# K.S. INSTITUTE OF TECHNOLOGY Bangalore - 109 



## DEPARTMENT OF CHEMISTRY

## ENGINEERING CHEMISTRY

## LABORATORY MANUAL

SUBJECT CODE: 18CHEL 16/26
NAME : $\qquad$
USN NO $\qquad$
SEMESTER : $\qquad$
BRANCH $\qquad$
SECTION $\qquad$
BATCH NO :

## SAFETY RULES

1. DO NOT perform unauthorized experiments or work in a laboratory alone
2. Approved eye protection must be worn at all times in the laboratory
3. Long hair and loose clothing must be confined while in a laboratory
4. Appropriate clothing must be worn at all times while in the laboratory
5. Closed shoes with socks must be worn at ALL times
6. Know the location and proper use of fire extinguishers, fire blankets, safety showers and first aid kits.
7. Before obtaining any chemicals carefully read the label on the reagent bottles.
8. Eating and drinking are not allowed in a chemistry laboratory
9. Thoroughly wash your hands after leaving the laboratory
10. Never direct the open end of test tube toward yourself or anyone else
11. Never pour water into concentrated acid
12. Liquid and solid waste containers must be properly used at all times
13. All personal belongings should be placed in the bookcases as you enter the laboratory.
14. Clean up any spill immediately

## K.S. Institute of Technology Department of

## Chemistry

## Institute Vision

To strive continuously to impart quality technical education with ethical values, employable skills and research of global standards

## Institute Mission

- To attract and retain highly qualified, experienced and committed Faculty.
- To create relevant infrastructure of global standard.
- Network with industry and premier institutions to encourage emergence of new ideas by providing Research and Development Facilities to achieve excellence.
- To inculcate the professional and ethical values among young students so that; they utilize the knowledge and skills acquired in transforming society.


## INSTRUCTIONS TO CANDIDATES

## FOR CONDUCTING VOLUMETRIC ANALYSIS

$\checkmark$ Wash all the apparatus with tap water once.
$\checkmark$ Wash all the apparatus in deionised water once.
$\checkmark$ Prepare the solution in volumetric flask and mix well.
$\checkmark$ Rinse the burette with the solution prepared in volumetric flask.
$\checkmark$ Fill the burette with the same solution up to zero mark.
$\checkmark$ Rinse the pipette with the solution to be pipetted.
$\checkmark$ Pipette out the given solution in the bottle and release into conical flask.
$\checkmark$ Never rinse the conical flask with any solution.
$\checkmark$ Conduct the titration by adding other reagents to be added and indicator.

## Engineering Chemistry Laboratory

Subject Code : 18CHEL16/18CHEL26 Marks :40
Hours : 03 Exam. Hours :03

Total Hours : 50
Exam. Hours : 03
Exam. Marks : 100

## Course Objectives:

To provide students with practical knowledge of quantitative analysis of materials by classical and instrumental methods for developing experimental skills in building technical competence

| No. of <br> Experiments | Molumetric Experiments | Marks |
| :--- | :--- | :--- | :--- |
| 1. | Introduction and Determination of COD of wastewater |  |
| 2. | Determination of percentage of Copper in brass solution using standard sodium thiosulphate solution. |  |
| 3. | Estimation of Iron in haematite ore solution using standard $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ solution by external indicator <br> method. |  |
| 4. | Estimation of Total hardness of water by EDTA complexometric method, |  |
| 5. | Estimation of CaO in cement solution by rapid EDTA method. |  |
| 7. | Estimation of FAS potentiometrically using standard $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ solution. |  |
| 8. | Estimation of Acids in acid mixture conductometrically. |  |
| 9. | Estimation of Copper colorimetrically. |  |
| 10. | Estimation of pKa value of weak acid by using pH meter. |  |
| 11. | Determination of Viscosity co-efficient of the liquid using Ostwald's viscometer. |  |
| 12. | Estimation of Sodium in the given sample of water using Flame Photometer. |  |


| 18CHEL16/26 | Course Outcome | Bloom's <br> Cognitive <br> Level |
| :---: | :--- | :--- |
| CO1 | Estimate the amount of analyte present in the solution using the principles of electro <br> analytical techniques (pH Meter, Condutometer, Potentiometer, Flame Photometry <br> and Photoelectric colorimeter) | Apply |
| $\mathbf{C O 2}$ | Determine the viscosity coefficient of liquid using Ostwald's Viscometer | Apply |
| $\mathbf{C O 3}$ | Estimate the amount of Cao in cement and Total Hardness of water by <br> Complexometric Titration | Apply |
| $\mathbf{C O 4}$ | Estimate the \% of copper in brass by Iodometric Titration. | Apply |
| $\mathbf{C O 5}$ | Estimate the amount of Iron in hematite ore and C.O.D. in waste water by redox <br> titration and estimate the percentage of chlorine in bleaching powder by iodometric <br> titration. |  |

K. S. INSTITUTE OF TECHNOLOGY

DEPARTMENT OF CHEMISTRY
LABORATORY INTERNAL ASSESSMENT

Name of the Student :
Sem \& Sec :
Subject Name: ENGINEERING CHEMISTRY LAB
Subject Code :18CHEL16/26

| $\begin{aligned} & \text { SL. } \\ & \text { NO } \end{aligned}$ | NAME OF THE EXPERIMENTS | $\begin{aligned} & \text { OBSERVAT } \\ & \text { ION BOOK } \\ & (10 \mathrm{M}) \end{aligned}$ | $\begin{aligned} & \text { VIVA } \\ & (10 \mathrm{M}) \end{aligned}$ | $\begin{aligned} & \text { RECO } \\ & \text { RD } \\ & \text { (10) } \end{aligned}$ | $\begin{aligned} & \text { TOTAL } \\ & \text { (30) } \end{aligned}$ |
| :---: | :---: | :---: | :---: | :---: | :---: |
| 1 | DETERMINATION OF COD IN WASTE WATER SAMPLE |  |  |  |  |
| 2 | ESTIMATION OF COPPER IN BRASS SOLUTION |  |  |  |  |
| 3 | ESTIMATION OF IRON IN HAEMATITE ORE SAMPLE |  |  |  |  |
| 4 | ESTIMATION OF TOTAL HARDNESS OF WATER IN THE GIVEN WATER SAMPLE |  |  |  |  |
| 5 | ESTIMATION OF CALCIUM OXIDE IN CEMENT SOLUTION |  |  |  |  |
| 6 | DETERMINATION OF CHLORINE IN THE GIVEN BLEACHING POWDER |  |  |  |  |
| 7 | POTENTIOMETRIC TITRATION OF FAS VS K2Cr2O7 |  |  |  |  |
| 8 | CONDUCTOMETRIC ESTIMATION OF STRENGTH OF MIXTURE OF ACIDS |  |  |  |  |
| 9 | COLORIMETRIC ESTIMATION OF COPPER |  |  |  |  |
| 10 | DETERMINATION OF PkaVALUE OF WEAK ACID |  |  |  |  |
| 11 | DETERMINATION OF VISCOSITY CO-EFFICIENT of given LIQUID |  |  |  |  |
| 12 | ESTIMATION OF SODIUM IN GIVEN SAMPLE BY FLAME PHOTOMETRY |  |  |  |  |

Lab Internal Assessment Test (10)

WRITE UP OF THE EXPERIMENT (3) + EXPEIMENT CONDUCTION (5) + VIVA (2)
$=$ TOTAL (10)

## RUBRICS FOR LABORATORY EVALUATION

|  | Exceptional(10-9) | Acceptable(8-6) | Marginal(5-1) | Unsatisfactory(0) |
| :--- | :--- | :--- | :--- | :--- |
| Record (10) | The Record is well <br> written and clearly <br> explains what the <br> experiments are <br> accomplishing | The record is <br> written and useful <br> in understanding | The record is <br> vague only the <br> reader can <br> understand. | The record does not <br> help the reader <br> understand |
| Experiments <br> conduction(In the <br> Observation book)(10) | The experiments <br> executes and meets <br> all of the <br> specifications. | The experiments <br> executes correctly <br> with no errors and <br> meets few of the <br> specifications. | The experiments <br> executes with few <br> result errors | experiments does <br> not execute |
| Viva(10) | Demonstrates deep <br> knowledge; answer <br> the questions with <br> explanations and <br> elaboration. | Adequate <br> knowledge of most <br> topics; answer the <br> questions, but fails <br> to elaborate. | Superficial <br> knowledge of <br> topic; only able to <br> answer <br> basic questions. | Not able to answer <br> basic questions |

## Record (10) + Experiments conduction (In the Observation book)(10) + Viva(10) + Internal Test (10)= TOTAL (40)

## Observation and Calculations:

## Part-A:

Weight of the bottle + Mohr's salt $=\mathrm{W}_{1}=$ $\qquad$ g .

Weight of the empty bottle
$=\mathrm{W}_{2}=$ $\qquad$ g.

Weight of the Mohr Salt
$=\left(\mathrm{W}_{1}-\mathrm{W}_{2}\right)=$ $\qquad$

$$
\mathrm{N}_{\mathrm{FAS}}=\frac{\left(\mathrm{W}_{1}-\mathrm{W}_{2}\right) \times 4}{\text { Eq. wt.of } \mathrm{FAS}(392)}
$$

$$
=\text {. }
$$

$\qquad$ N

## Part-B:

Sample titration

| Burette <br> Conical flask | $: \quad$ Standard FAS |  |
| :--- | :--- | :--- |
|  | 25.0 ml of waste water sample +10.0 ml of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}+$ |  |
|  |  |  |
| Indicator | $:$ | $2-3$ of $1: 1 \mathrm{H}_{2} \mathrm{SO}_{4}$ |

$$
\text { Regular } \quad \text { Repetition }
$$

| Burette <br> Reading in ml | I | II | III | IV | V | VI |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| Final burette <br> Reading |  |  |  |  |  |  |
| Initial burette <br> Reading |  |  |  |  |  |  |
| Volume of <br> FAS <br> consumed |  |  |  |  |  |  |

Concordant Value $\mathrm{V}_{1}=-----------\mathrm{ml}$.

## DETERMINATION OF C.O.D IN WASTE WATER

Aim: To estimate the chemical oxygen demand in wastewater.
Principle: Chemical oxygen demand parameter has been introduced to measure the total oxidisable impurities present in the sewage. This includes both, biologically oxidisable and biologically inert but chemically oxidisable impurities in the water. It is the amount of oxygen equivalents of acidified potassium dichromate required to oxidize the organic and inorganic impurities in waste water. It is expressed in $\mathrm{mg} . \mathrm{dm}^{-3} \mathrm{or} \mathrm{ppm}$. A known volume of the wastewater sample is refluxed with excess of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ solution in sulphuric acid medium and in the presence of $\mathrm{AgSO}_{4}$ and $\mathrm{HgSO}_{4} . \mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ oxidizes all oxidisable impurities. $\mathrm{AgSO}_{4}$ catalyses the oxidation of straight chain organic compounds, aromatics and pyridine. $\mathrm{HgSO}_{4}$ avoids the interference of $\mathrm{Cl}^{-}$ions by forming soluble complex with them. In the absence of $\mathrm{HgSO}_{4}, \mathrm{Cl}^{-}$ions precipitate silver ions as AgCl . The amount of unreacted $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ is determined by titration with standard Mohr's salt (ferrous ammonium sulphate) solution. The amount of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ solution consumed corresponds to the COD of the water sample. To calculate the COD, a blank titration (without the water sample) is carried out.

Ferroin [(1, 10-phenanthroline-iron (II)] complex is used as the indicator in the titration to detect the end point. The indicator is intensely red in colour. Strong oxidizing agents oxidize the indicator to iron (III) complex, which has a bluish green colour.

$$
\begin{gathered}
{\left[\mathrm{Fe}\left(\mathrm{C}_{12} \mathrm{H}_{8} \mathrm{~N}_{2}\right)_{3}\right]^{3+}+\mathrm{e}^{-} \leftrightarrow \leftrightarrow \quad\left[\mathrm{Fe}\left(\mathrm{C}_{12} \mathrm{H}_{8} \mathrm{~N}_{2}\right)_{3}\right]^{2+}} \\
\text { Bluish greep red }
\end{gathered}
$$

As long as $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ solution is present in the solution, the indicator is in the oxidized form, imparting bluish green colour to the solution. The solution becomes bluish green as the titration (with FAS) proceeds (due to the presence of untreated $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ ) and when $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ is completely exhausted, the added FAS solution which is in oxidizing agents impart red-brown colour to the solution. Therefore, the end point is marked by the colour change from bluish-green to reddish-brown. Addition of con. $\mathrm{H}_{2} \mathrm{SO}_{4}$ to $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ facilitates the liberation of nascent oxygen required for oxidation of waste water. A test tube of dil. $\mathrm{H}_{2} \mathrm{SO}_{4}$ is added during dissolution of Mohr's salt (standard solution) to prevent the hydrolysis of the salt in the solution.
The reaction between Mohr's salt and $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ can be represented as follows.
$\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}+4 \mathrm{H}_{2} \mathrm{SO}_{4} \rightarrow \mathrm{~K}_{2} \mathrm{SO}_{4} \mathrm{Cr}_{2}\left(\mathrm{SO}_{4}\right)_{3}+4 \mathrm{H}_{2} \mathrm{O}+3(\mathrm{O})$
$\left.2 \mathrm{FeSO}_{4}+\mathrm{H}_{2} \mathrm{SO}_{4}+(\mathrm{O}) \rightarrow \mathrm{Fe}_{2}\left(\mathrm{SO}_{4}\right)_{3}+\mathrm{H}_{2} \mathrm{O}\right] \times 3$
$\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7+} 6 \mathrm{FeSO}_{4}+7 \mathrm{H}_{2} \mathrm{SO}_{4} \rightarrow \mathrm{~K}_{2} \mathrm{SO}_{4}+\mathrm{Cr}_{2}\left(\mathrm{SO}_{4}\right)_{3}+3 \mathrm{Fe}_{2}\left(\mathrm{SO}_{4}\right)_{3}+7 \mathrm{H}_{2} \mathrm{O}$

## Blank titration

Burette : Standard FAS
Conical flask : 10.0 ml of standard $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}+1: 1$ Sulphuric acid Indicator : Ferroin.
Colour change: Bluish green to reddish brown.

| Burette <br> Reading in ml | I |
| :--- | :---: |
| Final burette <br> reading |  |
| Initial burette <br> reading |  |
| Volume of FAS <br> consumed |  |

$$
(\mathrm{NV})_{\text {FAS }}=(\mathrm{N} V)_{\text {Oxygenfor wastewater }}
$$

$$
\mathrm{N}_{\text {Oxygenfor wastewater }}=\frac{\mathrm{N}_{\mathrm{FAS}} \mathrm{~V}_{\mathrm{FAS}}}{\mathrm{~V}_{\text {wastewater }}}\left(\mathrm{V}_{\mathrm{FAS}}=\mathrm{V}_{\text {blanktitre value }}-\mathrm{V}_{\text {sampletitre value }}\right)
$$

$$
=\text {. }
$$

$\qquad$

Wt. of oxygen / litre of wastewater $=\mathrm{N}_{\text {oxygenfor wastewater }} \times$ Eq. wt of oxy gen (8)
$\qquad$

Wt. of oxygen in $\mathrm{mg} /$ liter of waste water $=\mathrm{a} \times 1000$

Result: $\operatorname{COD}$ of given waste water $=$ $\qquad$ mg /litre.

Signature of staff member with date: $\qquad$ .

## Procedure:

## Part-A: Preparation of standard Mohr's salt solution:

Weigh accurately the given Mohr's salt, transfer it in to a 250 ml standard flask, dissolve in a little amount of distilled water and a t.t of dilute $\mathrm{H}_{2} \mathrm{SO}_{4}$ and make up to the mark with distilled water. Shake well for uniform concentration.

## Part-B: Determination of COD:

Pipette out 25.0 ml of the wastewater sample in to a 250 ml conical flask. Pipette out 10.0 ml of $0.1 \mathrm{~N} \mathrm{~K} \mathrm{~K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ solution into the flask and add 10 ml of $1: 1 \mathrm{H}_{2} \mathrm{SO}_{4}$ (containing silver sulphate and $\mathrm{HgSO}_{4}$ ) with constant shaking of the flask. Add 2-3 drops of Ferroin indicator and titrate against standard Mohr's salt solution till the colour changes from bluish green to reddish brown.

## Blank Titration:

Repeat the above procedure by taking 25 ml -distilled water in the place of waste water sample

Result: COD of the given sample of water is $\qquad$ $\mathrm{mg} / \mathrm{dm}^{3}$

## Viva Voce

1) Define C.O.D.
2) What is the significance of this experiment?
3) What is the role of $\mathrm{AgSO}_{4}$ and $\mathrm{HgSO}_{4}$ ?
4) Why is dilute $\mathrm{H}_{2} \mathrm{SO}_{4}$ be added to $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ during titration?
5) Explain the colour change at the end point?
6) Why is dilute $\mathrm{H}_{2} \mathrm{SO}_{4}$ added in the preparation of standard solution of FAS?
7) What are the advantages of COD over BOD?
8) Why should $\mathrm{H}_{2} \mathrm{SO}_{4}$ be added to $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ during titration?
9) Give the compostion of ferroin indicator?
10) Expand FAS? Give the chemical nature of FAS?
11) What is the blank titration? What is its purpose?
12) Why this estimation also called back-titration?
13) How is COD expressed?

## Observation and Calculations:

| Volume of Brass solution $=$ |  |
| :--- | :--- |

Burette : Standard sodium thiosulphate solution.

| Conical flask | 25 ml .of Brass solution $+\mathrm{NH}_{4} \mathrm{OH}$ (drop wise till bluish white ppt.) $+\mathrm{CH}_{3} \mathrm{COOH}$ (to dissolve the ppt. and 2-3 drops excess) + 10 ml of KI |
| :---: | :---: |
| Indicator | Starch (near the end point) |
| Colour change: | Deep blue to milky white. |


| Regular |  |  |  |  |
| :--- | :---: | :---: | :---: | :---: |
| Volume of Brass solution <br> (ml.) |  |  |  |  |
| Burette reading in ml | I | II | III | IV |
| Final burette reading |  |  |  |  |
| Initial burette reading |  |  |  |  |
| Vol. of $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ |  |  |  |  |

Any one of the appropriate titre value (say ' $b$ ' ml ) is to be considered for calculations.

Normality of Sodium thiosulphate used $=$ $\qquad$ N (say ' $a$ ')

## ESTIMATION OF COPPER IN BRASS

Aim: To estimate the percentage of copper in the given sample of brass.
Principle: Brass is an alloy of copper and zinc. When dissolved in conc. nitric acid, both the metals get converted into their nitrates.

$$
\mathrm{Cu}+4 \mathrm{HNO}_{3} \rightarrow \mathrm{Cu}\left(\mathrm{NO}_{3}\right)_{2}+2 \mathrm{H}_{2} \mathrm{O}+2 \mathrm{NO}_{2} \uparrow(\text { Brown fumes })
$$

When brass solution is treated with excess of potassium iodide solution, cupric ions oxidize potassium iodide to liberate equivalent quantity of iodine. During the liberation of $\mathrm{I}_{2}$ from KI, $\mathrm{Cu}^{2+}$ gets reduced to $\mathrm{Cu}^{+}$with a change in oxidation state by 1 .

$$
2 \mathrm{Cu}\left(\mathrm{NO}_{3}\right)_{2}+4 \mathrm{KI} \rightarrow \mathrm{Cu}_{2} \mathrm{I}_{2} \downarrow+4 \mathrm{KNO}_{3}+\mathrm{I}_{2}
$$

The liberated iodine is titrated with sodium thiosulphate solution using starch as indicator.

$$
2 \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}+\mathrm{I}_{2} \rightarrow \mathrm{Na}_{2} \mathrm{~S}_{4} \mathrm{O}_{6}+2 \mathrm{NaI}
$$

(Sodium tetrathionate)
The oxides of nitrogen present in the brass solution are destroyed by adding urea. The presence of oxides of nitrogen if not destroyed will be responsible for the liberation of extra iodine from KI, as they are also good oxidizing agents, thus increasing the titre value

The excess nitric acid present in the brass solution is neutralized by adding $\mathrm{NH}_{4} \mathrm{OH}$ solution till a pale blue precipitate of $\mathrm{Cu}(\mathrm{OH})_{2}$ is obtained. Otherwise, being an oxidizing agent, nitric acid may also liberate iodine from KI. The $\mathrm{Cu}(\mathrm{OH})_{2}$ precipitate is dissolved with dilute acetic acid. Other mineral acids are not preferable, as they will bring down the pH to a very small value, at which the liberation of $\mathrm{I}_{2}$ from KI by $\mathrm{Cu}^{2+}$ is not quantitative and the pH of experiment is hence maintained at around 5

As it is titrated with $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$, free Iodine reacts with it. Near the end point, when almost free iodine is exhausted in the solution, starch is added. Starch reacts with little iodine to form a blue coloured complex. Further addition of sodium thiosulphate dissociates the starch-iodine complex, consumes iodine and liberates starch, thereby discharging the blue colour. For brass solution, the colour change is from blue to milky white as $\mathrm{Cu}_{2} \mathrm{I}_{2}$ precipitate is already present in the solution.

Starch indicator is added near the end point and not in the beginning for the following reasons. 1) At high concentration of Iodine, starch may form an insoluble starch- $\mathrm{I}_{2}$ complex. 2) The iodine also gets adsorbed on colloidal starch particles in the starch solution. The adsorbed iodine cannot be liberated by the addition of sodium thiosulphate, and the amount of iodine getting adsorbed is more when iodine concentration is high. Thus the error is minimized by adding starch near the end point.

1000 ml of $1 \mathrm{~N} \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ solution $=63.54 \mathrm{~g}$ of Cu
$b \mathrm{ml}$ of $\mathrm{aN} \mathrm{Na} \mathrm{N}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ solution $=\frac{b \times a \times 63.54}{1000} \mathrm{~g}$ of $\mathrm{Cu}\left(\mathrm{say}^{\prime} \mathrm{c}^{\prime}\right)$

$$
\% \text { of } C u=\frac{c \times 100}{W} \quad(\mathbf{W}=1 \mathbf{g m} .)
$$

Result: Percentage of Cu in Brass sample $=$ $\qquad$ $\%$

Signature of staff member with date: $\qquad$ .

## Procedure:

Given amount of brass solution taken in 250 ml . standard flask and make upto thr mark with distilled water. pipette out 25 ml . of brass solution in conical flask, add $\mathrm{NH}_{4} \mathrm{OH}$ drop wise until a bluish white precipitate persists. Add acetic acid dropwise till bluish white precipitate disappears then add 2drops of acetic acid in excess, followed by 10 ml of $10 \%$ KI. Titrate the liberated iodine against sodium thiosulphate solution taken in the burette until the colour of the solution changes to pale yellow. Then add starch solution till solution becomes blue in colour, and continue the titration till the blue colour changes to milky white. Repeat the titration for concordant values.

Result: Percentage of copper in the given sample of brass is

## Viva Voce

1) What are the constituents of brass?
2) What is an alloy?
3) Why is urea added in the determination?
4) What is the function of $\mathrm{NH}_{4} \mathrm{OH}$ ?
5) Why is $\mathrm{CH}_{3} \mathrm{COOH}$ added to the brass solution?
6) On adding KI to brass solution, brownish - yellow precipitate is obtained, what is it due to?
7) Why is starch added towards the end of the titration?
8) What is the only principle of iodometric titration?
9) Why should only freshly prepared starch be used and explain its role?
10) What is that white precipitate that is left behind after the end point?
11) How is iodine estimated related to copper present in brass sample?
12) How is iodine retained in the reaction mixture though it is a gas?

## Observation and Calculations:

## Part- A:

Weight of the bottle $+\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ crystals $=\mathrm{W}_{1}=$ g

Weight of the empty bottle
Weight of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ crystals
$=\mathrm{W}_{2}=$ g $=\left(\mathrm{W}_{1}-\mathrm{W}_{2}\right)=$ g.

$$
\mathrm{N}_{\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}}=\frac{\left(\mathrm{W}_{1}-\mathrm{W}_{2}\right) \times 4}{\text { Eq. wt. of } \mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}(49)}
$$

= -------------------N

Part-B:
Burette : Standard $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$
Conical flask : $\quad 25.0 \mathrm{ml}$ of haematite ore solution +5 ml of $4 \mathrm{~N} \mathrm{HCl}+$ boil + $\mathrm{SnCl}_{2}$ Drop wise till solution becomes colorless, 2 drops in excess + cool +5 ml of $\mathrm{HgCl}_{2}+2 \mathrm{t}$ t. of water

Indicator : Potassium ferricyanide [external]
Colour change: Disappearance of blue colour
Regular Repetition

| Burette <br> Reading in ml | I | II | III | IV | V | VI |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| Final burette <br> Reading |  |  |  |  |  |  |
| Initial burette <br> Reading |  |  |  |  |  |  |
| $\mathbf{V o l u m e ~ o f ~}_{\mathbf{K}_{\mathbf{2}} \mathbf{C r}_{\mathbf{2}} \mathbf{O}_{7}}$ |  |  |  |  |  |  |

Concordant Value $\mathrm{V}_{1}=-----------\mathrm{ml}$.

## ESTIMATION OF IRON IN HAEMATITE

Aim: To estimate the percentage of iron in haematite ore solution.
Principle: Haematite is an oxide ore of iron, existing as ferric oxide $\left(\mathrm{Fe}_{2} \mathrm{O}_{3}\right)$. On treating with HCl , ferric oxide dissolves in the acid as ferric chloride.

$$
\mathrm{Fe}_{2} \mathrm{O}_{3}+6 \mathrm{HCl} \rightarrow 2 \mathrm{FeCl}_{3}+3 \mathrm{H}_{2} \mathrm{O}
$$

Since iron in $\mathrm{FeCl}_{3}$ is already in the highest possible stable oxidation state $\left(\mathrm{Fe}^{3+}\right)$, it cannot be oxidized further by potassium dichromate solution, which is also an oxidizing agent.. Therefore, it is first reduced to ferrous state and then titrated with potassium dichromate. Ferric chloride is reduced by stannous chloride in presence of hydrochloric acid.

$$
\begin{aligned}
& 2 \mathrm{FeCl}_{3}+\mathrm{SnCl}_{2} \rightarrow 2 \mathrm{FeCl}_{2}+\mathrm{SnCl}_{4} \\
& \mathrm{~K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}+8 \mathrm{HCl} \rightarrow 2 \mathrm{KCl}+2 \mathrm{CrCl}_{3}+4 \mathrm{H}_{2} \mathrm{O}+3(\mathrm{O}) \\
& {\left[2 \mathrm{FeCl}_{2}+2 \mathrm{HCl}+(\mathrm{O}) \rightarrow 2 \mathrm{FeCl}_{3}+\mathrm{H}_{2} \mathrm{O}\right] \times 3} \\
& \mathrm{~K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}+14 \mathrm{HCl}+6 \mathrm{FeCl}_{2} \rightarrow 2 \mathrm{KCl}+2 \mathrm{CrCl}_{3}+6 \mathrm{FeCl}_{3}+7 \mathrm{H}_{2} \mathrm{O}
\end{aligned}
$$

From the equation, 2 moles of $\mathrm{FeCl}_{2}=2$ moles of $\mathrm{FeCl}_{3}=1$ atom of oxygen
A slight excess of stannous chloride is added to ensure complete reduction of $\mathrm{FeCl}_{3}$ to $\mathrm{FeCl}_{2}$. The excess of $\mathrm{SnCl}_{2}$ added is destroyed by adding strong solution of mercuric chloride, $\mathrm{SnCl}_{2}$ is a good reducing agent and can react with potassium dichromate (oxidizing agent) if excess $\mathrm{SnCl}_{2}$ is not neutralized.

$$
\mathrm{SnCl}_{2}+2 \mathrm{HgCl}_{2} \rightarrow \mathrm{SnCl}_{4}+\underset{\text { (Mercurous chloride) }}{\mathrm{Sg}_{2} \mathrm{Cl}_{2} \downarrow(\text { silky white) }}
$$

A black precipitate of finely divided mercury may be produced if too much $\mathrm{SnCl}_{2}$ is present (sufficient mercuric chloride solution should be added at once). The finely divided mercury (if formed) reduces $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$. Therefore the trial must be discarded if a black precipitate is produced. The trial should also be discarded if no precipitate is obtained on addition of mercuric chloride because the complete reduction of ferric ions to ferrous ions is not ensured.

Potassium ferricyanide is used as an external indicator to detect the end point of the titration. Potassium ferricyanide reacts with ferrous ions to produce an intense deep color due to the formation of ferrous ferri cyanide complex.

$$
2 \mathrm{~K}_{3}\left[\mathrm{Fe}(\mathrm{CN})_{6}\right]+3 \mathrm{Fe}^{2+} \rightarrow \mathrm{Fe}_{3}\left[\mathrm{Fe}(\mathrm{CN})_{6}\right]_{2}+6 \mathrm{~K}^{+}
$$

Ferrous ferricyanide (Blue)
Therefore when a drop of titrated solution is treated with a drop of potassium ferricyanide solution, blue colour is developed as long as ferrous ions are present in the solution. At the end point when ferrous ions are completely converted into ferric ions, a drop of the titrated solution does not develop blue colour with the indicator.

Weight of the Haematite ore given in $250 \mathrm{ml}=\mathrm{Wg}=4.128 \mathrm{~g}$

$$
\begin{aligned}
& (\mathrm{NV})_{\mathrm{K}_{2} \mathrm{C}_{2} \mathrm{O}_{7}}=(\mathrm{NV})_{\mathrm{Fe} \text { in hematite ore }} \\
& \mathrm{N}_{\mathrm{Fe}}=\frac{(\mathrm{NV})_{\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}}}{\mathrm{~V}_{\mathrm{Fe}}}
\end{aligned}
$$

$$
\begin{aligned}
& =\ldots \ldots \ldots \ldots \ldots \ldots \ldots . \mathrm{N} \\
& \text { Wt of Fe lliter of ore }=N_{F e} \times \text { atomic weight of } \mathrm{Fe} \text { (55.85) }
\end{aligned}
$$

Result: Percentage of Iron in given Haematite ore $=$
$\qquad$ .

## Procedure:

## Part-A: Preparation of standard $\mathrm{K}_{\mathbf{2}} \mathrm{Cr}_{\mathbf{2}} \mathrm{O}_{\mathbf{7}}$ solution

Weigh accurately about 1.0 g of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ crystals into a 250 ml standard flask. Dissolve the salt in little distilled water. Make up the solution to the mark and shake well for uniform concentration.

## Part-B: Estimation of iron:

Pipette out 25.0 ml of haematite solution into a clean conical flask. Add 5 ml of 4 N HCl and heat to boiling. To the hot solution, add $\mathrm{SnCl}_{2}$ solution drop wise from a burette till the yellow color is discharged completely. Add two drops in excess. Cool the solution and add 5 ml of $\mathrm{HgCl}_{2}$ solution at a stretch and shake well. A silky white precipitate should be obtained. (If black precipitate or no precipitate is obtained, discard the solution). Add two test tubes of distilled water.
Place a number of drops of freshly prepared potassium ferricyanide solution on a wax paper. Add 1 ml of potassium dichromate solution from the burette into the conical flask and shake well. With the help of a thin glass rod take a drop of the titrated solution and bring it in contact with an indicator drop. The colour of the indicator drop turns blue. Likewise after each addition of $1 \mathrm{ml} \mathrm{K} 2_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ solution, shake the solution well and test the solution drops as before. Repeat this till a drop of the titrated solution does not develop blue color with the indicator. This titration gives the range of the end point.

Again pipette out 25.0 ml of the haematite solution into a conical flask. Treat with $\mathrm{SnCl}_{2}$ and $\mathrm{HgCl}_{2}$ as before. Add 2 test tubes of distilled water. Now add most of the potassium dichromate solution required at a stretch and further titrate drop wise testing as explained earlier, till no blue color is developed with the indicator. Repeat for agreeing values.
Result: Percentage of Iron in given Haematite ore is

## Viva Voce

1) What is the main constituent of iron ore?
2) Why is it necessary to add very small excess of $\mathrm{SnCl}_{2}$ to the hot ore solution?
3) What happens if the $\mathrm{HgCl}_{2}$ solution is added?a) When the solution is still hot.
b) Drop by drop or insufficient amount
4) What is meant by internal indicator, external indicator \& self-indicator? Give one example of each?
5) Why is $\mathrm{K}_{3}\left(\mathrm{Fe}(\mathrm{CN})_{6}\right)$ not be used as internal indicator?
6) Describe the detection of the end point with this indicator?
7) Why has the external indicator method been largely replaced with internal indicator methods?
8) What is the color of indicator with $\mathrm{Fe}^{2+}$ ions? What is it due to?
9) Explain the colour change at the end point?
10) Give the chemical nature of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ and $\mathrm{SnCl}_{2}$
11) What is the silky white ppt. due to?

## Part B:

| Burette | $:$ Standard EDTA solution. |
| :--- | :--- |
| Conical flask | $: 25.0 \mathrm{ml}$ of hard water $+2 \mathrm{ml} \mathrm{NH}_{4} \mathrm{OH}-\mathrm{NH}_{4} \mathrm{Cl}$ buffer |
| Indicator | $:$ A pinch of Eriochrome Black T |
| Colour change | $:$ Wine red to blue |

Regular
Repetition

| Burette <br> Reading in ml | I | II | III | IV | V | VI |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| Final burette <br> Reading |  |  |  |  |  |  |
| Initial burette <br> Reading |  |  |  |  |  |  |
| Volume of <br> EDTA in cm |  |  |  |  |  |  |

Concordant Value $\mathrm{V}_{\text {EDTA }}=-----------\mathrm{ml}$.

## ESTIMATION OF HARDNESS OF WATER

Aim: To estimate the total hardness of the given water sample.
Principle: Hardness of water is due to the presence of dissolved salts of calcium, magnesium and other metal ions. Hard water does not give lather easily with soap, as the ions causing hardness react with soap to form insoluble soap. Temporary hardness is due to bicarbonates of calcium and magnesium, which can be removed by boiling. On boiling, the bicarbonates are converted into insoluble carbonates of calcium and magnesium, which can be removed by chemical treatment. The sum of temporary hardness and permanent hardness is known as total hardness. The hardness is expressed in ppm equivalents of calcium carbonate or $\mathrm{mg} / \mathrm{l}$.

Hardness is determined by titrating a known volume of water sample with EDTA, using Eriochrome Black T (EBT) as indicator, in presence of ammonium hydroxideammonium chloride buffer ( pH 10 ). Ethylene Diamine Tetra Acetic acid (EDTA) is slightly soluble in water. Hence its disodium salt, which is very much soluble in water, is generally used. EDTA reacts quantitatively with the metal ions to form a soluble, and stable complex.

When a few drops of the indicator EBT are added, it forms a wine red coloured complex with the metal ions.
$\mathrm{M}^{2+}+\mathrm{EBT} \rightarrow \mathrm{M}-\mathrm{EBT}$ complex (winered) $\left(\mathrm{M}^{2+}=\mathrm{Ca}^{2+}\right.$ or $\left.\mathrm{Mg}^{2+}\right)$
During titration, EDTA reacts, preferentially with free metal ions present in the solution. Near the end point, when the free metal ions are exhausted in the solution, further addition of EDTA dissociates the M-EBT complex, consumes the metal ions and releases free indicator, which is blue in colour. Therefore the colour change is wine red to blue. The pH of the solution is maintained around 10 using $\mathrm{NH}_{4} \mathrm{OH}-\mathrm{NH}_{4} \mathrm{Cl}$ buffer to maintain the neutrality of the reaction. Therefore the indicator EBT is suitably selected as it shows a colour change from the wine red to blue between $\mathrm{pH} 9-11$.

$$
\mathrm{M}_{\mathrm{CaCO}_{3}} \text { in hard water }=\frac{(\mathrm{MV})_{\text {EDTA }}}{\mathrm{V}_{\text {Hard water }}}
$$

$\qquad$

Weight of $\mathrm{CaCO}_{3} /$ litre of hardwater $=\mathrm{M}_{\mathrm{CaCO}_{3}} \times$ Molecular weight of $\mathrm{CaCO}_{3}$

$$
=\mathrm{M}_{\mathrm{CaCO}_{3}} \times 100
$$

= -------------------- g (say ‘a’)

Wt of $\mathrm{CaCO}_{3} /$ million $\mathrm{cm}^{3}=\frac{\mathrm{a} \times 10^{6}}{1000}$
$=$

Result: Total hardness of given water sample is $\qquad$ ppm of $\mathrm{CaCO}_{3}$.

Signature of Staff member with date: $\qquad$ .

## Procedure:

Part - A: Preparation of standard solution of EDTA: Weigh accurately the given amount of EDTA and transfer into a 250 ml standard flask. Dissolve completely with 5 ml of $\mathrm{NH}_{3}$ and sufficient amount of water. Make up the solution to the mark and shake well for uniform concentration.

Part- B: Estimation of Hardness of Water: Pipette out 25.0 ml of hard water in to a clean conical flask. Add 2 ml of buffer solution and a pinch of Eriochrome Black T indicator. Titrate against EDTA solution taken in the burette until the colour of the solution changes from wine red to blue. Repeat the titration for concordant values.

Result: Total hardness in the given sample of water $=$ $\qquad$ .ppm of $\mathrm{CaCO}_{3}$

## Viva Voce

1) What is hard water?
2) What is hardness of water due to?
3) What do you mean by Total hardness of water?
4) How is the total hardness of water expressed?
5) What is EDTA? Write the structure of it?
6) What is a buffer solution?
7) Why is ammonia-ammonium chloride buffer added?
8) How do you account for the change in the colour of solution at the end point?
9) What is the significance of hardness determination?
10) Why experiment is carried at pH 10 ?
11) Why is EDTA and EBT is specifically chosen as titrant and indicator respectively in the experiment?

## Observation and Calculations:

Part A


Weight of EDTA $=\left(W_{1}-W_{2}\right)=------------------$ g

$$
\mathrm{M}_{\text {EDTA }}=\frac{\text { Weight of EDTA } \times 4}{\text { Molecular weight of EDTA }(372.24)}
$$

$\qquad$

## Part B

Burette : Standard EDTA
Conical flask : 25.0 ml of cement solution +10 ml of glycerol and diethyl amine mixture +10 ml of NaOH Shake well for 3-5 min. +1 t.t. of distilled water.
Indicator : A pinch of Patton and Reeder's indicator
Colour change: Wine red to Blue.

| Burette <br> Reading in ml | I | II | III | IV | V | VI |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| Final burette <br> Reading |  |  |  |  |  |  |
| Initial burette <br> Reading |  |  |  |  |  |  |
| Volume of <br> EDTA (ml.) |  |  |  |  |  |  |
| Regular |  |  |  |  |  |  |

Concordant Value $\mathrm{V}_{1}=$ $\qquad$

## Date:

## ESTIMATION OF CALCIUM OXIDE IN CEMENT

Aim: To estimate calcium oxide in the given sample of cement solution.
Principle: Cement mainly contains silica and also compounds of calcium, aluminium, magnesium, and iron. When cement dissolved in acid, silica remains undissolved. On treating with ammonia, aluminum and iron can be precipitated as their hydroxides and separated. The provided solution of cement, therefore, contains calcium and magnesium ions.

To estimate the calcium content in the given solution, a known volume of it is titrated with standard EDTA solution. The interference of magnesium ions is avoided by precipitating them as hydroxide. This is done by adding 4 N NaOH and pH of the solution is maintained 12.5 by adding 5 ml of diethyl amine to the cement solution. The reaction mixture containing calcium can be titrated with EDTA by using Patton and Reeder's indicator . The indicator combines with calcium ions forming a pink coloured complex.

$$
\mathrm{Ca}^{2+}+\text { Ind } \rightarrow \mathrm{Ca}-\text { Ind Complex (Pink) }
$$

Near the end point, when free calcium ions are exhausted in the solution, further addition of EDTA dissociates Ca-Indicator complex, consumes the calcium ions and releases free indicator, which is blue in colour. Therefore, the colour change is pink to blue.
A sharper end point may be obtained by adding 5 ml of $1: 1$ glycerol. Glycerol reduces adsorption of the complex on the surface of the precipitate.

## Procedure:

## Part-A: Preparation of standard solution of EDTA:

Weigh accurately about 4.5 g of EDTA and transfer into a 250 ml standard flask. Dissolve it completely with 5 mL of $\mathrm{NH}_{3}$ and sufficient amount of water. Make up the solution to the mark and shake well for uniform concentration.

Part-B: Estimation of calcium oxide in cement solution:
Pipette out 25.0 ml of the cement solution into a clean conical flask. To the flask add 10 ml of glycerol and diethyl amine mixture. Then add 10 ml of 4 N NaOH solution. Mix the solution thoroughly for 3-5 minutes. Add a test tube of distilled water and a pinch of Patton and Reeder's indicator. Titrate against the EDTA solution taken in the burette till the colour changes from wine red to blue. Repeat for agreeing values.

Result: Percentage of CaO in Cement sample $=$ $\qquad$

$$
\begin{aligned}
& \text { Weight of the cement given in } 250 \mathrm{ml} \text { solution }=\mathrm{Wg}=1.28 \\
& (\mathrm{MV})_{\mathrm{EDTA}}=(\mathrm{MV})_{\mathrm{CaO} \text { in cement solution }} \\
& \mathrm{M}_{\mathrm{CaO}} \text { in cement }=\frac{\mathrm{MV}_{\mathrm{EDTA}}}{\mathrm{~V}_{\text {cement solution }}} \\
& =\ldots \ldots \ldots \ldots . . \text { M } \\
& \text { Wt. of } \mathrm{CaO} / l i \text { tre of cement solution. }=\mathrm{M}_{\mathrm{CaO}} \times \mathrm{Mol} \mathrm{Wt} \mathrm{of} \mathrm{CaO} \\
& =\mathrm{M}_{\mathrm{CaO}} \times 56 \\
& \text { = ---------------g. (say ‘a') } \\
& \text { Wt. of } \mathrm{CaO} / 250 \mathrm{ml} \text { of cement }=\frac{\mathrm{a}}{4} \\
& =\ldots \ldots \ldots \ldots . . \text { g (say 'b' }) \\
& \text { Percentage of } \mathrm{CaO} \text { in cement sample }=\frac{\mathrm{b} \times 100}{\mathrm{~W}}
\end{aligned}
$$

Result: Percentage of CaO in Cement sample $=$ $\qquad$ \%

Signature of Staff member with date:

## Viva Voce

1) What are the chief constituents of cement?
2) How is cement solution prepared?
3) Why is it that Eriochrome blackT indicator not used in this experiment why?
4) What is the purpose of adding glycerol, diethyl amine and NaOH to cement solution?
5) What is the significance of this experiment?
6) It is necessary to increase the pH of the experiment from 10 to 12 ? If so why? How it is increased?

## Observation and Calculations

Part A: Preparation of Bleaching powder solution
Part A: Preparation of standard $\mathrm{Na}_{2} \mathrm{~S}_{\mathbf{2}} \mathrm{O}_{\mathbf{3}}$ solution
Weight of weighing bottle and $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ salt: ................. $g$
Weight of empty weighing bottle: .g
Weight of $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ salt: $\qquad$ ..g

|  | Weight of $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ salt X 4 | X |
| :---: | :---: | :---: |
| Normality of $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ : | uivalent weight of $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ |  |


| Regular |  |  |  |  |
| :--- | :---: | :---: | :---: | :---: |
| Burette I II <br> Reading in ml   | III |  |  |  |
| Final burette <br> Reading |  |  |  |  |
| Initial burette <br> Reading |  |  |  |  |
| Volume of <br> Na2 $\mathbf{S}_{2} \mathbf{O}_{3}$ <br> consumed <br> (ml.) |  |  |  |  |

Part B: Determination of \% of Chlorine in the given Bleaching powder sol:
Burette: Standard Sodium thiosulphate solution.
Conical flask: 25 cm 3 bleaching powder solution +1 t.t dist. Water $++1 / 4$ t.t Acetic acid $+5 \mathrm{ml} .10 \%$
KI.
Indicator : 2-3 drops of freshly prepared starch.
End point: Disappearance of dark blue colour.
$1000 \mathrm{~cm}^{3}$ of $1 \mathrm{~N} \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ solution $=35.5 \mathrm{~g}$ of Chlorine
Therefore $\mathrm{Vcm}^{3}$ of $1 \mathrm{~N} \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}=\frac{\mathrm{Xx} \mathrm{V} \mathrm{x} \mathrm{35.5}}{1000}=\quad-\quad=\quad(\mathrm{ag})$

Weight of Bleaching powder taken=W $\qquad$ g

Percentage of Chlorine $=\left(\right.$ a) $\times 100=\frac{\times 100=\text { of }}{W}$ Chlorine

REPORT: The percentage of the available chlorine in the given sample of Bleaching powder is $=$ $\qquad$

# Determination of chlorine in a given Bleaching powder by Iodometric method 

## Principle:

Available chlorine in a sample of bleaching powder is estimated volumetrically. A known mass of bleaching powder as a suspension of water is treated with excess of potassium iodide solution. The solution is the acidified with a strong solution of acetic acid. The liberated iodine is titrated against standard sodium thiosulphate solution using starch as indicator.

$$
\mathrm{CaOCl}_{2}+2 \mathrm{CH}_{2} \mathrm{COOH}+2 \mathrm{KI} \longrightarrow \mathrm{CaCl}_{2}+3 \mathrm{CH}_{3} \mathrm{COOK}+3 \mathrm{I}_{2}+\mathrm{H}_{2} \mathrm{O}
$$

$$
2 \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}+\mathrm{I}_{2} \longrightarrow 2 \mathrm{NaI}+\mathrm{Na}_{2} \mathrm{~S}_{4} \mathrm{O}_{6}(\text { Colour less })
$$

## Procedure:

## Part A: Preparation of Sodium Thiosulphate solution:

Weigh exactly the given amount of $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ crystals and transfer on to the funnel placed on a clean $250 \mathrm{~cm}^{3}$ volumetric flask. Dissolve it in small quantities of deionized water and make it up to the mark and mix well to get uniform concentration.

## Part B :

$25 \mathrm{~cm}^{3}$ of the prepared solution is pipetted out into a clean conical flask followed by one test tube of distilled water, And add $1 / 4$ test tube of glacial acetic acid followed by 5 ml .of $10 \%$ Potassium Iodide The liberated Iodine is titrated against standard Sodium thiosulphate solution which is taken in burette till a pale yellow colour is obtained. 2-3 drops of freshly prepared starch indicator is added. The resulting blue solution obtained is titrated against the same solution till the blue colour just discharges. The titration is repeated for concordant values.

REPORT: The percentage of the available chlorine in the given sample of Bleaching powder is $=$ $\qquad$

## Observations and Calculations

## Graph:



Volume of FAS solution pipetted out $=25 \mathrm{ml}$
Normality of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ used $\quad=0.05 \mathrm{~N}$

| $\begin{aligned} & \text { Vol.of } \mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7} \\ & \text { (V) } \mathrm{ml} \end{aligned}$ | $\begin{gathered} \text { Emf E } \\ (\mathrm{mv}) \end{gathered}$ | $\begin{gathered} \Delta \mathrm{V} \\ \left(\mathrm{~V}_{2}-\mathrm{V}_{1}\right) \mathrm{ml} \end{gathered}$ | $\begin{gathered} \Delta \mathrm{E} \\ \left(\mathrm{E}_{2}-\mathrm{E}_{1}\right) \end{gathered}$ | $\begin{aligned} & \Delta \mathrm{E} / \Delta \mathrm{V} \\ & (\mathrm{mv} / \mathrm{ml}) \end{aligned}$ |
| :---: | :---: | :---: | :---: | :---: |
| 0.0 |  | ---------- | ----------- | ----------- |
| 0.5 |  |  |  |  |
| 1.0 |  |  |  |  |
| 1.5 |  |  |  |  |
| 2.0 |  |  |  |  |
| 2.5 |  |  |  |  |
| 3.0 |  |  |  |  |
| 3.5 |  |  |  |  |
| 4.0 |  |  |  |  |
| 4.5 |  |  |  |  |
| 5.0 |  |  |  |  |
| 5.5 |  |  |  |  |
| 6.0 |  |  |  |  |
| 6.5 |  |  |  |  |
| 7.0 |  |  |  |  |
| -- |  |  |  |  |
| -- |  |  |  |  |
| -- |  |  |  |  |
| -- |  |  |  |  |
| -- |  |  |  |  |
| -- |  |  |  |  |
| -- |  |  |  |  |

Take 6 more readings after peak rise

## POTENTIOMETRIC TITRATION OF FAS VS K $\mathbf{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$

Aim: To determine the strength of FAS solution (Mohr's salt solution) potentiometrically, by titrating with a standard $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ Solution.

Principle: Redox titrations can be carried out potentiometrically using platinum calomel electrode combination in a manner similar to acid-base neutralizations. For the reaction
Re duced form $\rightarrow$ Oxidised form $+n$ electrons
The potential is given by Nernst equation
$E=E^{o}+\frac{0.0591}{n} \log \frac{[\text { Oxidised form }]}{[\text { Re duced form }]}$
Where $\mathrm{E}^{0}$ is the standard potential of the system. The potential of the system is thus controlled by the ratio of concentration of the oxidized to that of the reduced species present. As the reactions proceeds the ratio increases and hence the potential also increases more rapidly in the vicinity of the end point of titration. A plot of change in potential against volume is characterized by a sudden change of potential at the equivalent point. At the end point potential is determined by
(Since $\left[\mathrm{Fe}^{2+}\right]=0$ )
$\mathrm{E}=0.7: \mathrm{E} \quad=1.0 \mathrm{~V}, \mathrm{E}=1.7 \mathrm{~V}$
Hence there is large increase in potential at the end point
Procedure: Pipette out $25.0 \mathrm{~cm}^{3}$ of ferrous ammonium sulphate solution into a beaker. Add 2 test tube of dilute sulphuric acid. Immerse the platinum-calomel electrode assembly into the beaker containing the solution. Connect the electrodes to the potentiometer. Note down the emf of the cell before the addition of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$. Now add 0.5 ml of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ from the semi micro burette. Allow the solution to mix well and measure the potential. Continue the procedure till a sudden rise in emf of the cell is observed. Take about 5-6 more readings.
Determine the end point by plotting $\frac{\Delta E}{\Delta V}$ against the volume as shown in the figure.
Result: Normality of the given FAS solution $=$ $\qquad$ g/ttr.

Weight of FAS $/ \mathrm{ltr}=$ $\qquad$ g/ltr.

Weight of Fe present in one liter $=$ $\qquad$ $\mathrm{g} / \mathrm{tr}$.

$$
\mathrm{N}_{\mathrm{FAS}}=\frac{(\mathrm{NV})_{\mathrm{K}_{2} \mathrm{C}_{2} \mathrm{O}_{7}}}{\mathrm{~V}_{\mathrm{FAS}}}
$$

Amount of FAS per liter $=\mathrm{N}_{\mathrm{FAS}} \times \mathrm{Eq}$. Wt. of FAS (392)
$\qquad$

392 g of FAS contains 55.85 g Fe
$a g$ of FAS contains $\frac{55.85 \times a}{392}$
$=. . . . . . . . . . . . . . . . g$
Result: Normality of the given FAS solution $=$
Weight of FAS / ltr $=\ldots$ g/ltr.
Weight of Fe present in one liter $=\ldots \mathrm{g} / \mathrm{ltr}$.

Signature of Staff member with date: $\qquad$ .

## Viva Voce

1) What is the Principle of potentiometric titration?
2) What are the electrodes used in this experiment?
3) What are the advantages of potentiometricTitration?
4) How do you account for an abrupt increase in the potential of the solution at one point of titration?
5) What is the role of dilute $\mathrm{H}_{2} \mathrm{SO}_{4}$ ?
6) Write the reaction that takes place during titration?

| Vol. of NaOH | Conductivity( $\mathrm{ohm}^{-1} / \mathrm{m}$ ) |
| :---: | :---: |
| 0 |  |
| 0.5 |  |
| 1 |  |
| 1.5 |  |
| 2 |  |
| 2.5 |  |
| 3 |  |
| 3.5 |  |
| 4 |  |
| 4.5 |  |
| 5 |  |
| 5.5 |  |
| 6 |  |
| 6.5 |  |
| 7 |  |
| 7.5 |  |
| 8 |  |
| 8.5 |  |
| 9 |  |
| 9.5 |  |
| 10.0 |  |
| 10.5 |  |
| 11.0 |  |
| 11.5 |  |
| 12.0 |  |
| 12.5 |  |
| 13.0 |  |
| 13.5 |  |
| 14.0 |  |
| 14.5 |  |
| 15.0 |  |
| 16.0 |  |
| 16.5 |  |
| 17.0 |  |
| 17.5 |  |
| 18.0 |  |
| 18.5 |  |
| 19.0 |  |

## CONDUCTOMETRIC TITRATION

Aim: To determine the strength of mixture of Acetic acid and Hydrochloric acid using standard NaOH Solution conductometically.

Principle: The determination of end point of a titration by means of conductivity measurement is called conductmetric titration. Conductivity of a solution at a constant temperature depends upon the, number of ions per unit volume and charge on the ion and their mobility. In this particular experiment, the standard NaOH is added from the burette to the measured volume of mixture of acetic acid and hydrochloric acid solution taken in the beaker, in which the conductivity cell is dipped. The conductivity readings corresponding to various increments of NaOH are plotted against the volume of NaOH .
Upon addition of a strong base to a mixture of acids the conductance falls till the strong acids is neutralized owing the replacement of $\mathrm{H}^{+}$ions by less mobile cation $\left(\mathrm{Na}^{+}\right)$. Once all strong acid is neutralized the conductance increases as the weak acid is converted to its ionized salt. Once the neutralization is complete the conductance increases sharply due to high mobility of $\mathrm{OH}^{-}$ions
$\mathrm{H}^{+}+\mathrm{Cl}^{-}+\mathrm{Na}^{+}+\mathrm{OH}^{-}----------------\mathrm{Na}^{+} \mathrm{Cl}^{-}+\mathrm{H}_{2} \mathrm{O}$
$\mathrm{CH}_{3} \mathrm{COOH}+\mathrm{Na}^{+}+\mathrm{OH}^{-}------------------\mathrm{CH}_{3} \mathrm{COO}^{-} \mathrm{Na}^{+}+\mathrm{H}_{2} \mathrm{O}$

## Procedure:

The conductivity meter is calibrated. 50 ml of given mixture of acids is rundown from the burette into clean 100 ml beaker and stried with a magnetic stirrer. The conductivity cell is dipped in the solution. The conductance of the solution before addition of base is noted down.
The burette is rinsed and filled with std NaOH solution. Small portion of base ( 1 ml ) is added each time and the conductance of the solution corresponding to various increments are noted down. A graph is plotted between the conductance value (y-axis) and volume of NaOH ( x -axis). Two equivalence points are obtained from the graph. First inflection point gives the volume of alkali used by strong acid and the second inflection point gives the volume of alkali by the weak acid.

From the volume of alkali consumed the strength of acid can be determined.


## Neutralization <br> point of $\mathbf{H C l}$ <br> Neutralization point of $\mathrm{CH}_{3} \mathrm{COOH}$

## Observations and Calculations:

$$
\begin{array}{lrl}
\text { Strength of standard } \mathrm{NaOH} \text { solution } & \mathrm{N}_{\mathrm{NaOH}} & =\ldots \ldots \ldots . \\
& =50 \mathrm{~cm}^{3} \\
\text { Volume of acid mixture pipetted out } & & =\ldots \ldots \ldots \mathrm{cm}^{3}
\end{array}
$$

Volume of NaOH required to neutralize both HCl and $\mathrm{CH}_{3} \mathrm{COOH}\left(\mathrm{V}_{2}\right)=$ ..... $\mathrm{cm}^{3}$
Volume of NaOH required to neutralize $\mathrm{CH}_{3} \mathrm{COOH}=\left(\mathrm{V}_{2}-\mathrm{V}_{1}\right)=$ ..... $\mathrm{cm}^{3}$Therefore, the strength of HCl
$\mathrm{N}_{\mathrm{HCl}}=$

$\qquad$ ..... (V1)
$=$ ..... N
Weight of HCl present in $1 \mathrm{dm}^{3}$ of acid mixture $=\mathrm{N}_{\mathrm{HCl}} \times$ Eq.wt of HCl
$=\ldots \ldots . . . . x 36.5 \mathrm{~g} / \mathrm{dm}^{3}$

$$
=\ldots \ldots \ldots \ldots \ldots \mathrm{g} / \mathrm{dm}^{3}
$$

The strength of $\mathrm{CH}_{3} \mathrm{COOH} \mathrm{N}_{\mathrm{CH} \text { coor }}=\ldots \ldots \ldots \ldots\left(\mathrm{N}_{\mathrm{NaOH}}\right) \times \ldots \ldots \ldots \ldots .\left(\mathrm{V}_{2}-\mathrm{V}_{1}\right)$
$\qquad$
Weight of $\mathrm{CH}_{3} \mathrm{COOH}$ present in $1 \mathrm{dm}^{3}$ of acid mixture $=\mathrm{N}_{\mathrm{CH} \text { cooh }} \mathrm{x}$ Eq.wt of $\mathrm{CH}_{3} \mathrm{COOH}$

$$
\begin{aligned}
& =\ldots \ldots \ldots . . \times 60 \mathrm{~g} / \mathrm{dm}^{3} \\
& =\ldots \ldots \ldots \ldots \cdot \mathrm{g} / \mathrm{dm}^{3}
\end{aligned}
$$

## Result:

1) Weight of HCl present in $1 \mathrm{dm}^{3}$ of acid mixture $\quad=\ldots \ldots \ldots \ldots \mathrm{g} / \mathrm{dm}^{3}$
2) Weight of $\mathrm{CH}_{3} \mathrm{COOH}$ present in $1 \mathrm{dm}^{3}$ of acid mixture $=\ldots \ldots \ldots \ldots \mathrm{g} / \mathrm{dm}^{3}$

Signature of Staff member with date: $\qquad$ .

## Viva Voce

1) What is the principle of conductometric titration?
2) State Ohm's law.
3) What is conductance? What is its unit?
4) What is conductivity? What is its unit?
5) How do you explain the titration curve on the basis of mobility of ions?
6) What are the advantages of conductometric titration?
7) What is the type of conductivity determined by this experiment? Give its unit
8) What are the electrodes used?
9) What is the type of conductivity measured in this experiment?
10) How is conductance and conductivity related?
11) What are the factors on which conductivity depends on?

## Observations and Calculations:

Graph:


| Volume of $\mathrm{CuSO}_{4}$ in ml. | Absorbance |
| :---: | :---: |
| 2 |  |
| 4 |  |
| 6 |  |
| 8 |  |
| 10 |  |
| Unknown |  |

## From the graph

Volume of $\mathrm{CuSO}_{4} .5 \mathrm{H}_{2} \mathrm{O}$ in test solution $=$ $\qquad$ ml (say V ml )

Working solution concentration $\quad=4 \mathrm{mg} / \mathrm{cc}$
249.6 mg of $\mathrm{CuSO}_{4} .5 \mathrm{H}_{2} \mathrm{O}$ contains 63.55 mg of $\mathrm{Cu}^{2+}$

Therefore 4 mg of $\mathrm{CuSO}_{4} .5 \mathrm{H}_{2} \mathrm{O}$ contains $\frac{63.55 \times 4}{249.6}$

$$
=\ldots 1.02 \mathrm{mg}
$$

1 ml of $\mathrm{CuSO}_{4} .5 \mathrm{H}_{2} \mathrm{O}$ contains 1.02 mg of $\mathrm{Cu}^{2+}$
V ml of $\mathrm{CuSO}_{4} .5 \mathrm{H}_{2} \mathrm{O}$ contains $\mathrm{V} \times 1.02 \mathrm{mg}$ of $\mathrm{Cu}^{2+}$
Since the stock solution is diluted to 50 times
$\mathrm{Cu}^{2+}$ present in test solution $\frac{V \times a}{50}$
$\qquad$
Result: Concentration of $\mathrm{Cu}^{2+}$ in test solution $=$ $\qquad$ mg

## COLORIMETRIC ESTIMATION OF COPPER

Aim: To estimate copper colorimetrically using colorimeter.
Principle: When a monochromatic light of intensity $\mathrm{I}_{0}$ is incident on a transparent medium a part $I_{a}$ is absorbed, a part $I_{r}$ is reflected and the remaining part $I_{t}$ is transmitted.

$$
\mathrm{I}_{0}=\mathrm{I}_{\mathrm{a}}+\mathrm{I}_{\mathrm{r}}+\mathrm{I}_{\mathrm{t}}
$$

For a glass-air interface $\mathrm{I}_{\mathrm{r}}$ is negligible. Therefore,

$$
\mathrm{I}_{0}=\mathrm{I}_{\mathrm{a}}+\mathrm{I}_{\mathrm{t}}
$$

$\mathrm{I}_{\mathrm{t}} / \mathrm{I}_{0}=\mathrm{T}$ called the transmittance, $\log 1 / \mathrm{T}=\log \mathrm{I}_{0} \mathrm{I}_{\mathrm{t}}$ is called the absorbance or optical density A. The relation between absorbance A , concentration C (expressed in mol lit ${ }^{-1}$ ) and path length t (expressed in cm ) is given by Beer- Lambert's law,

$$
\mathrm{A}=\log \mathrm{I}_{0} / \mathrm{I}_{\mathrm{t}}=\mathrm{ECt}
$$

Where $€$ is the molar extinction coefficient, $€$ is a constant for a given substance at a given wavelength. If the path length (cell thickness) is kept constant, then, A $\alpha$ C. Hence a plot of absorbance against concentration gives a straight line as shown in figure (calibration curve). The color is measured using a spectrophotometer at a wavelength where the absorbance is maximum.

A series of standard solutions containing cupric ions is treated with ammonia to get deep blue cuprammonium complex and is diluted to a definite volume.

$$
\mathrm{Cu}^{2+}+4 \mathrm{NH}_{3} \rightarrow\left[\mathrm{Cu}\left(\mathrm{NH}_{3}\right)\right]^{2+}
$$

Deep blue
The absorbance of each of these solutions is measured at 620 nm since the complex shows maximum absorbance at this wavelength. The concentration is plotted against absorbance to get a calibration curve.
A known volume of the test solution is treated with strong ammonia and diluted to the same volume as above. The absorbance of this solution at 620 nm is measured and its concentration is determined from the calibration curve.

## Procedure:

Part-A
Standardization of the Colorimeter:
"Set Zero" and "Set 100 " Knobs must be at a minimum. Using an opaque colorimetric tube adjust the "Set Zero" Knob to get \% transmittance. Using blank solution in the colorimetric tube adjust the "Set 100 " knob to get $100 \%$ transmittance with out disturbing "Set Zero" Knob. Switch on the colorimetric tube on the OD mode.

## Part-B

Place the given copper sulphate solution (stock solution) in a burette and transfer 2,4,6,8 and 10 ml of the solution into separate, labeled $50 \mathrm{~cm}^{3}$ volumetric flasks. Add $2.5 \mathrm{~cm}^{3}$ of ammonia $\left(\mathrm{NH}_{4} \mathrm{OH}\right)$ solution to each of them and make up to the mark with distilled water. Stopper the flasks and mix the solutions well. To the test solution taken in a 50 $\mathrm{cm}^{3}$ standard flask, add 2.5 ml of ammonia solution and make up to the mark. Mix well. Prepare a blank solution by diluting $2.5 \mathrm{~cm}^{3}$ of ammonia solution in $50-\mathrm{cm} 3$ volumetric flask to the mark with ion exchange water and mix well. After 10 minutes, measure the absorbance of the solutions against blank at 620 nm using a spectrophotometer.

Tabulate the readings as shown. Draw a calibration curve by plotting volume of copper (in mg per $\mathrm{cm}^{3}$ ) against absorbance. Using the calibration curve determine the unknown volume of copper sulfate and calculate the amount of copper in the solution.

Note: Ammonia should be added to all the solutions including the test solution at the same time.

Result: Concentration of $\mathrm{Cu}^{2+}$ in test solution $=$ $\qquad$ mg.

## Signature of Staff member with date:

$\qquad$ .

## Viva Voce

1) What is colorimetry?
2) Define Wave length, Wave number \& frequency?
3) State Beer law, Lambert's law and Beer-Lambert's law?
4) Define Transmittance, absorbance and Molar extinction co-efficient?
5) Why is ammonia added \& why is it that the same amount of ammonia is added \& at the same time?
6) Why are different volumes of solution taken in the flasks?
7) What is blank solution?
8) What is the usefulness of calibration curve?
9) Why is the estimation done at fixed wavelength?
10) How is transmittance and absorbance related?
11) Is it possible to get a linear curve with transmittance parameter?
12) State the calorimeter \& colorimeter?

## Observation and Calculations:

Graphs:


Equivalence point, $V_{e}$


Half equivalence point

| Vol.of NaOH (V)mL | pH | $\begin{gathered} \Delta \mathrm{V} \\ \left(\mathrm{~V}_{2}-\mathrm{V}_{1}\right) \mathrm{ml} \end{gathered}$ | $\begin{aligned} & \Delta p H \\ & \left(\mathrm{pH}_{2}-\mathrm{pH}_{1}\right) \end{aligned}$ | $\frac{\Delta p H}{\Delta V}$ |
| :---: | :---: | :---: | :---: | :---: |
| 0.0 |  | -------- | ---------- | --------- |
| 0.5 |  |  |  |  |
| 1.0 |  |  |  |  |
| 1.5 |  |  |  |  |
| 2.0 |  |  |  |  |
| 2.5 |  |  |  |  |
| 3.0 |  |  |  |  |
| 3.5 |  |  |  |  |
| 4.0 |  |  |  |  |
| 4.5 |  |  |  |  |
| 5.0 |  |  |  |  |
| 5.5 |  |  |  |  |
| 6.0 |  |  |  |  |
| 6.5 |  |  |  |  |
| 7.0 |  |  |  |  |
| 7.5 |  |  |  |  |
| 8.0 |  |  |  |  |
| 8.5 |  |  |  |  |
| 9.0 |  |  |  |  |

## DETERMINATION OF pK ${ }_{\mathrm{a}}$ VALUE OF WEAK ACID

Aim: To determine the $\mathrm{pk}_{\mathrm{a}}$ value of weak acid Potentiometrically using a pH meter.
Principle:
A weak acid, like acetic acid dissociates partially in its aqueous solution

$$
\mathrm{CH}_{3} \mathrm{COOH}+\mathrm{H}_{2} \mathrm{O} \Leftrightarrow \mathrm{H}_{3} \mathrm{O}^{+}+\mathrm{CH}_{3} \mathrm{COO}^{-}
$$

The equilibrium constant of dissociation of the acid is known as dissociation constant of the acid $\mathrm{K}_{\mathrm{a}}$ is given by

$$
K_{a}=\frac{\left[\mathrm{H}_{3} \mathrm{O}\right]\left[\mathrm{CH}_{3} \mathrm{COO}^{-}\right]}{\left[\mathrm{CH}_{3} \mathrm{COOH}\right]}
$$

The solution is considered to be very dilute and activity of water in very dilute solution is assumed to be 1

$$
\left[\mathrm{H}_{3} \mathrm{O}\right]=\frac{K_{a}\left[\mathrm{CH}_{3} \mathrm{COOH}\right]}{\left[\mathrm{CH}_{3} \mathrm{COO}^{-}\right]}
$$

Taking - $\log$ on both sides

$$
\begin{aligned}
& -\log \left[\mathrm{H}_{3} \mathrm{O}\right]=-\log K_{a}+\log \frac{\left[\mathrm{CH}_{3} \mathrm{COO}^{-}\right]}{\left[\mathrm{CH}_{3} \mathrm{COOH}^{2}\right]} \\
& p H=p K_{a}+\log \frac{\left[\mathrm{CH}_{3} \mathrm{COO}^{-}\right]}{\left[\mathrm{CH}_{3} \mathrm{COOH}\right]}
\end{aligned}
$$

When acetic acid is titrated against NaOH solution, pH of the titrant solution increases in accordance to the above equation since concentration of sodium acetate increases and concentration of unreacted acid decreases.
When acid is half titrated $\left[\mathrm{CH}_{3} \mathrm{COO}^{-}\right]=\quad\left[\mathrm{CH}_{3} \mathrm{COOH}\right]$
Hence $\mathrm{pKa}=\mathrm{pH}$ of the half equivalence point.

## Procedure:

Pipette out $25.0 \mathrm{~cm}^{3}$ of given weak acid solution into a beaker. Immerse the glasscalomel electrode assembly into the beaker containing the solution. Properly connect the electrodes to the pH meter. Note down the pH of the solution before the addition of NaOH . Now add 0.5 ml of NaOH from the semi micro burette. Allow the solution to mix well and measure the pH . Continue the procedure till a sudden rise in pH of the solution is observed. Take about 5-6 more readings, beyond that
Determine the end point by plotting $\frac{\Delta p H}{\Delta V}$ against the volume of NaOH as shown in the figure. Now plot the graph taking the volume of NaOH along the X -axis and pH along the Y - axis. Then determine the pKa value from the graph.

$$
\begin{aligned}
& \mathrm{V}_{\mathrm{NaOH}}=\ldots \ldots \ldots \ldots \ldots \ldots \mathrm{ml} \\
& \frac{V_{\mathrm{NaOH}}}{2}=\ldots \ldots . . . . . . . m l
\end{aligned}
$$

Result: pKa value of weak acid

## Viva Voce

1) What is weak acid?
2) What is $K_{a}$ ? What is $p K_{a}$ ?
3) What is pH ? Explain pH scale?
4) How $\mathrm{pH} \& \mathrm{pKa}$ is related?
5) What are the electrodes used in this experiment? Mention their type?
6) How do you account for sudden increase in pH of solution at one point of titration?
7) How is pKa related to strength of acid?
8) What is the relationship between $\mathrm{pH} \& \mathrm{pOH}$ ?
9) When does pH become equal to pKa ?
10) What type of electrode is?
i) Glass electrode?
ii) Calomel electrode?

## Observations and Calculations:

Lab temperature $=$ $\qquad$ ${ }^{\circ} \mathrm{C}$
Density of given liquid $\mathrm{d}_{\mathrm{L}}=$ $\qquad$ $\mathrm{g} / \mathrm{cm}^{3}$
Density of water $\mathrm{d}_{\mathrm{w}}=$ $\mathrm{g} / \mathrm{cm}^{3}$
Viscosity of water $\mathrm{n}_{\mathrm{w}}=$ $\qquad$ mill poise

| Trial <br> No. | $t_{\mathrm{L}}$ (secs.) | $\mathrm{t}_{\mathrm{w}}$ (secs.) |
| :---: | :---: | :---: |
| 1 |  |  |
| 2 |  |  |
| 3 |  |  |
| Average |  |  |



Co-efficient of viscosity of liquid,

$$
\eta_{L}=\frac{t_{L} d_{L}}{t_{W} d_{W}} \times \eta_{W}
$$

Result: Co-efficient of viscosity of liquid= $\qquad$ millipoise.
$\qquad$ .

## Experiment No. 11

Date:

## CO-EFFICIENT OF VISCOSITY OF LIQUID

Aim: To determine the co-efficient of viscosity of the given liquid using Ostwald's viscometer.

Principle: Viscosity arises due to internal friction between moving layers of molecules. A liquid flowing through a cylindrical tube of uniform diameter is expected to move in the form of molecular layers. A layer close to the surface is almost stationary while that at the axis of the tube moves faster than any other intermediate layer. A slow moving layer exerts a drag or friction on its nearest moving layer backwards. This property of liquid by which it retards or opposes motion between layers is called viscosity. The co-efficient of viscosity is defined as tangential force/unit area required to maintain a unit velocity gradient between any two successive layers of a liquid situated unit distance apart. The co-efficient of viscosity of a liquid is given by poiseiulle's formula

$$
\mathrm{V}=\frac{\Pi \rho \mathrm{r}^{4} \mathrm{t}}{8 \eta 1} \quad \begin{aligned}
& \mathrm{V}=\text { Volume of liquid } \\
&
\end{aligned} \quad \begin{aligned}
& 1=\text { radius of tube } \\
& \eta=\text { length of tube } \\
& \rho=\text { Pressure difference between the } 2 \text { ends of tubes. }
\end{aligned}
$$

If equal volumes of 2 different liquids are allowed to flow through the same tube under identical conditions. Then

$$
\begin{equation*}
\frac{\eta_{\mathrm{L}}}{\eta_{w}}=\frac{\mathrm{t}_{\mathrm{L}} \mathrm{~d}_{\mathrm{L}}}{\mathrm{t}_{\mathrm{w}} \mathrm{~d}_{\mathrm{w}}} \tag{1}
\end{equation*}
$$

$\mathrm{t}_{\mathrm{L}}, \mathrm{t}_{\mathrm{w}}$ - time taken by the test liquid, water chosen to flow through the same distance in the tube respectively.
$\mathrm{d}_{\mathrm{L},} \mathrm{d}_{\mathrm{w}}$ - densities of test liquid and water respectively under same room temperature respectively.
$\eta_{\mathrm{L},} \eta_{\mathrm{w}}-$ Viscosity co-efficient of test liquid and water respectively under same room temperature.
Thus co-efficient of viscosity of given test liquid could be found.

## Procedure:

Clean the viscometer with water, rinse with acetone and dry it. Fix the viscometer vertically to stand in a constant temperature bath (beaker containing water). Using a clean pipette, transfer $20 \mathrm{~cm}^{3}$ of liquid in to the wider limb. Allow the viscometer to stand for some time so that the liquid attains the temperature of the bath. Suck the water above the upper mark A of the viscometer. Allow it to flow freely through the capillary. When the level of water just crosses the lower mark B, note the time of flow in seconds ( $\mathrm{t}_{\mathrm{L}}$ ). Repeat to get agreeing values. Pour out the liquid, rinse the viscometer with acetone and dry it. Repeat the experiment with exactly the same volume of water and find the time of flow ( $\mathrm{t}_{\mathrm{w}}$ seconds).
Calculate the coefficient of viscosity of the liquid using Eq. (1).

Result: Co-efficient of viscosity of liquid = $\qquad$ millipoise.

## Viva Voce

1) What is viscosity?
2) Define viscosity co-efficient of liquid?
3) What is the SI unit of viscosity co-efficient?
4) How does the viscosity vary with temperature?
5) Why is the same Volume of water \& liquid used in the experiment?
6) Write poiseuille's equation \& Define the terms involved?
7) Why should the viscometer be dry before measurements are done?
8) Why is acetone used for cleaning the viscometer?
9) Name the factors influencing viscosity and viscosity Co-efficiency?

## Observations and Calculations: <br> For Sodium

| Volume of NaCl | Emission Response |
| :---: | :---: |
| 2.5 |  |
| 5.0 |  |
| 7.5 |  |
| 10 |  |
| Unknown |  |

Working solution concentration $\quad=0.1 \mathrm{mg} / \mathrm{cc}$
58.5 mg of NaCl contains 23 mg of $\mathrm{Na}^{+}$

Therefore 0.1 mg of NaCl contains $\frac{23 \times 0.1}{58.5}$
$=\ldots \ldots \ldots . . \mathrm{mg}$ of Na
Result: Concentration of Na in the given water sample $=$ $\qquad$

Signature of Staff member with date: $\qquad$ .

## FLAME PHOTOMETRY

Aim: To estimate the sodium and potassium content in the given sample of water by flame photometry.

## Principle:

The principle involved in flame photometry is that when a solution containing a metallic compound is aspirated into a flame, a vapour containing metal atoms will be formed. Some of these metal atoms in gaseous state may be raised to an energy level which is sufficiently high to permit the emission of radiation, which is characteristic to the metal under investigation. This method is generally used for the analysis of sodium, potassium, calcium and lithium.

The layout of a simple flame photometer is shown below.


Air at a given pressure is passed into the atomizer, and the suction produced by this air draws a solution of the sample into the atomizer, where it joins the air stream as a fine mist and passes into the burner. Here in a small mixing chamber, the air meets the fuel gas supplied to the burner at a given pressure and the mixture is burnt. Radiations from the resulting flame passes through a lens, and finally through an optical filter which permits only the radiation characteristic of the element under investigation to pass through the photocell. The output from the photocell is measured on a suitable digital read-out system.

Procedure: Pipette out 2.5, 5, 7.5 and 10 ml of standard NaCl solution into four different 50 ml standard flasks. Make the solutions up to the mark and shake well for uniform concentration. Take the first standard solution and aspirate into the flame of the Flame photometer. The flame emission intensity is measured by flame photometer using sodium filter $(598 \mathrm{~nm})$. The same is repeated for all standard solutions with distilled water being sprayed to the flame in between the trials. Then aspirate the given sample of water into the flame and note down the value of emission response. Plot the graph by taking emission response along the Y axis and concentration along the X axis. Calculate the concentration of the sodium in the water sample, in the same way proceed for potassium.

Result: Amount of Sodium present in the given sample of water = $\qquad$
Amount of Potassium present in the given sample of water $=$ $\qquad$

## Viva Voice

1) What is ment by emission?
2) What is flame photometer?
3) What are alkali and alkaline earth metals
4) What are various events that occur when a solution containing metal atom ion atomized through a flame in flame photometry?
5) What are the different components of flame photometer?
6) Why is analysis of sodium advantageous in flame photometer?
7) What is role of filter in flame photometry?
8) What are the errors that occurs in flame photometry?
9) What are the factors that influence this experiment?
10) Name the fuel and oxidation used in this experiment?

## Procedure write up

Note: The student has to write the procedure of two experiments in brief in 10 minutes. The correct format is given below.

## 1. Hard water.

Part-A: Preparation of standard solution of EDTA: Weigh accurately about 4.5 g of EDTA and transfer into a 250 ml standard flask. Dissolve completely with 5 ml of $\mathrm{NH}_{3}$ and sufficient amount of water. Make up the solution to the mark and shake well for uniform concentration.

$$
M_{\text {EDTA }}=\frac{\text { Weight of EDTA } \times 4}{\text { Molecular weight of EDTA }(372.24)}
$$

## Part - B: Estimation

Burette : Standard EDTA solution.
Conical flask : 25.0 ml of hard water $+2 \mathrm{ml} \quad \mathrm{NH}_{4} \mathrm{OH}---\mathrm{NH}_{4} \mathrm{Cl}$ buffer solution.
Indicator : A pinch of Eriochrome Black T
Colour change : Wine red to blue
Repeat the titration for concordant values and by the volume of EDTA Consumed calculate the total hardness of the given water sample.

## 2. Cement solution.

## Part-A: Preparation of standard solution of EDTA:

Weigh accurately given amount of EDTA and transfer into a 250 ml standard flask. Dissolve completely with 5 ml of $\mathrm{NH}_{3}$ and sufficient amount of water. Make up the solution to the mark and shake well for uniform concentration.

$$
M_{\text {EDTA }}=\frac{\text { Weight of EDTA } \times 4}{\text { molecular weight of EDTA }(372.24)}
$$

## Part B: Estimation

Burette : Standard EDTA
Conical flask : 25.0 ml of cement solution +10 ml .of glycerol \& diethyl amine with constant stirring +10 ml of $\mathrm{NaOH}+1$ t.t. of distilled water. + shake well for 3 min .
Indicator : a pinch of Patton and Reeder's indicator
Colour change: Wine red to blue.
Repeat the titration for concordant values and by the volume of EDTA consumed calculate the percentage of CaO in Cement sample.

## 3. Cu in Brass.

Given amount of Brass solution taken in 250 ml . standard flask \& make up to the mark with distilled water, Pipette out 25 ml . of brass solution in to conical flask.

Burette : Standard Sodium thiosulphate solution.
Conical flask : 25 ml . of brass solution $+\mathrm{NH}_{4} \mathrm{OH}$ (drop wise till bluish white ppt.) + $\mathrm{CH}_{3} \mathrm{COOH}$ (to dissolve the ppt. \& 2-3 drops excess) $+10 \% 10 \mathrm{ml} \mathrm{KI}$
Indicator : Starch (near the end point)
Colour change: Deep blue to milky white.
By the volume of $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ consumed calculate the percentage of copper.

## 4. Iron in haematite solution.

## Part-A: Preparation of standard $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ solution

Weigh accurately given amount of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ crystals into a 250 ml standard flask. Dissolve the salt in little distilled water. Make up the solution to the mark and shake well for uniform concentration.

$$
\mathrm{N}_{\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}}=\frac{\left(\mathrm{W}_{1}-\mathrm{W}_{2}\right) \times 4}{\text { Eq. Wt. of } \mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}(49)}
$$

## Part-B: Estimation

Burette : Standard $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$
Conical flask : 25.0 ml of Haematite ore solution +5 ml of 4 N HCl , boil $+\mathrm{SnCl}_{2}$
drop wise till solution becomes colorless, 2 drops in excess, cool, 5 ml of $\mathrm{HgCl}_{2}+1$ t.t. of water

Indicator : Potassium ferricyanide [external]
Colour change: Disappearance of blue colour
Repeat for concordant values \& by the volume of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ consumed calculate the percentage of Iron in given Haematite ore solution.

## 5. C.O.D in waste Water

## Part-A: Preparation of standard Mohr's salt solution:

Weigh accurately the given Mohr's salt, transfer it in to a 250 ml standard flask, dissolve in a little amount of distilled water and a t.t of dilute $\mathrm{H}_{2} \mathrm{SO}_{4}$ and make up to the mark with distilled water. Shake well for uniform concentration.

$$
\mathrm{N}_{\mathrm{FAS}}=\frac{\left(\mathrm{W}_{1}-\mathrm{W}_{2}\right) \times 4}{\text { Eq.. Wt.of FAS }(392)}
$$

## Part-B:

## 1. Sample titration

| Burette $:$ <br> Conical flask $:$ <br>  Standard FAS <br>  25.0 ml of waste water sample +10.0 ml of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}+10$ <br> ml of $1: 1 \mathrm{H}_{2} \mathrm{SO}_{4}$  |  |  |
| :--- | :--- | :--- |
| Indicator | $:$ | Ferroin $(2-3$ drops $)$. |
| Colour change | $:$ | Bluish green to reddish brown. |

## 2. Blank titration

| Burette | : $\quad$ Standard FAS |  |
| :--- | :--- | :---: |
| Conical flask | $:$ | 10.0 ml of standard $\quad \mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}+1: 1$ Sulphuric acid |

Indicator : Ferroin.
Colour change: Bluish green to reddish brown.
Repeat the titrations for concordant values \& by the volume of FAS consumed calculate the COD of given water sample.

## 6) Determination of chlorine in a given Bleaching powder by Iodometric method

Part A: Weigh out the given $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ crystals in to a clean 250 cm 3 volumetric flask.
Dissolved in distilled water and dilute up to the mark. Mix well to get uniform concentration.
Weight of $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ salt X 4
Normality of $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}=$
Equivalent weight of $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ (248.2)
Part B :
Burette : Standard Sodium thiosulphate solution.
Conical flask : 25 cm 3 bleaching powder solution +1 t.t dist. Water $+1 / 4 \mathrm{t} . \mathrm{t}$ Acetic acid +5 ml . of $10 \%$ KI. Titrate till pale yellow
Indicator : 2-3 drops of freshly prepared starch.
End point : Disappearance of dark blue colour.
REPORT : From the strength and volume of $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ Percentage of Chlorine is calculated.

## 7. Potentiometric Titration of FAS vs. $\mathbf{K}_{2} \mathbf{C r}_{\mathbf{2}} \mathrm{O}_{7}$.

Pipette out $25.0 \mathrm{~cm}^{3}$ of ferrous ammonium sulphate solution into a beaker. Add 2 test tube of dilute sulphuric acid. Immerse the platinum-calomel electrode assembly into the beaker containing the solution. Properly connect the electrodes to the potentiometer. add standard $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ from the semi micro burette in increments of 0.5 ml and measure the potential after each addition. Continue the procedure till a sudden rise in emf of the cell is observed. Take about 5-6 more readings
Determine the end point by plotting $\Delta \mathrm{E} / \Delta \mathrm{V}$ against the volume.


## 8. Colorimetric estimation of Copper.

Draw out $5.0,10.0,15.0,20.0$ and 25.0 ml 50 ml of given $\mathrm{CuSO}_{4}$ solution into separate, labeled $50 \mathrm{~cm}^{3}$ volumetric flasks. Add $5 \mathrm{~cm}^{3}$ of ammonia solution to each of them and also into the test solution of unknown concentration. Make up to the mark with ion exchange water. Stopper the flasks and mix the solutions well. Measure the absorbance of each of these the solutions against blank at 620 nm using a colorimeter.
Draw a calibration curve by plotting concentration of copper (in mg per $\mathrm{cm}^{3}$ ) against absorbance. Using the calibration curve determine the concentration of copper in the test solution and calculate the amount of copper in the solution.


## 9. Conductometric titration.

Run down 50 ml of acid mixture into a clean 250 ml beaker from the burette. Dip the conductivity cell in the solution. Connect the conductivity cell to the conductivity meter and measure the initial conductivity. Now add NaOH from a burette in increments of 1 ml and measure the conductivity. Plot a graph of conductivity on Y-axis v/s Volume of NaOH on X -axis


Neutralization
point of $\mathbf{H C l}$

Neutralization point of $\mathrm{CH}_{3} \mathrm{COOH}$

## 10. $\mathrm{pK}_{\mathrm{a}}$ of Weak acid.

Pipette out 25 ml of the given weak acid into a beaker. Immerse the glass electrode + calomel electrode assembly in to it. Connect the electrodes to pH meter and measure the pH . Now add NaOH from a burette in increments of 0.5 ml and measure the pH after each addition. Continue it until a sudden rise in pH is observed. Then take about 5-6 more readings.
Determine the end point by plotting $\frac{\Delta p H}{\Delta V}$ against the volume of NaOH as shown in the figure. Now Plot the graph taking the volume of NaOH along the X -axis and pH along the Y - axis then determine the pKa value from the graph.


## 11. Co-efficient of viscosity of liquid

Pipette out 20 ml of given liquid in to a wide limb of the viscometer and suck the water through the other limb. Record the time of flow between two fixed points, one above and one below the bulb in the narrow limb of viscometer. Repeat, record another two readings and take the average time of flow. Pour out the liquid, Rinse the viscometer with acetone and dry it. Now pipette 20 ml of water in to the wider limb and determine the average of time of flow for water as before. Record Lab temperature. Calculate the viscosity coefficient of the given liquid using the expression,

$$
\eta_{L}=\frac{t_{L} d_{L}}{t_{W} d_{W}} \times \eta_{W}
$$

$t_{L}, t_{w}$ - time taken by the test liquid, water chosen to flow through the same distance in the tube respectively.
$d_{L}, d_{w}$ - densities of test liquid and water respectively under same room temperature respectively.
$\eta_{\mathrm{L}}, \eta_{\mathrm{w}}$ - Viscosity co-efficient of test liquid and water respectively under same room temperature.
Thus co-efficient of viscosity of given test liquid could be found.

## 12. Flame photometry

Pipette out $2.5,5,7.5$ and 10 ml of standard NaCl solution into four different 50 ml standard flasks. Make the solutions up to the mark and shake well for uniform concentration. Take the first standard solution and aspirate into the flame of the Flame photometer. The flame emission intensity is measured by flame photometer using sodium filter ( 598 nm ). The same is repeated for all standard solutions with distilled water being sprayed to the flame in between the trials. Then aspirate the given sample of water into the flame and note down the value of emission response. Plot the graph by taking emission response along the Y axis and concentration along the X axis. Calculate the concentration of the sodium in the water sample, in the same way proceed for potassium.

## VIVA-VOCE QUESTIONS AND ANSWERS <br> GENERAL

## 1. Normality (N)

It is the no. of gram equivalents of the solute dissolved in one $\mathrm{dm}^{3}$ of the solvent.
2. Define Molarity (M)

It is the number of moles of the solute dissolved in one $\mathrm{dm}^{3}$ of the solvent.
3. Define Molality (m).

It is the number of moles of the solute dissolved in one kg of the solvent.
4. Define a mole.

A mole is the amount of substance, which contains Avogadro number $\left(6.0122 \times 10^{23}\right)$ of particles.
5. Basicity of an acid?

Basicity of an acid is the number of replaceable hydrogen present in one molecules of the acid
6. Give the Basicity of some common acids?

Basicity of $\mathrm{HCl}, \mathrm{HNO}_{3}$, and $\mathrm{CH}_{3} \mathrm{COOH}$ (acetic acid) is one.
Basicity of $\mathrm{H}_{2} \mathrm{SO}_{4}$ and $\mathrm{H}_{2} \mathrm{C}_{2} \mathrm{O}_{4}$ (oxalic acid) is two.
Basicity of $\mathrm{H}_{3} \mathrm{PO}_{4}$ is three.
7. Why the Basicity of $\mathrm{CH}_{3} \mathbf{C O O H}$ is one even through three are four hydrogen atoms in one molecule of the acid?
The Basicity is one because the number of replaceable hydrogen is only one. [The hydrogen in -COOH group is replaceable; the hydrogen in $-\mathrm{CH}_{3}$ group in not replaceable].
8. Define the equivalent weight of an acid?

Equivalent weight of an acid $=\frac{\text { Molecular weight of acid }}{\text { basicity of the acid }}$
9. Define acidity of a base?

Acidity of a base is the number of replaceable hydroxyl group present in one molecule of the base.
10. Give the acidity of some bases?

Acidity of $\mathrm{NaOH}, \mathrm{KOH}$ and $\mathrm{NH}_{4} \mathrm{OH}$ is one.

Acidity of $\mathrm{Ca}(\mathrm{OH})_{2}$ and $\mathrm{Ba}(\mathrm{OH})_{2}$ is two.
Acidity of $\mathrm{Al}(\mathrm{OH})_{3}$ is three.
11. Define Equivalent Weight of a base?

Equivalent weight of a base $=\frac{\text { Molecular weight of base }}{\text { Acidity of the base }}$
12. Given (a) Normality and (b) Gram equivalent weight, how can we find out Mass of the Substance per $\mathbf{d m}^{3}$ of solution?
Mass of substance per $\mathrm{dm}^{3}=$ Normality $x$ Gram equivalent weight.
13. How mass of substances per $\mathrm{dm}^{3}$ of solution related to Molarity and gram molecular weight.
Mass of substance per $\mathrm{dm}^{3}=$ Molarity x Gram molecular weight.
14. What are indicators?

Indicators are the substances, which indicate the end point in a titration by change of colour.
15. What are different types of indicators used in volumetric analysis?

1. Acid base indicators, e.g. Phenolphthalein.
2. Metal ion indicators, eg. Eriochrome black-T, Patton and Reader's indicator
3. Redox indicator, eg, Ferroin
4. Internal indicator eg, phenolphthalein, Eriochrome black-T, Patton and Reader's
5. External indicator, eg, potassium ferricynide
6. Self indicator, eg, Potassium permanganate
7. Define the equivalent of an oxidizing agent?

It is the number of parts by mass of the oxidizing agent which contains 8 parts by mass of available oxygen for oxidation. [Gram equivalent mass of an oxidizing agent may be defined as the mass of the substance which can accept one mole of electron]
17. What is a standard solution?

A standard solution is one, which contains a known mass of the solute dissolved in a known volume of the solvent.

## DETERMINATION OF pKa VALUE OF WEAK ACID

1. What is a weak acid?

A weak acid is an acid, which ionizes to a small extent in solution.
2. Give an example for a weak acid?

Acetic acid $\mathrm{CH}_{3} \mathrm{COOH}$, Formic acid HCOOH .
3. What are the ions formed by the dissociation of acetic acid?

Dissociation of acetic acid gives acetate $\left(\mathrm{CH}_{3} \mathrm{COO}^{-}\right)$ions and hydrogen ions $\left(\mathrm{H}^{+}\right)$ $\mathrm{CH} 3 \mathrm{COOH} \longrightarrow \mathrm{CH}_{3} \mathrm{COO}^{-}+\mathrm{H}^{+}$
4. What are strong acids?

A strong acid is an acid, which ionizes completely in solution
5. Give example for strong acids

Strong acids: $\mathrm{HCl}, \mathrm{HNO}_{3}$, and $\mathrm{H}_{2} \mathrm{SO}_{4}$
6. What is Ka?

Ka is the dissociation constant of weak acid. It is given by the equation
$\mathrm{Ka}=\frac{\left[\mathrm{CH}_{3} \mathrm{COO}^{-}\right]}{\left[\mathrm{CH}_{3} \mathrm{COOH}\right]} \underline{\left[\mathrm{H}^{+}\right]}$
7. What is pKa of weak acid?
pKa of weak acid is the negative logarithm to base 10 of dissociation constant of the
weak acid $. \mathrm{pKa}=-\log _{10} \mathrm{Ka}$
8. What is pH ?
pH is the negative logarithm of base 10 of molar concentration of hydrogen ions i.e. $\mathrm{pH}=-\log _{10}\left(\mathrm{H}^{+}\right)$
9. How are $\mathbf{p H} \& \mathbf{p K a}$ related?

They are related by Henderson- Haselbalch equation, which is given by

$$
\mathrm{pH}=\mathrm{pKa}+\log \frac{[\text { Salt }]}{[\text { Acid }]}
$$

Where [salt] \& [acid] are molar concentration of salt \& the acid respectively
10. What are the electrodes used in the measurement of pH ?

Glass electrode - Calomel electrode assembly is used in the measurement of pH .
11. How pH becomes equal to pKa at half equivalence point?

At half equivalence point molar concentration of salt is equal to molar concentration of the acid i.e. $($ salt $)=($ acid $)$

Therefore $\log ($ salt $) /($ acid $)=\log 1=0$
Thus $\mathrm{pH}=\mathrm{pKa}+0=\mathrm{pKa}$

## 12. Explain $\mathbf{p H}$ scale

$\mathrm{pH}=0-7$ : Acidic: $\mathrm{pH}=7-14$ : Basic; $\mathrm{pH}=7$ : Neutral
13. Why does $\mathbf{p H}$ increase suddenly at the equivalence point?

At the equivalence point, the base has neutralized all the weak acid. Afterwards, the concentration of hydroxyl ions increases resulting in a sudden increase of pH .
14. How are Ka \& strength of weak acid related?

Higher the Ka, stronger is the acid.
15. How are pKa \& strength of weak acid related?

Higher the pKa , weaker is the acid.
16. Why glass electrode called an ion selective electrode?

The glass electrode is called an ion selective electrode because, it is able to respond to certain specific ions ( $\mathrm{H}^{+}$ions) only and develop a potential while ignoring the other ions in a solution.

## POTENTIOMETRIC TITRATION OF FAS VS K $2_{2} \underline{C R}_{2} \underline{\mathrm{O}}_{7}$

1. What is Potentiometric titration?

The determination of the equivalent point of redox titration on the basis of potential measurement is called a Potentiometric titration.
2. What are the electrodes used in the determination of FAS potentiometrically? The indicator electrode used is the platinum electrode (acts as anode) and the reference electrode used is the calomel electrode (acts as cathode).
3. Give the principle of Potentiometric titration

The principle in Potentiometric titration is the measurement of the emf between two electrodes, an indicator electrode and a reference electrode. In these titrations, measurements of emf are made while the titration is in progress. The equivalence point of the reaction is revealed by a sudden change in potential in the plot of emf readings against the volume of titration.
4. What is an indicator electrode?

The electrode whose potential is dependent upon concentration of the ions in solution is termed as indicator electrode. eg: platinum electrode .
5. What is the full form of FAS?

The full form of FAS is ferrous ammonium sulphate.
6. What is the reaction occurs between FAS and $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ in Potentiometric titration? Acidified $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ oxidizes ferrous sulphate (FAS) to ferric sulphate. $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ itself gets reduced to chromic sulphate.
7. What is the determining factor in the oxidation- reduction reaction?

The determining factor is the ratio of the concentration of the oxidation and reduction forms i.e. $\mathrm{Fe}^{2+} \leftrightarrow \mathrm{Fe}^{3+}+\mathrm{e}^{-}$
8. Why is $\mathrm{H}_{2} \mathrm{SO}_{4}$ added to FAS solution during emf measurement?

Reaction between FAS and $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ is a redox reaction, where oxidizing agent $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ oxidizes $\mathrm{Fe}^{2+}$ to $\mathrm{Fe}^{3+}$ only in the presence of acidic medium.
9. What is the oxidation state of iron in FAS?

It is +2

## 10. Why the emf is rises steeply after the equivalent point?

This is because, the potential of the solution before the equivalence point is determined $\mathrm{Fe}^{2+} \rightarrow \mathrm{Fe}^{3+}$ by system only i.e., 0.75 V , while at equivalence point, it is determined by both $\mathrm{Fe}^{3+}$ and $\mathrm{Cr}_{2} \mathrm{O}_{7}{ }^{2-}$ ions which is $=1.04 \mathrm{~V}$. But beyond equivalence point, the potential of the solution is determined by $\mathrm{Cr}_{2} \mathrm{O}_{7}^{2-} / \mathrm{Cr}^{3+}$ only i.e. $=1.33 \mathrm{~V}$. Therefore, just after the equivalence point, the potential of the solution raises steely.
11. What is single electrode potential?

The potential that is developed when an electrode is in contact with a solution containing its own ions is called single electrode potential.
12. What is emf?

The emf is the potential difference required to drive a current across the electrodes.

## COLORIMETRIC DETERMINATION OF COPPER

1. What is Colorimetry?

Chemical analysis involves measurement of absorption of light in the visible region is known as Colorimetry.
2. What is the range of wavelengths of light in the visible region? 400 nm to 700 nm . ( $\mathrm{nm}=$ nano meter $=10^{-9}$ meter )
3. What forms the basis for the colorimetric determination?

The variation of colour of a system with change in concentration of some component forms the basis for the colorimetric determination.
4. What is photoelectric colorimeter?

It is an instrument, which measures the amount of light absorbed using a photocell
5. What is the basic principle of colorimetric measurements?

It consists of comparing under well defined conditions, the colour produce by the substance in unknown amount with the same colour produced by a known amount of the material being determined.
6. Why filters are used in colorimetric experiment?

Filters are used for selecting desired spectral region
7. What is wavelength?

The distance between any two successive peaks or troughs of wave is the called wavelength. It is represented by $\lambda$. It can be expressed meters.
8. What is wave number?

Wave number is reciprocal of wavelength. It is the number of waves present in one meter length. It can be expressed in $\mathrm{m}^{-1}$
9. What is the wavelength at which copper estimation is done?

At 620 nm
10. What is frequency?

It is the number of waves passing through a point second. It is represented by " $v$ " It is expressed in Hz .
11. State Beer's law.

The intensity of a beam of monochromatic light decreases exponentially as the concentration of the absorbing substance increases arithmetically.

## 12. State Lamberts law.

The intensity of the transmitted light decreases exponentially as the thickness of the absorption medium increases arithmetically.
13. State Beer- Lamberts law.

The amount of light absorbed is directly proportional to the concentration (c) of the solution and directly proportional to the path length (1)
$\mathrm{A}=\mathrm{Ccl}$
Where, $\mathrm{C}=$ molar extinction coefficient.

## 14. What is a calibration curve?

It is the plot of absorbance against concentration of solutions. For solutions obeying Beer's law, this is a straight line.
15. What is transmittance?

It is the ratio of the intensity of transmitted $\operatorname{light}\left(\mathrm{I}_{\mathrm{t}}\right)$ to that of the incident $\operatorname{light}\left(\mathrm{I}_{\mathrm{o}}\right) . \mathrm{I}_{\mathrm{t}}$ / $\mathrm{I}_{\mathrm{o}}=\mathrm{T}$

## 16. Why are different volumes of solution taken in the flasks?

Difference volumes of solution are taken to prepare standard solutions of different concentration, which are used to plot a calibration curve.

## 17. What is blank solution?

Blank solution is used to calibrate the apparatus.
18. Why is blank solution used in colorimetric estimation?

To nullify absorbance caused due to the coloring impurities present in the reagents.
19. Why is ammonia added? Why is the same amount of ammonia added to different volume of $\mathrm{CuSO}_{4}$ solution?
Ammonia is added to increase the intensity of color (due to $\left[\mathrm{Cu}\left(\mathrm{NH}_{3}\right)\right] \mathrm{SO}_{4}$, a dark blue complex). Same amount ammonia is added to nullify the absorbance due to any coloring impurities present in ammonia.

## 20. Why is estimation of copper done at 620 nm wavelength?

The estimation of copper is carried at 620 nm wavelength because; the $\left[\mathrm{Cu}\left(\mathrm{NH}_{3}\right)_{4}\right]$ $\mathrm{SO}_{4}$ complex shows a maximum absorbance at 620 nm .

## CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE

1. State ohm's law.

Ohm's law states that the current, I (ampere), flowing in a conductor is directly proportional to the applied emf, E (volt) and inversely proportional to the resistance, R (ohm) of the conductor.
2. What is conductance?

The reciprocal of resistance is called conductance
3. What is conductivity?

The reciprocal of resistivity is called conductivity.
4. What is the unit of conductance?

The unit of conductance is ohm ${ }^{-1}$ or S .
5. Mention the different types of conductance?

A solution may have the following conductance.

1. Specific conductance
2. Equivalent conductance.
3. Molar conductance.
4. Which of the above conductance measured during conductometric titration?

The specific conductance is measured.
7. What is specific conductance?

It is the conductance of a solution placed between two electrodes of $1 \mathrm{~cm}^{2}$ area and kept 1 cm apart.
8. What is equivalent conductance?

It is the conductance of the solution, which contains 1-gram equivalent of solute, when placed between two electrodes of 1 cm apart.
9. What is molar conductance?

It is the conductance of a solution, which contains I gram molecular weight of a substance, when placed between two electrodes of 1 cm apart.
10. What is cell?

A device, which produces an electromotive force and delivers an electric current as the function.
11. What is the principle involved in conductometric titration?

In conductometric titration, there is a sudden change in conductance of a solution near the end point. Hence the end point is determined graphically by plotting conductance against titre values. The principle underlying conductometric titration is the replacement of ions of a particular conductance by ions of different conductance during the titration.
12. What factor determines the conductance of a solution?

Two factors determining the conductance of a solution are. Mobility of ions- higher the mobility, higher is the conductance and
Number of ions- more the number of ions in solution more is the conductance.
13. In the titration of HCl with a strong base $(\mathbf{N a O H})$, the conductance decreases then increases steeply. Why?
Upon adding a strong base to a strong acid, the conductance falls due to the replacement of highly mobile $\mathrm{H}^{+}$ions of the strong acid $(\mathrm{HCl})$ by less mobile $\mathrm{Na}^{+}$ions of the base. The conductance falls till all the $\mathrm{H}^{+}$ions are replaced (i.e. till HCl is neutralized completely). The conductance then rises steadily as the weak acid $\left(\mathrm{CH}_{3} \mathrm{COOH}\right)$ is converted into its salt. Finally, the conductance rises steeply as excess of alkali $\left(\mathrm{OH}^{-}\right.$ ions) is introduced, i.e., due to highly mobile $\mathrm{OH}^{-}$ions.

## FLAME PHOTOMETRIC DETERMINATION OF SODIUM

1. What is the basis of flame photometry?

It is based on the measurement of intensity of the light emitted when a color is introduced into a flame. The wavelength of the color tells us the element is and the colors intensity tells us how much of the element is present.
2. Why the flame photometry is also called flame emission spectroscopy?

Because of the use of a flame to provide the energy of excitation to atoms introduced into the flame.
3. Give the sequence of events that occur in flame photometry.

Liquid sample containing element aspirated into a flame $\rightarrow$ Formation of the liquid droplets $\rightarrow$ Evaporation of droplets resulting in the formation of residue $\rightarrow$ Decompose of residue into neutral atoms $\rightarrow$ Formation of excited atoms and emission of radiation from atoms and emission of radiation from atoms $\rightarrow$ wavelength and intensity of emitted radiation measured by flame photometry.
4. What are the applications of flame photometry?
a). Qualitative analysis - used to detect elements of groups I \& II of the periodic table.
b). Quantitative analysis- used for the rapid quantitative determination of the elements in-group I \& II of the periodic table.
c). Carry out simultaneous analysis.
d). Used in the analysis of biological fluids and tissues. In these elements like $\mathrm{Na}, \mathrm{k}, \mathrm{Al}$, $\mathrm{Ca}, \mathrm{Co}$, and Fe are detected. Also used for determining $\mathrm{Na}, \mathrm{k}, \mathrm{Al}, \mathrm{Ca}, \mathrm{Co}$, and Fe in soil analysis.
5. What are the factors that influence the intensity of emitted radiation in a flame photometer?
a). Viscosity - The addition of a substance which increase the viscosity of the Solution (e.g. Sucrose) decreases the intensity of the light emission. This decreases results, due to a reduction in the efficiency of atomization.
b). Presence of acids decreases the light intensity. This decrease arises due to the disturbance of the initial dissociation equilibrium.
c). Presence of other metals alter the intensity of emitted radiation.
6. What are the limitations of flame photometry?
a). It does not provide information about the molecular form of the metal present in the original sample.
b). cannot be used for the detection of the halides or the inert gases.
c). only liquid samples may be used.
d). only a few elements can be analyzed.
7. What are the advantages of this spectroscopy?
a). Well understood technique.
b). Low running cost and maintenance costs.
c). Measurements is possible in a wide range of fluid systems.

## DETERMINATION OF VISCISITY CO-EFFICIEN OF A GIVEN LIQUID USING OSTWALD'S VISCOMETER

1. What is viscosity?

Viscosity arises due to internal friction between moving layers of molecules. A moving layer exerts a drag or friction on its nearest moving layer backward. This property of a liquid by which it retards or opposes motion between the layers is called viscosity.
2. What is viscosity co-efficient of a liquid?

The viscosity co-efficient of a liquid is defined as the tangential force per unit area required maintaining a unit velocity gradient between any two successive layers of a liquid situated unit distance apart.
3. What is density of liquid?

The density of a liquid is its mass/ its volume.
4. The density of a substance is expressed relative to what?

The density of a substance is expressed relatively to that of water at $4^{\circ} \mathrm{C}$.
5. What is SI unit of viscosity co-efficient?

The viscosity co-efficient is expressed as $\mathrm{kg} / \mathrm{m} . \mathrm{s}$.
6. What is the equation used to represent the influence of temperature on viscosity?

The influence of temperature on viscosity is best represented by an empirical equation $\eta$ $=A e^{B / R T}$ where $A$ and $B$ are constants for a given liquid
7. What are the factors that influence the viscosity of a liquid?

1. Increase in molecular weight results in an increase in viscosity.
2. Branched chain compounds have higher viscosity than those involving straight chain compounds.
3. The polar compounds are more viscous than the polar ones. The presence of hydrogen bonds causes the viscosity to increase.
4. Temperature has marked influences by the viscosity of a liquid.
5. What is the law base on the viscous flow of liquid through capillary tubes?

The law based in the viscous flow of liquid through capillary tubes Poiseuilles
law. It is expressed as $\eta=\frac{\pi \mathrm{pr}^{4} \mathrm{t}}{8 \mathrm{vl}}$
Where $\eta=$ Viscosity co-efficient; $p=$ Hydrostatic pressure; $r=$ Radius of the tubes; $t=$
Time required for the volume; $\mathrm{v}=$ of the liquid to flow through the tube of the length, 1 .
9. How does the viscosity vary with temperature?

The viscosity of a liquid usually decreases with the rise of temperature.
10. Why should the viscometer be dried before the measurements are done?

The viscometer should be dried to avoid formation of an emulsion, which changes the rate of flow of the liquid.
11. Why acetone used for cleaning viscometer?

Acetone is a volatile organic liquid. To dry the viscometer quickly, it is rinsed with acetone.
12. Why viscometer not rinsed with the given liquid or water?

If the viscometer is rinsed with the given liquid or water before measuring the flow times the volume taken will be more than a definite known volume.
13. Why do require laboratory temperature for viscosity determination?

Because, the physical constants like density and viscosity of a liquid vary with temperature.
14. How is the viscosity of a liquid related to its mobility?

Viscosity of a liquid is inversely proportional to its mobility.
15. Why is viscometer dipped in water bath?

Viscometer is dipped in water bath to maintain constant temperature.

## DETERMINATION OF TOTAL HARDNESS WATER

1. What is hard water?

Hard water is water, which does not give lather with soap easily and also produces scale in hot water pipes, heaters, boilers, etc.
2. How are the water classified based on the degree of hardness?

Soft water $\quad 0-75 \mathrm{mg} /$ liter
Moderately hard water $75-150 \mathrm{mg} / \mathrm{liter}$
Hard water $\quad 150-300 \mathrm{mg} / \mathrm{liter}$
Very hard water Greater than $300 \mathrm{mg} /$ liter
3. How is hardness of water caused?

Hardness of water caused by divalent metallic cations in association with anions such as $\mathrm{HCO}_{3}{ }^{-}, \mathrm{SO}_{4}{ }^{2-}, \mathrm{Cl}^{-}$etc. The principle hardness causing cations are calcium and magnesium ions.
4. How the bicarbonates are introduced into water although Ca (or $\mathbf{M g}$ ) is in the insoluble form in the nature?
Limestone and Dolomite consist of carbonates of calcium and magnesium, which are insoluble water. The rainwater containing CO 2 when comes in contact with these carbonates form soluble bicarbonates.
5. How is hardness in water classified?

Hardness in water is classified as i). Temporary hardness and ii). Permanent hardness.
6. What is the difference between temporary and permanent hardness?

Temporary hardness is due to unstable bicarbonates of Ca and Mg , while permanent hardness is due to more stable $\mathrm{Cl}^{-}$and $\mathrm{SO}_{4}{ }^{2-}$ of Ca and Mg .
7. How is temporary hardness removed?

The temporary hardness of water can be removed by simple boiling water during which bicarbonates decompose to give insoluble carbonates.
$\mathrm{Ca}\left(\mathrm{HCO}_{3}\right)_{2} \xrightarrow{\Delta} \mathrm{CaCO}_{3} \downarrow+\mathrm{H}_{2} \mathrm{O}+\mathrm{CO}_{2}$
8. What do you mean by total hardness water?

The sum of the both temporary and permanent hardness is called the total hardness of water.
9. How express the total hardness of water?

Total hardness is expressed in terms of parts per million (ppm) of $\mathrm{CaCO}_{3}$.
10. What is the full form of EDTA?

EDTA is Ethylene diamine tetra acetic acid.
Department of Chemistry, K. S. I. T, Bangalore-109

## 11. Give the structure of EDTA.


12. Why is disodium salt of EDTA preferred to EDTA?

EDTA is sparingly soluble in water. Its disodium salt is more soluble as it is ionisable and hence it is preferred.
13. Why is ammonia solution added while preparing EDTA solution?

Ammonia solution is added during the preparation of EDTA solution to increase the rate of dissolution of the salt.
14. What is buffer solution?

The solution, which resists changing in its pH value even after adding small amount of an acid or a base to it, is called a buffer solution.
15. Why is ammonia-ammonium chloride buffer added?

Ammonia-ammonium chloride buffer is added to maintain a pH of 10 , the desired pH for the titration. Otherwise, pH decreases as $\mathrm{H}^{+}$ions are released due to the substitution of metal ions for $\mathrm{H}^{+}$ions in EDTA.
16. What is the chemical name of Eriochrome Black T?

The chemical name of Eriochrome Black-T is sodium 1- (1- hydroxyl-2-naphthylazo)-6-nitro-2-naohthol-4-sulphaonate SHNNNS).
17. Why is the indicator Eriochrome Black-T (EBT) shows wine red colour at the beginning and blue colour at the titration?
When a small amount of Eriochrome Black-T, which is blue in colour, is added to a hard water with a pH of about 10 , it combines with a few calcium and magnesium ions to form a weak complex which is wine red in colour as shown in the below.

$$
\begin{aligned}
& \mathrm{M}^{\mathrm{n}+}+\underset{\mathrm{EBT}}{ } \rightarrow \mathrm{M}-\mathrm{EBT} \\
& \text { Blue } \quad \text { wine red complex }
\end{aligned}
$$

During the titration with EDTA, all free hardness ions are complexed according to the equation,

$$
\mathrm{M}+\mathrm{EBT}+\mathrm{EDTA} \rightarrow \underset{\text { Stable complex }}{\mathrm{M}-\mathrm{EDTA}}+\underset{\text { blue }}{\mathrm{EBT}}
$$

This action frees the Eriochrome Black-T indicator from the complex, (M-EDTA), and the wine red colour changes to a blue colour at the end of the titration.
18. Why are the titrations involving EDTA carried out slowly towards the end point? The titrations involving EDTA are carried out slowly towards the end point, because, the rate of formation of the metal complex of EDTA is very slow.
19. What is the application of hardness data in environmental engineering practice? Hardness of water is an important consideration in determining the suitability of water for domestic and industrial uses.
Determination of hardness serves as a basic for routine control of softening processes.

## DETERMINATION OF CaO IN CEMENT SOLUTION

1. What is cement?

Cement is a lime based building material used to bind together caresses aggregates.
2. What are the constituents of cement?

Oxides of calcium, magnesium, iron, Aluminium and silicon are the constituents of cement.
3. Mention the general composition of Portland cement.
$\mathrm{CaO}=60-66 \%, \mathrm{SiO}_{2}=17-25 \%$
$\mathrm{Al}_{2} \mathrm{O}_{3}=3-8 \%, \mathrm{Fe}_{2} \mathrm{O}_{3}=2-6 \%, \mathrm{MgO}=0.1-5.5 \%, \mathrm{Na}_{2} \mathrm{O}$ and $\mathrm{K}_{2} \mathrm{O}=0.5-1.5 \%$
4. What is the prime constituent of cement?

Calcium oxide is the prime constituent of cement. Because the quality of cement depends on $\%$ of CaO .
5. How cement solution is prepared?

Accurately weighed amount of cement is warmed with moderately conc. HCl till cement dissolves. Insoluble silica is filtered off the filtrate is the cement solution.
6. What is the role of glycerol?

Glycerol is added to get sharp end point.
7. What is the function of diethylamine?

Diethylamine is added to maintain a pH of about 12.5 .
8. Which indicator is used in determination of $\mathbf{C a O}$ in cement solution? And why? Patton and Reeder's indicator is used in the determination of CaO in cement solution as this indicator gives a sharp colour changes in the pH ranges 12-14.
9. Why Eriochrome Black-T indicator is cannot be used in this experiment?

1. Eriochrome Black-T indicator cannot be used in this experiment, because, it forms a very weak complex with calcium ions in the pH range 10-11 only.
2. EBT used to determine both $\mathrm{Ca} \& \mathrm{Mg}$ (Total hardness). Whereas Patton and Reeder's indicator only for Calcium.
3. What is the chemical name of Patton and Reeder's indicator?

The chemical name of Patton and Reeder's indicator is 2-hydroxy-1-(2-hydroxy-4-sulpho-1-naphthylazo)-3-naphthoic acid (HHSNNA).
11. Why this titration is called Rapid EDTA method?

The titration is called rapid EDTA method because, by this method, $\mathrm{Ca}^{2+}$ ions in cement solution are estimated directly and quickly by using standard EDTA solution without removing the other metal ions.
12. What is the role of $\mathbf{N a O H}$ in this titration?

NaOH reacts with $\mathrm{Mg}^{2+}$ ions and precipitates it out in the form of $\mathrm{Mg}(\mathrm{OH})_{2}$ from the cement solution. Calcium \& Magnesium both are having a same chemical and physical property that's why we are masking only Mg.

> DETERMINATION OF COPPER IN BRASS

1. What is brass?

Brass is an alloy of copper and zinc.
2. What is an alloy?

An alloy is a homogeneous mixture of two or more metals.
3. Give other examples for an alloy of copper?

Bronze (this is an alloy of copper and tin)
4. What are the main constituents of brass?

Copper (50-60\%) and Zinc (20-40)
5. How is Brass dissolved?

Brass is dissolved in hot nitric acid.
6. What is the reaction occurring when brass is dissolved in nitric acid?

Copper undergoes oxidation from element state to cupric $\left(\mathrm{Cu}^{2+}\right)$ state. A part of nitric acid undergoes reduction to nitrogen dioxide.

$$
\mathrm{Cu}+4 \mathrm{HNO}_{3} \rightarrow \mathrm{Cu}\left(\mathrm{HNO}_{3}\right)_{2}+\mathrm{NO}_{2}+2 \mathrm{H}_{2} \mathrm{O}
$$

7. What is the colour of $\mathbf{N O}_{\mathbf{2}}$ gas?

Reddish brown.
8. What is the oxidation state of copper in brass?

Zero.
9. What is the oxidation state of copper in cupric nitrate?

2+
10. Why is urea added to the brass solution?

Urea is added to destroy excess of nitrous acid and oxides of nitrogen, which interfere with the determination. If they are not destroyed, they also oxides KI to $\mathrm{I}_{2}$, which should have not done by $\mathrm{Cu}^{2+}$ ions.
11. Why is ammonium hydroxide added to the brass solution?
$\mathrm{NH}_{4} \mathrm{OH}$ is added to neutralize any acid present in the solution.
12. What is the need to neutralize the mineral acid?

The mineral acid, $\mathrm{HNO}_{3}$ is a powerful oxidizing agent. It also oxidizes KI to $\mathrm{I}_{2}$.
Therefore the amount of $\mathrm{I}_{2}$ liberated does not correspond to the exact concentration of $\mathrm{Cu}^{2+}$ ions and hence erroneous result.
13. Why is acetic acid added to the brass solution?

Acetic acid is added to neutralize the excess of ammonium hydroxide and to make the solution slightly acidic.
14. Why is potassium iodide added to the brass solution although copper in brass is determined?
Cupric ions do not react with sodium thiosulphate solution. However, cupric ions oxidize KI and iodine is liberated. The amount of iodine liberated is equal to the amount of cupric ions in the solution.
15. What is the reaction that occurs between cupric ions and potassium iodide?

It is a redox reaction.

$$
2 \mathrm{Cu}^{2+}+4 \mathrm{KI} \rightarrow \mathrm{Cu}_{2} \mathrm{I}_{2}+4 \mathrm{~K}^{+}+\mathrm{I}_{2}
$$

Copper is reduced from cupric state ( $2+$ state) to cuprous state ( $1+$ state). Iodine is oxidized from -1 state to elemental (zero) state.
16. What is the name of $\mathrm{Cu}_{2} \mathrm{I}_{2}$ ?
$\mathrm{Cu}_{2} \mathrm{I}_{2}$ is cuprous iodide.
17. What is the oxidation state of copper in cuprous iodide?

It is +1 .
18. What is the colour of cuprous iodide?

White.
19. Is cuprous iodide soluble in water?

No, that is why it appears as a precipitate.
20. Why does the blue solution of brass turn brown after adding potassium iodide? Iodide is librated, which forms a brown colored solution after dissolving in excess KI.
21. Which is the indicator used in determination of copper in brass?

Starch indicator.
22. Why is starch added towards the end of the titration?

Starch should be added only when the concentration of iodide is low. If we add starch when concentration of iodide is high, starch forms stable complex with iodine. This makes the detection of end point difficult.
23. What is the white precipitate left after the end point?

The white precipitate is $\mathrm{Cu}_{2} \mathrm{I}_{2}$ (cuprous iodide) already present.
24. How is iodine estimated?

The amount of iodine is estimated by titrating it against standard sodium thiosulphate $\left(\mathrm{NaS}_{2} \mathrm{O}_{3}\right)$ solution using starch as indicator.
25. What is the reaction that occurs between iodine and sodium thiosulphate?

Iodine reacts with sodium thiosulphate to form sodium iodide and sodium tetrathionate. $2 \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}+\mathrm{I}_{2} \rightarrow \mathrm{Na}_{2} \mathrm{~S}_{4} \mathrm{O}_{6}+2 \mathrm{NaI}$

DETERMINATION OF IRON IN HAEMATITE ORE

1. What is the main constituent of haematite ore?

The main constituents of haematite ore is $\mathrm{Fe}_{2} \mathrm{O}_{3}$
2. What other constituent is present in haematite ore? Silica $\left(\mathrm{SiO}_{2}\right)$.
3. What is the oxidation state of iron in haematite ore? +3
4. What is the role of stannous chloride $\left(\mathbf{S n C l}_{2}\right)$ in the determination of iron in haematite ore?
Stannous chloride acts as a reducing agent. It reduces $\mathrm{Fe}^{3+}$ to $\mathrm{Fe}^{2+}$ i.e. ferric to ferrous. $2 \mathrm{FeCl}_{3}+\mathrm{SnCl}_{2} \rightarrow 2 \mathrm{FeCl}_{2}+\mathrm{SnCl}_{4}$.
5. What happens to stannous chloride in the above reaction?

Stannous chloride ( +2 ) is oxidized to stannic chloride ( +4 ).
6. What happens when the excess of stannous chloride is not removed?

If the excess of stannous chloride is not removed, the ferric ions formed during the course of the titration get reduced to ferrous ions. As a result, the volume of titrant, potassium dichromate consumed will be more
7. Why is mercuric chloride added?

Mercuric chloride is added to remove excess stannous chloride. Mercuric chloride oxidizes stannous chloride to stannic chloride. It gets reduced to mercurous chloride. $\mathrm{SnCl}_{2}(2+)+2 \mathrm{HgCl}_{2}(2+) \rightarrow \mathrm{Hg}_{2} \mathrm{Cl}_{2}(1+)+\mathrm{SnCl}_{4}(4+)$.
8. Is mercurous chloride soluble in water?

No. It is not soluble. That is why it appears as a precipitate, which is silky white in colour.
9. What is the oxidation state of tin is stannous chloride $\left(\mathbf{S n C l}_{2}\right)$ ?
$+2$
10. What is the oxidation state of tin is stannic chloride? $+4$
11. What is the oxidation state of mercury in mercuric chloride $\left(\mathbf{H g C l}_{\mathbf{2}}\right)$ ? +2
12. What is the oxidation state of mercury in mercurous chloride $\left(\mathbf{H g}_{2} \mathbf{C l}_{\mathbf{2}}\right)$ ?
$+1$
13. Which indicator is used in the determination of iron in haematite ore?

Potassium ferricynide, $\left[\mathrm{K}_{3} \mathrm{Fe}(\mathrm{CN})_{6}\right]$, is used as an external indicator.
14. What is the colour of $\left[\mathrm{K}_{3} \mathrm{Fe}(\mathrm{CN})_{6}\right]$ with ferrous ion?

Deep blue colour.
15. What is the compound with deep blue colour?

Ferrous ferricynide complex
16. Why the colour of indicator drops is remains unchanged at the end point?

Only ferrous ions form a blue colour with potassium ferricynide. Since there are no $\mathrm{Fe}^{2+}$ ions left in the conical flask at the end point, no blue colour is formed. In the conical flask, at the end point, $\mathrm{Fe}^{3+}$ ions are present, but they do not give colour with potassium ferricynide.
17. What happens to $\mathrm{Fe}^{2+}$ when you titrate $\mathrm{Fe}^{\mathbf{2 +}}$ solution with $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ ? $\mathrm{Fe}^{2+}$ is oxidized to $\mathrm{Fe}^{3+}$ by potassium dichromate.
18. What is the reaction that occurs during titration? Acidified potassium dichromate oxidizes ferrous ions present in the haematite ore solution to ferric state. It itself gets reduced to chromic ion.

$$
6 \mathrm{Fe}^{2+}+\mathrm{Cr}_{2} \mathrm{O}_{7}+14 \mathrm{H}^{+} \rightarrow 6 \mathrm{Fe}^{3+}+2 \mathrm{Cr}^{3+}+7 \mathrm{H}_{2} \mathrm{O}
$$

19. Why is potassium ferricynide not used as an internal indicator?

Potassium ferricynide cannot be used as an internal indicator in the determination of iron in haematite because, potassium ferricynide combines irreversibly with ferrous ion to form a deep blue ferrous ferricynide (Turnbull's) These ferrous ions involved in complex formation are not available for reaction with potassium dichromate. Moreover, the end point cannot be detected, as there is no colour change.
$3 \mathrm{Fe}^{2+}+2 \mathrm{~K}_{2}\left[\mathrm{Fe}(\mathrm{CN})_{6}\right] \rightarrow \mathrm{Fe}_{3}\left[\mathrm{Fe}(\mathrm{CN})_{6}\right]_{2}+4 \mathrm{~K}^{+}$
20. Why can't we use potassium ferrocynide as an indicator?

Potassium ferrocynide does not react with ferrous ions.
DETERMINATION OF COD OF WASTE WATER

1. What is meant by industrial sewage?

The waste water coming out from the industrial establishments such as chemical plants, fertilizer industries, leather tanneries, sugar and paper industries, bewereries, textiles mills, oil refineries, pharmaceutical units is called as industrial sewage.
2. What is the full form of COD?

COD stands for chemical oxygen demand.
3. What is chemical oxygen demand?

It is the amount of oxygen required for oxidation of biologically and non-biologically oxidizable organic compound present in $1 \mathrm{dm}^{3}$ of waste water by a strong oxidizing agent such as acidified potassium dichromate.
4. What is full form of BOD?

BOD stands for biological oxygen demand.
5. What is biological oxygen demand?

It is the amount of oxygen requires for the biological oxidation of organic compounds present in $1 \mathrm{dm}^{3}$ of wastewater over a 5 days period at $20^{\circ} \mathrm{C}$.
6. What is the role of silver sulphate in the determination of COD?

Silver sulphate acts as catalyst in the oxidation of straight chain aliphatic hydrocarbons and acetic acid. Oxidation is effective in presences of silver ions.
7. What is the role of mercuric sulphate in the determination of COD?

Chloride ions normally present in high concentration in waste water undergo oxidation in COD test and cause erroneous high results. Mercuric ions of mercuric sulphate bind the halide ions present in water to form poorly ionized mercuric chloride and prevent the formation of AgCl precipitate by making halide ions unavailable.
8. What are the oxidizable impurities present in waste water?

Waste water contains organic impurities such as straight chain aliphatic compounds, straight chain alcohols, aromatic compounds etc.
9. What is the indicator used in COD experiment?

Ferroin indicator (Ferrous 1, 10 phenanthroline sulphate)

## 10. What is the colour at the end point?

Blue green to reddish brown
11. Why the sulphuric acid is added during the preparation of standard FAS solution?Sulphuric acid is added to prevent the hydrolysis of ferrous sulphate into ferrous hydroxide.
12. What is the role of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ in COD analysis?

Potassium dichromate oxidizes the organic matter present in waste water.
13. What are the products formed on oxidation of organic matter?
$\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ oxidizes organic matter to $\mathrm{CO}_{2}$ and $\mathrm{H}_{2} \mathrm{O}$
14. Which organic compounds are not oxidized in COD analysis?

Aromatic hydrocarbons and pyridine are not oxidized in COD test.
15. Mention a few application of COD analysis?

The COD test is used in analysis of industrial waste water.
The COD test gives the pollution level of industrial waste water.
16. Among COD and BOD, which is higher in value?

COD is higher in value
17. What is the unit in which COD is expressed?

COD is expressed in mg of oxygen per $\mathrm{dm}^{3}$ of waste water.

## Determination of chlorine.

1. What is Disinfection?

Disinfection means Killing of disease producing bacteria and other microorganisms
2. Why Bleaching powder is mixed in water?

Bleaching powder is mixed in water for Disinfection of water
3. What is the meaning of Residual chlorine

It means the Chlorine available after completion of the disinfection
4. What is Potable water?

Water free from disease producing elements and bacteria.
5. How much residual chlorine should be present in water?
$0.2 \mathrm{mg} /$ litre
6. How Residual chlorine is detected in water

Residual chlorine is detected by Orthotolidine
7. Chlorine is often added to wastewater for disinfection before effluent discharge
A potential problem with this procedure is toxic chlorinated hydrocarbons may be formed.
8. Water requires how much of chlorine to destroy the microorganisms present.
$2 \mathrm{mg} / \mathrm{L}$
9. At what pH Chlorination is effective?
6.8 to 7.2
10. At what pH titration should be carried out?
pH3-4
11. How Chlorine is made commercially?

Chlorine is made commmercially from sodium chloride
12. How the active ingredient in many laundries bleaches?

The active ingredient in many laundry bleaches is sodium hypochlorate
13. The Moist litmus paper placed in chlorine gas turns blue litmus paper to which colour?

Moist litmus paper placed in chlorine gas turns blue litmus paper red.

## Scheme of Valuation

Subject: Engineering Chemistry Lab Code: 18CHEL16/18CHEL26

| Description |  |  |  | Max. marks |  | Part A marks |  |  | Part B marks |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Procedure write-up |  |  |  | 15 |  | 08 |  |  | 07 |  |  |
| Conduction |  |  |  | 52 |  | 26 |  |  | 26 |  |  |
| Calculation, Graph works \& Result |  |  |  | 18 |  | 09 |  |  | 09 |  |  |
| Viva-Voce |  |  |  | 15 |  | 07 |  |  | 08 |  |  |
| Part-A INSTRUMENTAL |  |  |  |  |  | Part-B VOLUMETRY |  |  |  |  |  |
| Potentiometry, Colorimetry and Flame Photometry |  | pKa$\&$Viscosity |  | Conductometry |  | Total hardness, CaO in cement, Cu in Brass, Fe in Haematite and COD |  | Alkalinity of Water |  |  |  |
|  |  | Phenolphthalene Methyl Orange <br> Best 01(out of Best 01(out of 03) <br> titre value <br> 03 )  |  |  |  |  |
| Error $\left(\mathrm{cm}^{3}\right)$ | Mark s |  |  | Error (\%) | Marks | Error $\left(\mathrm{cm}^{3}\right)$ | Marks | $\begin{aligned} & \text { Error } \\ & \left(\mathrm{cm}^{3}\right) \end{aligned}$ | Marks | $\begin{aligned} & \text { Error } \\ & \left(\mathrm{cm}^{3}\right) \end{aligned}$ | Marks | $\begin{aligned} & \text { Error } \\ & \left(\mathrm{cm}^{3}\right) \end{aligned}$ | $\begin{gathered} \text { Mark } \\ \mathrm{s} \end{gathered}$ |
| $\pm 0.50$ | 26 | $\pm 5.0$ | 26 | $\pm 0.50$ | $13+13$ | $\pm 0.2$ | 13+13 | $\pm 0.2$ | 13 | $\pm 0.2$ | 13 |
| $\pm 0.51-0.60$ | 22 | $\pm 5.1-6.0$ | 22 | $\pm 0.51-0.60$ | 11+11 | $\pm 0.3$ | $11+11$ | $\pm 0.3$ | 11 | $\pm 0.3$ | 11 |
| $\pm 0.61-0.70$ | 18 | $\pm 6.1-7.0$ | 18 | $\pm 0.61-0.70$ | 9+9 | $\pm 0.4$ | 9+9 | $\pm 0.4$ | 9 | $\pm 0.4$ | 9 |
| $\pm 0.71-0.80$ | 14 | $\pm 7.1-8.0$ | 14 | $\pm 0.71-0.80$ | 7+7 | $\pm 0.5$ | 7+7 | $\pm 0.5$ | 7 | $\pm 0.5$ | 7 |
| $\pm 0.81-1.0$ | 10 | $\pm 8.1-10.0$ | 10 | $\pm 0.81-1.0$ | 5+5 | $\pm 0.6$ | 5+5 | $\pm 0.6$ | 5 | $\pm 0.6$ | 5 |
| $> \pm 1.0$ | Zero | $> \pm 10$ | Zero | $> \pm 1.0$ | Zero | $> \pm 0.6$ | Zero | $> \pm 0.6$ | Zero | > $\pm 0.6$ | Zero |
| Graph : 5 marks <br> Calculation : 4 marks |  | pKa : Two graphs 5+4 marks <br> Viscosity: <br> Calculation: 9 marks |  | Graph :5 marks <br> Calculation : 4 marks |  | Calculation: 9 marks <br> Note: Best two (out of three) titre values to be considered for the valuation of volumetric experiments except Alkalinity of water experiment. |  |  |  |  |  |

