \times 50 \times 1000}{20}$$
$$= -----ppm$$

$$HCO_{3}^{-} = \frac{(M-2P) \times Normality \ of \ HCl \times 50 \times 1000}{20}$$

$$= ----- ppm$$

Ex. No. 8 Date:

DETERMINATION OF MOLECULAR WEIGHT OF A POLYMER BY VISCOSITY METHOD

AIM:

To determine the average molecular weight of given polymer by U-tube viscometer method and you are provided with a 5 wt% of polyvinyl alcohol solution.

PRINCIPLE AND PROCEDURE:

The flow time (t_0) for the pure solvent, distilled water is measured by pipetting out 10 ml into the u-tube viscometer. Then, from the given polymer solution, 0.25%, 0.5%, 0.75%, 1% and 1.25% solutions are prepared by using graduated pipette and 25 ml standard flask. 10 ml of each concentration is pipetted out into the u-viscometer and the flow time (t) for different concentration is noted.

The specific viscosity is given by: $\eta_{SP} = \left(\frac{t}{t_o}\right) - 1$ and η_{SP}/c is plotted against concentration, c.

The straight line intersects at Y –axis gives intrinsic viscosity (η_i). For polyvinyl alcohol-water system, the ' α ' and 'K' values are 45.3×10^{-5} dl/g and 0.64 respectively.

The molecular weight of the given polymer is calculated by using expression: $\eta_i = KM^{\alpha}$

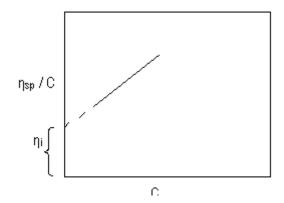
RESULT:

The molecular weight of the given polymer sample =

OBSERVATION:

S. No.	Concentration of the polymer solution, (c)	Time of flow (t)	$\eta/\eta_0 = t/t_0$	$\eta_{\rm sp} = (t/t_0) - 1$	η_{SP} / c
1.	Distilled water	(t_{o})			
2.	0.25%				
3.	0.5%				
4.	0.75%				
5.	1.0%				
6.	1.25%				

MODEL GRAPH:



CALCULATION

$$\eta_i = KM^{\alpha}$$

$$\log \eta_i = \log K + \alpha \log M$$

$$\log M = \frac{\log \eta_i - \log K}{\alpha}$$

$$\log M = \frac{\log \eta_i - \log(45.3 \times 10^{-5})}{0.64}$$

 $[\eta_i$ - Refer graph]

$$\log M = \frac{\log \eta_i - (-3.3437)}{0.64}$$

$$M = anti \log of \left[\frac{\log \eta_i - (-3.3437)}{0.64} \right]$$



Ex. No. 1: ESTIMATION OF MIXTURE OF ACIDS BY CONDUCTOMETRY

Aim: To estimate the amount of acids present in the whole of the given solution. You are provided with standard 0.2 N sodium hydroxide solution.

Procedure:

Burette Solution : Standard NaOH

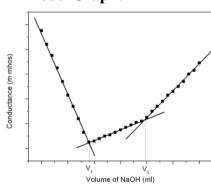
: 20ml of made up HCl solution + 20ml of distilled water Pipette Solution

End point : Obtained from graph

Model Table:

Volume of	Conductance
NaOH (ml)	(mMhos)

Model Graph:



Equivalent weight of HCl = 36.5Equivalent weight of $CH_3COOH = 60$

Result:

1.

The amount of HCl present in the whole of the given solution

$$= \frac{Normality\ of\ HCl \times 36.5 \times 100}{1000}\ grams$$

2.

The amount of CH₃COOH present in the whole of the given solution

$$= \frac{Normality \ of \ CH_3COOH \times 60 \times 100}{1000} \ grams$$

Ex. No. 2: ESTIMATION OF FERROUS ION BY POTENTIOMETRY

Aim: To estimate the amount of ferrous ions present in the whole of the given solution. You are provided with s standard 0.1 N potassium dichromate solution.

Procedure:

Burette Solution : Standard K₂Cr₂O₇

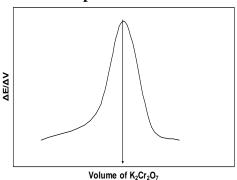
: 20ml of made up ferrous solution + 20ml dil. H₂SO₄ Pipette Solution

End Point : Obtained from the graph

Model Table:

Sl. No	Volume of K ₂ Cr ₂ O _{7,} V (ml)	Observed emf, E (mV)	ΔE (mV)	ΔV (ml)	$\Delta E / \Delta V$ (mV/ml)

Model Graph:



solution ×

Regulation: 2019

Result:

Strength of Fe²⁺ solution (N₂) V_1 (Obtained from graph) $\times 0.1$

20 Amount of Fe²⁺ ions present in 1000 ml of the Normality of Fe²⁺

solution (Y) Equivalent weight

 $N_2 \times 55.85$ Amount of Fe^{2+} ions present in 100 ml of the =

solution 10

Amount of Fe²⁺ present in the whole of the given solution

Ex. No. 3: DETERMINATION OF pKa VALUE OF GLYCINE BY pH METRY

Aim: To determine the titration curve of the given amino acid, glycine and to use this curve to estimate its pK_a values.

Procedure:

Titration I: Glycine vs. HCl

Burette solution: 0.1N HCl

Pipette solution: 10 ml of 0.1 N glycine + 20 ml distilled water

pKa value obtained from the graph.

Titration II: Glycine vs. NaOH

Burette solution: 0.1N NaOH

Pipette solution: 10 ml of 0.1 N glycine + 20 ml distilled water

pKa value obtained from the graph.

Model Tables:

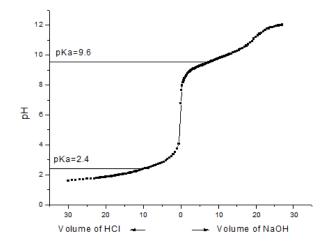
Glycine Vs HCl

	S.	Volume of	Observed
No.		0.1 N HCl	pН
1		0	
2		1	
3		2	

Glycine Vs NaOH

J					
S. No.	Volume of 0.1 N NaOH	Observed pH			
1	0				
2	1				
3	2				

Model Graph:



Result: The two pK_a values of glycine are _____ and __

Ex. No. 4: ESTIMATION OF IRON BY PHOTOCOLORIMETRY

Aim: To estimate the amount of Fe³⁺ ion present in the given water sample using spectrophotometer.

Procedure:

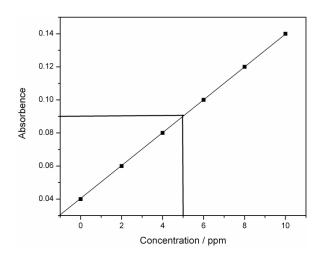
Preparation of standard iron solutions: 2 ppm, 4 ppm, 6 ppm, 8 ppm and 10 ppm Standard iron solution + 1 ml of 1:1 HCl + 1 ml of potassium thiocynate made up to 25 ml.

Model Table:

 λ = 480 nm. Distilled water Blank = zero absorbance

S.No	Concentration	Absorbance
1	2 ppm	
2	4 ppm	
3	6 ppm	
4	8 ppm	
5	10 ppm	
6	Unknown	

Model Graph:



Result: The amount of iron present in the given solution = _____ ppm.

Ex. No. 5: ESTIMATION OF TOTAL HARDNESS OF WATER

Aim: To estimate the total hardness of the given sample of water by EDTA method

Titration I: Standardization of EDTA solution

Burette : EDTA solution

Pipette solution : 20 ml of std. hard water + 5ml buffer solution

Indicator : Eriochrome Black T

End point : Colour change from wine red to steel blue

1 ml of EDTA = $\frac{20}{V_1}$ mg of CaCO₃ equivalent hardness

Titration II: Estimation of total hardness of water

Burette : Standardised EDTA solution

Pipette solution : 20ml water sample + 5ml buffer solution

Indicator : Eriochrome Black T

End point : Colour change from wine red to steel blue

Total hardness of given water sample = $1000 \times \frac{V_2}{V_1}$ ppm.

Result:

Total hardness of the given sample of water = ppm (parts of $CaCO_3$ per million parts of water).

Ex. No. 6: ESTIMATION OF PERCENTAGE OF COPPER IN AN ORE

Aim: To estimate the percentage of copper in the given ore. You are provided with a standard 0.5 N potassium dichromate solution and an approximately 0.05 N sodium thio sulphate solution.

Procedure:

Titration I: Standardization of sodium thiosulphate

Burette Solution : Sodium thiosulphate

Pipette solution : Std. potassium dichromate + 20ml dil.H₂SO₄ +10ml of 10% KI

Indicator : Starch

End Point : Disappearance of blue colour (Appearance of pale green colour)

Titration II: Estimation of copper

Burette Solution : Std. Sodium thiosulphate

Pipette solution : Copper ion solution +10ml of 10% KI

Indicator : Starch

End Point : Disappearance of blue color (Appearance of creamy white precipitate)

Result:

The amount of copper present in one liter of the solution = Normality \times 63.54

= x g/lit.

The amount of copper present in the whole of the given solution $=\frac{x\times100}{1000}$

= y g

The percentage of copper in the given ore $= \frac{y \times 100}{0.5}$

= _____9

Regulation: 2019

Ex. No. 7: DETERMINATION OF ALKALINITY OF WATER

Aim: To determine the types and amount of alkalinity in the given sample of water. You are provided with a solution of Na₂CO₃ of strength 0.01N and a link solution of HCl solution are provided.

Procedure:

Titration I: Standardization of HCl

Burette solution: 20 ml of std. Na₂CO₃

Pipette solution: HCl

Indicator: 2-3 drops of methyl orange End point: Yellow to pale pink

Titration II: Estimation of alkalinity of water sample

Burette solution: 20 ml of water sample

Pipette solution: HCl

Indicator 1: 2-3 drops of phenolphthalein

End point 1: Disappearance of pale pink colour (Phenolphthalein End Point: P)

Indicator 2: 2-3 drops of methyl orange

End point 2: Reapperance of pale pink colour (Methyl orange End Point: M)

Equivalent weight of $CaCO_3 = 50$

Result: The individual amounts of alkalinity present in the given water sample can be calculated by using following expressions:

(i) If P > 1/2 M, Water sample contains $OH + CO_3^{2-}$ alkalinity.

$$OH^- = \frac{(2P - M) \times Normality \ of \ HCl \times 50 \times 1000}{20} = -----ppm$$

$$CO_3^{2-} = \frac{2(M-P) \times Normality \ of \ HCl \times 50 \times 1000}{20} = -----ppm$$

(ii) If P < 1/2 M, Water sample contains $CO_3^{2-} + HCO_3^{-}$ alkalinity.

$$CO_3^{2-} = \frac{2P \times Normality \ of \ HCl \times 50 \times 1000}{20} = -----ppm$$

$$HCO_3^- = \frac{(M-2P) \times Normality \ of \ HCl \times 50 \times 1000}{20} = -----ppm$$

Ex. No. 8: DETERMINATION OF MOLECULAR WEIGHT OF A POLYMER BY **VISCOSITY METHOD**

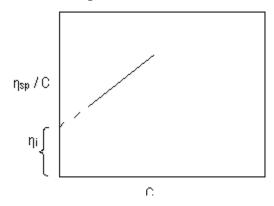
Aim: To determine the average molecular weight of given polymer by U-tube viscometer method and you are provided with a 5 wt% of polyvinyl alcohol solution.

Procedure: The flow time (t_0) for the pure solvent, distilled water is measured by pipetting out 10 ml into the u-tube viscometer. Prepare 0.25%, 0.5%, 0.75%, 1% and 1.25% solutions from the given polymer solution by using graduated pipette and 25 ml standard flask. 10 ml of each concentration is pipetted out into the u-viscometer and the flow time (t) for different concentration is noted.

Model table:

S. No.	Concentration of the polymer solution, (c)	Time of flow (t)	$\eta/\eta_0 = t/t_0$	$\eta_{sp} = (t/t_0) - 1$	η_{SP} / c
	Distilled water	(t_{o})			
	0.25%				
	0.5%				
	0.75%				
	1.0%				
	1.25%				

Model Graph:



Calculation:

The molecular weight of the given polymer is calculated by using expression: $\eta_i = KM^{\alpha}$

$$\eta_i = KM^{\alpha}$$

$$M = anti \log of \left[\frac{\log \eta_i - (-3.3437)}{0.64} \right]$$

Result:

The molecular weight of the given polymer sample =