## A LAB TEXT BOOK ON



## By

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## PREFACE

Chemistry is a branch of experimental science. So, it is very important that students of chemistry perform lab experiments to fully understand that the theories they study in lecture and in their textbook are developed from the critical evaluation of experimental data. The laboratory can also help the student to develop interest in the study of the science by clearly illustrating the principles and concepts involved. Finally, laboratory experimentation teaches students the opportunity to develop techniques and other manipulative skills that students of engineering must master for application in career and daily life. This Engineering Chemistry lab manual is designed to meet the need of GGSIP University BTech $1^{\text {st }}$ semester students.

The faculty of the Chemistry at AIT clearly understands the importance of laboratory work in the study of chemistry and committed to educate the student in lab skill and hopes that they will take full advantage of this opportunity. This Engineering Chemistry lab manual provides the students a clear, comprehensive and up to date information about the various practical of chemistry as applied to engineering chemistry.

In addition, students are encouraged to complete the report as soon after laboratory as possible, as this is much more efficient than waiting until the night before it is due. In conclusion, we view this manual as one of continual modification and improvement. We encourage you to discuss ideas for improvements or suggestions with author. Finally, we hope you find this laboratory manual helpful in your study of chemistry.

Dr. A.K.Jain<br>Dr. Era Upadhyay

## Paper Code: ETCH-153 P C <br> Paper: Engineering Chemistry Lab 21 <br> List of Experiments

1. Determine the percentage composition of sodium hydroxide in the given mixture of sodium hydroxide and sodium chloride.
2. Determine the amount of Oxalic acid and Sulphuric acid in one litre of solution, given standard sodium hydroxide and Potassium Permanganate.
3. Determine the amount of copper in the copper ore solution, provided hypo solution.
4. Argentometric titration one each by Vohlard's method and by Mohr's method.
5. Complexometric titrations.
6. Determine the heat of neutralization of strong acid with strong base.
7. Determine the surface tension of a liquid using drop weight method.
8. Determine viscosity of a given liquid (density to be determined).
9. Determine the reaction rate constant for the Ist order reaction.

10 . Determine the cell constant of a conductivity cell and titration of strong acid/strong base conductometrically.

## REFERENCE BOOKS:

1. Vogel's Textbook of Quantitative Chemical Analysis, Revised by G.H. Jeffery, J. Bassett,
J. Mendham and R.C. Denney.
2. Applied Chemistry: Theory and Practice by O.P. Vermani and A.K. Narula.
3. Laboratory Manual on Engg. Chemistry by S.K. Bhasin and Sudha Rani.

Ansal Institute of Technology
Prosecedty Pitions
Table of content
Preface ..... 2.
List of experiment ..... 3.
General rules for the safe working of students in the laboratory. ..... 5.
EXP No.1. Determine the percentage composition of sodium hydroxide in the given mixture of sodium hydroxide and sodium chloride. ..... 9.
EXP No.2. Determine the amount of Oxalic acid and Sulphuric acid in one litre of solution, givenstandard sodium hydroxide and Potassium Permanganate.11.
EXP No.3. Determine the amount of copper in the copper ore solution, provided hypo solution. ..... 14.
EXP No.4. Argentometric titration one each by Vohlard's method and by Mohr's method. ..... 17.
EXP No.5. Complexometric titrations. ..... 19.
EXP No.6. Determine the heat of neutralization of strong acid with strong base. ..... 22.
EXP No.7. Determine the surface tension of a liquid using drop weight method. ..... 25.
EXP No.8. Determine viscosity of a given liquid (density to be determined). ..... 30.
EXP No.9. Determine the reaction rate constant for the Ist order reaction. ..... 33.
EXP No.10. To find the cell constant of conductivity cell. ..... 35.
EXP. No.11. To find out the strength of a given strong acid solution by titrating it against strong basesolution Conductometrically38.
EXP.No.12. To Perform a conductometric titration between a weak acid and strong base and to find out thestrength of the acid.
41. Ansal Institute of Technology

General rules for the safe working of students in the laboratory.

## Laboratory Safety

1. State-approved safety goggles must be worn in the laboratory at all times. This is required by state health regulation. Failure to observe this requirement will result in your removal from the laboratory. The cost is approximately Rs100 (and trivial in comparison to your eyesight). Safety glasses, etc. are not acceptable. If you already have goggles, you will not be allowed in the laboratory and, consequently, will miss the experiment.
2. The use of contact lenses in the laboratory is absolutely prohibited. In the case of a splash or other emergency, they may interfere with the flushing of the eye, you may not be in a position to remove them, and those administering first aid may not know that you are wearing them or might not be able to remove them easily.
3. You must dress appropriately for the laboratory. Lab coats are required to be worn. Bare feet, sandals, or other open-toed shoes are not permitted in the laboratory. Shorts and short skirts are likewise not permitted; legs must be covered to below the knees. Headphones are not allowed in the laboratory. Failure to observe these requirements will result in your removal from the laboratory. Cotton clothing (including denim) is particularly susceptible to being eaten by acid solution. The laboratory is not a good place to wear your favorite clothes. Long hair should be tied back.
4. Learn the location and operation of the safety showers, emergency eyewashes and fire extinguishers in the laboratory. In the case of spill onto a person or clothing, the immediate action should be to flush with water and lots of it. Do not hesitate to yell for help. Report accidents to your instructor. $\mathrm{He} /$ she has been certified to administer first aid. If you are not familiar with the operation of the fire extinguishers, ask your instructor to explain them to you.

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5. The fire extinguishers should only be used for real emergencies since the chemicals they contain can cause considerable damage. In any emergency that requires the assistance of the fire department, aid car, or police, sends someone to the stockroom for assistance. Should clothing catch on fire, remain as calm as possible. Walk (do not run) to the safety shower and pull the ring to douse yourself with water. Alternatively, you may drop to the floor and roll to extinguish the flames.
6. Become familiar with all of the exits from the laboratory. A repeating siren and flashing of the FIRE indicator is the building evacuation signal. If this alarm goes off while you are in the lab, turn off any open flames, grab your valuables, and leave the building as quickly as possible.
7. Never attempt any unauthorized or unassigned experiments. Follow the experimental procedures explicitly, checking and double-checking the identity of all reagents before you use them. There are potentially hazardous combinations of chemicals present in the laboratory. If you have an idea for further investigation, discuss it with your instructor.
8. Clean up spills immediately. The next person to come along has no way of knowing if the clear liquid or white powder on the lab bench is innocuous or hazardous. Neutralize acid spills with sodium bicarbonate (baking soda) before cleaning them up. Spills of sulfuric acid solutions are particularly hazardous since only the water will evaporate, thereby making the solution more concentrated upon standing.
9. Never return unused reagents to their storage container. If you take more than you need, dispose of the excess in the appropriate manner. Use the reagents sparingly-they are expensive and time-consuming to prepare. When taking reagents, transfer the amount you need to a clean beaker or other suitable container for taking the material back to your desk. Never insert a pipette or any other object into a liquid reagent container. Finally, check and double-check the identity of all materials before using them.

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10. Do not pick up hot objects. Be sure your apparatus is cool before picking it up. Do not point the open end of a test tube or other vessel containing a reaction mixture toward yourself or anyone else. If the procedure calls for you to observe the odor of the contents of a vessel, hold it upright in front of you, gently fan some of the vapors toward your nose and sniff cautiously. Most chemical vapors are at least irritating, and many are quite toxic. Please do not taste any chemicals.
11. Do not eat, drink or smoke in the laboratory.
12. Playing of radios, tapes, CDs is not permitted. This includes small portable devices used with earphones or headsets.
13. Keep coats, backpacks and other non-essential materials away from areas where people are working.
14. Dispose of all broken glassware and other sharp objects in the cardboard glass disposal boxes. Custodial personnel will stop collecting trash after they find broken glass in the trashcans.
15. Wash hands often when working in lab, and always wash thoroughly before leaving.
16. Use the hood for evaporation of anything other than water. The vapors from your procedure alone may not present a problem but those from all the students in the lab could combine to create a hazard.
17. Do not leave a Bunsen burner or other heated apparatus unattended. The person working next to you may not know what is involved with your setup and may be working with a flammable material. Turn off open flames if you must leave your area. Make sure the gas taps are completely off whenever the Bunsen burner is not lit.
18. Waste Disposal Dispose of chemical reagents and other materials properly. The proper disposal of chemical wastes is essential to the health and safety of Institute faculty, staff, students and the surrounding community.
19. Chemical wastes must be managed and discarded in the most responsible and environmentally sound method available.

## Miscellaneous Policies and Procedures

Homework and Lab Report Due Dates. Lab reports are due one week from the date of performance, at the beginning of the lab period. Only a verifiable illness (Doctor's note) or prior permission of the instructor counts as excused absences. Unexcused lab reports will receive a grade of zero.
Lab Partners. For those experiments where students are to work in pairs, lab partners will be assigned randomly as announced by the instructor at the beginning of the lab period. You may not exchange lab partners. Both lab partners must be present for the entire experiment.

Copying. All lab reports are to be your own. Lab partners are to independently produce their lab reports. It is very easy for the grader to spot identical work among two or more students. In the event of copying, all students involved will receive a grade of zero; therefore do not give a copy of your lab report to another student.
Make-Up Labs. There will be no make-up labs.

## Experiment No. 1...

Date: $\qquad$
Objective: To find out the percentage composition of the given mixture of NaCl and NaOH .

Apparatus Requirements: Burette, pipette, conical flasks, beakers,
Chemical Requirement: 0.1 N HCl Sodium Hydroxide, Sodium chloride Phenolphthalein Indicator.

## Principle:

For the titration of a solution of NaOH and NaCl the other solution need is HCl solution. NaOH react with HCl solution whereas NaCl will remain as such.
$\mathrm{NaOH}+\mathrm{HCl} \longrightarrow \mathrm{NaCl}+\mathrm{H}_{2} \mathrm{O}$
The mixture solution will be titrated with the acid solution of HCl and we can find out the normality and strength of NaOH . By dividing the strength of NaOH from the total known strength of the solution we will find out the strength of NaCl and hence find the \% composition.

## Indicator:

Phenolphthalein

## End point

Pink to colorless.

## Procedure:

1. Rinse the burette with the given $\mathrm{N} / 10 \mathrm{HCl}$ solution.
2. Take the HCl in the burette and note the initial reading.
3. Pipette out the 10 ml of the solution in the conical flask
4. Add a drop of indicator in the solution
5. Add the acid solution from burette in to the conical flask till solution become colourless
6. Note the burette reading
7. Repeat the experiment to get three concordant reading

## Observations:

Normality of the HCl solution $=0.1 \mathrm{~N}$

Volume of the mixture of the in conical flask= 10 ml
Table:

| S.No. | Initial Readings | Final Readings | Volume 0.1 N HCI used (ml) |
| :---: | :---: | :---: | :---: |
| 1 |  |  |  |
| 2 |  |  |  |
| 3 |  |  |  |

## Calculations:

By the law of chemical equivalents $\mathrm{N}_{1} \mathrm{~V}_{1}=\mathrm{N}_{2} \mathrm{~V}_{2}$
Normality of given NaOH solution $\mathrm{N}_{2}=\mathrm{N}_{1} \mathrm{~V}_{1} / \mathrm{V}_{2}=0.1 \mathrm{~V}_{1} / 10=\mathrm{V}_{1} / 100$

As the equivalent weight of $\mathrm{NaOH}=40$
Strength of given NaOH solution $=\mathrm{N}_{2} \mathrm{x} 40 \mathrm{gm} / \mathrm{l}=\mathrm{y} . \ldots . \mathrm{g} / \mathrm{l}$

Because the amount of NaOH in the mixture is $\mathrm{g} / \mathrm{L}$
Amount of NaCl in the mixture $=(8-y) \mathrm{g} / \mathrm{L}=\mathrm{z} . . \mathrm{g} / \mathrm{L}$
The percentage of the $\mathrm{NaOH}=\mathrm{y} / 8 \times 100$,
The percentage of $\mathrm{NaCl}=\mathrm{z} / 8 \times 100$

## Result:

i) Normality of Sodium Hydroxide solution $=$. $\qquad$
ii) Strength of Sodium Hydroxide solution $=$ $\qquad$ .gms/l.
iii) The percentage of the $\mathrm{NaOH}=\mathrm{y} / 8 \times 100$,
iv) The percentage of $\mathrm{NaCl}=\quad \mathrm{z} / 8 \times 100$

## Questions:

1. What do you mean by back- titration?
2. What is the colour of phenolphthalein in acidic and base medium?
3. What is the structure of phenolphthalein?
4. What is the procedure to prepare a 0.1 N solution of NaOH ?

Experiment No......2.....
Date: $\qquad$
Objective: To determine the amount of oxalic acid and sulphuric acid in one litre of solution with given standard NaOH solution and standard KMnO 4 solution.
Apparatus Required: Burette, pipette, conical flasks, beakers, hot plate,
Chemical Required: Solution of oxalic acid and sulphuric acid, Sodium Hydroxide (0.1 N ), $\mathrm{KMnO}_{4}$ solution ( 0.1 N ), Phenolphthalein Indicator.
Principle: This experiment involves double titration.
I $^{\text {st }}$ Titration: NaOH reacts with oxalic acid as well as $\mathrm{H}_{2} \mathrm{SO}_{4}$ according to the following equation-
$2 \mathrm{NaOH}+(\mathrm{COOH})_{2} \cdot 2 \mathrm{H}_{2} \mathrm{O} \longrightarrow(\mathrm{COONa})_{2}+4 \mathrm{H}_{2} \mathrm{O}$
Sodium hydroxide Oxalic Acid Sodium oxalate Water

$$
\mathrm{H}_{2} \mathrm{SO}_{4}+2 \mathrm{NaOH} \longrightarrow \mathrm{Na}_{2} \mathrm{SO}_{4}+2 \mathrm{H}_{2} \mathrm{O}
$$

By titrating NaOH against the given mixture of oxalic acid and sulphuric acid, the total normality of oxalic acid and sulphuric acid can be determined.

## II ${ }^{\text {nd }}$ Titration:

The mixture of solution is titrated with $\mathrm{N} / 10 \mathrm{KMnO}_{4}$ which will react with oxalic acid in the presence of $\mathrm{H}_{2} \mathrm{SO}_{4}$.


So the normality of oxalic acid can be find out by $\mathrm{II}^{\text {nd }}$ titration and hence its strength is determined.

From the normality of the solution obtained in the $I^{\text {st }}$ titration, the normality of oxalic acid is subtracted and hence the normality of $\mathrm{H}_{2} \mathrm{SO}_{4}$ and its strength can be found out.

## Procedure:

## $I^{\text {st }}$ Titration:

i) Take 0.1 N NaOH solution in the washed and rinsed burette and pipette out 10 ml of mixture in the titration flask.
ii) Add a drop of phenolphthalein and titrate it till the end point.

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iii) Note down the volume of NaOH used.
iv) Repeat the process to get 3 concordant readings.

## II ${ }^{\text {nd }}$ Titration:

i) Wash and rinse the apparatus thoroughly. Take $0.1 \mathrm{~N} \mathrm{KMnO}_{4}$ solution in burette. Since it is a coloured solution we note the upper meniscus for taking the initial and subsequent readings.
ii) Pipette out 10 ml . of the mixture in the titration flask. Add almost 10 ml of sulphuric acid and then heat till titration flask is unbearable to $60-70^{\circ} \mathrm{C}$.
iii) Titrate till end point and note the volume of $\mathrm{KMnO}_{4}$ used.
iv) Repeat the process to get 3 concordant readings.

## Observations:

## Table-I

NaOH vs. Mixture

| S.No. | Volume of sample <br> solution taken (ml) | Burette Reading |  | Volume of standard NaOH <br> solution used (ml) |
| :--- | :--- | :--- | :--- | :--- |
|  |  | Initial | Final |  |
| 1. |  |  |  |  |
| 2. |  |  |  |  |
| 3. |  |  |  |  |

## Table-II

$\mathrm{KMnO}_{4}$ vs. Mixture (Only oxalic acid reacts)

| S.No. | Volume of <br> sample solution <br> taken (ml) | Burette Reading |  | Volume of $\mathrm{KMnO}_{4}$ <br> solution used (ml) |
| :--- | :--- | :--- | :--- | :--- |
|  | Initial | Final |  |  |
| 1. |  |  |  |  |
| 2. |  |  |  |  |
| 3. |  |  |  |  |

## Calculations:

From Titration - I:

$$
\begin{gathered}
\mathrm{N}_{1} \mathrm{~V}_{1}\left(\mathrm{H}_{2} \mathrm{SO}_{4}+\text { Oxalic Acid }\right)=\mathrm{N}_{2} \mathrm{~V}_{2}(\mathrm{NaOH}) \\
\mathrm{N}_{1}=0.1 \times \mathrm{V}_{2} / 20
\end{gathered}
$$

From Titration - II:

$$
\begin{aligned}
\mathrm{N}_{3} \mathrm{~V}_{3}(\text { Oxalic Acid }) & =\mathrm{N}_{4} \mathrm{~V}_{4}\left(\mathrm{KMnO}_{4}\right) \\
\mathrm{N}_{3} & =0.1 \times \mathrm{V}_{2} / 20
\end{aligned}
$$

Where,
Normality of $\left(\mathrm{H}_{2} \mathrm{SO}_{4}+\right.$ Oxalic Acid $)=\mathrm{N}_{1}$
Normality of Oxalic Acid) $=\mathrm{N}_{3}$
Normality of $\mathrm{H}_{2} \mathrm{SO}_{4}$
Strength of oxalic acid
$=\mathrm{N}_{1}-\mathrm{N}_{3}$

Strength of $\mathrm{H}_{2} \mathrm{SO}_{4}$
$=\mathrm{N}_{3} \times$ Eq. Wt. of oxalic acid
$=\left(\mathrm{N}_{1}-\mathrm{N}_{3}\right) \mathrm{x}$ Eq. Wt. of Sulphuric acid

## Result:

i) The strength of oxalic acid = $\qquad$ .gms/l.
ii) The strength of sulphuric acid
$=$ $\qquad$ .gms/l.

## Questions:

1. What do you mean by redox- titration?
2. What type of titrations are permagnate titrations?
3. Why can we not heat the solution upto $100^{\circ} \mathrm{C}$ ?
4. Why is $\mathrm{KMnO}_{4}$ solution not filtered through filter paper?
5. Why we do not use HCl or dil. $\mathrm{HNO}_{3}$ in $\mathrm{KMnO}_{4}$ titration?

Experiment No.: ...3.........

## Date:

Objective: To estimate the amount of Copper present in the given solution using a standard solution by provided Hypo solution.
Apparatus Required: 100 ml standard flask, Funnel, Burette, Iodometric flask, Conical Flask, Pipette, Simple balance with weights,
Chemicals Required: $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$, Hypo $\left(\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}\right)$, KI, Conc. HCl , dil. Acetic acid, $\mathrm{NaHCO}_{3}, \mathrm{H}_{2} \mathrm{SO}_{4}, \mathrm{CuSO}_{4}$, Ammonia solution, Starch \& distilled water.
Principle: Any cupric salt in neutral medium when treated with Potassium Iodide forms a white precipitate of cuprous iodide and iodine is set free quantitatively. The liberated Iodine is treated against Hypo using starch as the indicator.

## Chemical reactions:

$$
\begin{aligned}
& \mathrm{CuSO}_{4}+2 \mathrm{KI} \rightarrow \mathrm{CuI}_{2}+\mathrm{K}_{2} \mathrm{SO}_{4} \\
& 2 \mathrm{CuI}_{2} \rightarrow \mathrm{Cu}_{2} \mathrm{I}_{2}+\mathrm{I}_{2} \\
& \mathrm{I}_{2}+2 \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}+\mathrm{O}_{2} \rightarrow \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{6}+2 \mathrm{NaI} \\
& 2 \mathrm{CuSO}_{4}=\mathrm{I}_{2}=\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}
\end{aligned}
$$

## Procedure:

1. Preparation of standard potassium dichromate: Weigh out accurately the given pure crystalline sample of potassium dichromate and transfer into 100 ml standard (volumetric) flask provided with a funnel. Dissolve the dichromate in a small quantity of distilled water, and make upto the mark. The contents in the flask are shaken well for uniform concentration. Calculate the normality of potassium dichromate.
2. Standardization of sodium thiosulphate: Rinse the burette and fill it up with hypo solution without any air bubbles. Note the burette reading. Take about 20 ml of $10 \% \mathrm{KI}$ solution in a clean conical flask and add 2 grams of sodium bicarbonate followed by 5 ml of concentrate HCl gently rotate the flask for mixing the liquids. Rinse the pipette with a little of potassium dichromate solution and then transfer 20 ml of the same to the conical flask. Shake it well, stopper it, and keep it in dark place for 5 minutes Titrate the liberate iodine by running down hypo from the burette with constant stirring. When the solution attains a pale yellow colour add 2 ml of freshly prepared starch solution.

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The colour changes to blue. Continue the titration drop-wise till the colour changes from blue to light green indicating the end point. Repeat the titration for concurrent values.
3. Estimation of copper: Make up the given solution of copper to 100 ml with distilled water and shake well for uniform concentration. Pipette out 20 ml of this solution into a clean conical flask. Add few drops of ammonia when bluish white precipitate is obtained. Redissolve the precipitate in dilute acetic acid. Now add 10 ml of $5 \%$ KI, when iodine is liberated giving a brown colour. Titrate this solution against standard hypo solution till light yellow colour is obtained. Now add 2 ml of starch solution and continue the titration till blue colour changes to creamy white, which is the end point. Repeat the titration for concurrent values and calculate the amount of copper.

Preparation of Standard solution:
$\mathrm{W} 1=$ Weight of bottle + substance $=$ $\qquad$ gms
$\mathrm{W} 2=$ Weight of bottle = $\qquad$ gms
Weight of substance $=\left(\mathrm{W}_{1}-\mathrm{W}_{2}\right)=$ $\qquad$ gms.

$$
\text { Normality of the solution }=\frac{\left(W_{1}-W_{2}\right) \times 10}{\text { Equivalent Weight }}=\frac{\left(W_{1}-W_{2}\right) \times 10}{49}=
$$

| S.No | Volume of standard <br> $\left(\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}\right)$ solution $\left(\mathrm{V}_{1}\right)$ | Burette Reading |  | Volume of |
| :--- | :--- | :--- | :--- | :--- |
|  |  |  |  |  |

$\mathrm{N}_{1}=$ Normality of Potassium dichromate $=$
$\mathrm{V}_{1}=$ volume of Potassium dichromate $=20 \mathrm{ml}$
$\mathrm{N}_{2}=$ Normality of Hypo $=$ ?
$\mathrm{V}_{2}=$ Volume of Hypo $=$

$$
\begin{aligned}
& \mathrm{N}_{1} \mathrm{~V}_{1}=\mathrm{N}_{2} \mathrm{~V}_{2} \\
& \mathrm{~N}_{2}=\frac{\mathrm{N}_{1} \mathrm{~V}_{1}}{\mathrm{~V}_{2}}=
\end{aligned}
$$

$\mathrm{N}_{2}=$ Normality of Hypo $=$

| S.No | Volume of Copper <br> solution (V3) | Burette Reading |  | Volume of <br> consume (V4) |  |
| :--- | :--- | :--- | :--- | :--- | :---: |
|  |  | Initial | Final |  |  |
| 1 | 20 ml |  |  |  |  |
| 2 | 20 ml |  |  |  |  |
| 3 | 20 ml |  |  |  |  |
|  | $\mathrm{V}_{3}=20 \mathrm{ml}$ |  |  |  |  |

N3 $=$ Normality of Copper solution $=$ ?
V3 $=$ volume of Copper Solution $=20 \mathrm{ml}$
N4 = Normality of Hypo =
V4 = Volume of Hypo =

$$
\mathrm{N}_{3} \mathrm{~V}_{3}=\mathrm{N}_{4} \mathrm{~V}_{4}
$$

Normality of Copper solution $=\mathrm{N}_{3}=\underline{N}_{4} \mathrm{~V}_{4}$ $\mathrm{V}_{3}$
Amount of Copper present in the whole of the given solution $(100 \mathrm{ml})=\mathrm{N}_{3} \mathrm{X} 63.54$
Result: Amount of Copper present in the whole of the given solution $(100 \mathrm{ml})=$ $\qquad$ gm.

## Experiment No... 4

Date:
Objective: To determine volumetrically the strength of a given ammonium thiocyanate solution by Volhard's method, provided N/30 silver nitrate solution.
Apparatus Required: Burette, pipette, 2 beakers, titration flask, stand,
Chemical Required: Ammonium thiocyanate solution (unknown), Silver Nitrate (N/30),

## Ferric Alum Indicator

## Principle:

Ammonium thiocyanate and Potassium thiocynate solutions have to be standardised by titrating against silver nitrate solution because these salts are hygroscopic and thus their standard solutions cannot be prepared directly by, weighing.
In Volhard's method, the indicator employed is a solution of Ferric alum, Ammonium Ferric Sulphate $\mathrm{NH}_{4} \mathrm{Fe}\left(\mathrm{SO}_{4}\right)_{2} .12 \mathrm{H}_{2} \mathrm{O}$. When thiocyanate solution is added to a silver nitrate solution in the presence of nitric acid containing some ferric alum, a white precipitate of silver thiocyanate is produced initially. But when all $\mathrm{Ag}^{+}$ions have been precipitated out, even a slight excess of thiocyanate added further, gives a reddish-brown colouration due to the formation of ferric thiocyanate.

$$
\begin{array}{ll}
\mathrm{Ag}^{+}+\mathrm{SCN}^{-} \_ & \mathrm{AgSCN}(\text { White ppt. }) \\
\mathrm{Fe}^{3+}+3 \mathrm{SCN}^{-} \_ & \mathrm{Fe}(\mathrm{SCN})_{3} \\
& (\text { Ferric Thiocyanate }) \\
& (\text { Reddish Brown })
\end{array}
$$

## Procedure:

1. Rinse and fill the clean burette with the given thiocyanate solution.
2. Wash with water and then rinse the pipette with $\mathrm{AgNO}_{3}$ solution.
3. Pipette out 10 ml . of $\mathrm{N} / 30 \mathrm{AgNO}_{3}$ solution.
4. To it add 1 ml . of ferric alum indictor solution.
5. Titrate it against ammonium thiocyanate solution while shaking the flask vigorously. A white precipitate of AgSCN will be formed.
6. Appearance of brownish red colour indicates the end point. Take 3 concordant readings.

## Observations:

| S.No. | Volume of $\mathrm{AgNO}_{3}$ solution taken (ml) | Burette Reading |  | Volume of ammonium thiocyanate solution used (ml) |
| :---: | :---: | :---: | :---: | :---: |
|  |  | Initial | Final |  |
| 1. |  |  |  |  |
| 2. |  |  |  |  |
| 3. |  |  |  |  |

## Calculations:

$\mathrm{N}_{1} \mathrm{~V}_{1}$ of ammonium thiocyanate $=\mathrm{N}_{2} \mathrm{~V}_{2}$ of $\mathrm{AgNO}_{3}$
$\mathrm{N}_{1} V_{1}=1 / 30 \mathrm{X} 10$
$\mathrm{N}_{1}=1 / 3 V_{1}$
Strength of a given ammonium thiocyanate solution $=\mathrm{N}_{\mathrm{I}} \times$ Eq. Wt. of ammonium thiocyanate

## Result:

The strength of given ammonium thiocyanate solution was found to be

## Questions:

1. Enumerate some of the requirements for precipitation titrations.
2. Can we use volhard's method for the estimation of chloride ions?
3. What are the limitations of volhard's method?
4. Can peptizing agents interfere in the determination of end point?

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## Experiment No.: ...5..

## Date:

Objective: To estimate the strength of Calcium ions/Magnesium Ion of the given solution using a standard solution of EDTA by complexometric titration.
Apparatus Required: 100 ml standard flask, Burette, 250 ml Conical Flask, 20 ml Pipette \& Simple balance with weights,
Chemical Required: Ethylene di-amine tetra acetic acid (EDTA), Eriochrome Black-T (EBT) Indicator, Ammonia Buffer solution, Hard-water \& distilled water.

Principle: Hard water, which contains calcium and magnesium ions, forms a wine red colored complex with the indicator, Eriochrome Black-T. Ethylene diamine tetra acetic acid (EDTA) forms a colourless stable complex with free metal ion like $\mathrm{Ca}, \mathrm{Mg}$. i.e., Metal + Indicator $\longrightarrow$ Metal indicator complex (wine red colour)

When EDTA is added from the burette, it extracts the metal ions from the metal ionindicator complex thereby releasing the free indicator. (The stability of metal ionindicator complex is less than that of the metal ion- EDTA complex, and hence EDTA extracts metal ion form the ion indicator complex.)


$$
\begin{aligned}
& \text { Structure of EDTA } \\
& \text { EDTA }+ \text { Metal indicator complex } \rightarrow \text { Metal ion-EDTA + Indicator } \\
& \text { (wine red colour) } \\
& \text { (Blue) }
\end{aligned}
$$

The various EDTA species are abbreviated as $\mathrm{H}_{3} \mathrm{Y}, \mathrm{H}_{3} \mathrm{Y}^{-}, \mathrm{H}_{2} \mathrm{Y}^{-2}, \mathrm{HY}^{-3}$, and $\mathrm{Y}^{-4}$. The relative amounts of these species is a function of pH
At $\mathrm{pH}>12$ EDTA exist As $\mathrm{Y}^{-4}$
$\mathrm{pH} \leq 8$ EDTA exist As $\mathrm{HY}^{-3}$
$\mathrm{pH} \approx 5$ EDTA exist As $\mathrm{H}_{2} \mathrm{Y}^{-2}$
The reactions take place at a $\mathrm{pH}=10$ and the buffer is made by ammonium chloride and ammonium solution.

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Structure of EDTA -Metal complex

( 1-(hydroxy 2-naphthhylazo) 6 nitro-2-napthol-4-sulphonate)
Structure of Eriochrome Black-T
At $\mathrm{pH}<6$ the indicator exist as $\mathrm{H}_{2} \ln ^{-}(\mathrm{Red})$
$\mathrm{pH}<7$ the indicator exist as $\mathrm{Hln}^{-2}$ (blue)
$\mathrm{pH}<10$ the indicator exist as $\ln ^{-3}$ (orange)

## Procedure:

1. In a 250 ml conical flask pipette add 5 ml of prepared calcium solution.
2. Add 10 ml of buffer solution and 2-3 drop of EBT
3. Add EDTA from burette in to the flask till red colour of the solution change to blue colour.
4. Repeat the titration till to get three concordant readings

## Observation:

| S.No | Volume of Solution taken <br> in Flask (ml) | Burette reading |  | Volume of <br> EDTA <br> (ml) |  |
| :--- | :--- | :--- | :--- | :--- | :---: |
|  |  | Initial | Final |  |  |
| 1. |  |  |  |  |  |
| 2. |  |  |  |  |  |
| 3. |  |  |  |  |  |

## Calculation

EDTA forms 1:1 complex with Metal ions
As 1 molar solution of EDTA react with $=1$ mole of $\mathrm{Ca}^{+2}$ ions

$$
=40.08 \text { gram of } \mathrm{Ca}^{+2} \text { Ions }
$$

1 ml of 0.1 M EDTA $=4.008 \mathrm{mg}$ of $\mathrm{Ca}^{+2}$ Ions
Hence V ml of 0.1 M EDTA $=\mathrm{V} x 4.008 \mathrm{mg}$ of $\mathrm{Ca}^{+2}$ Ions
Or 5 ml of $\mathrm{CaCO}_{3}$ Solution has $=\mathrm{V} \times 4.008 / 5 \times 1000 \mathrm{mg} / \mathrm{L}$ of Calcium ions
$\mathrm{V} \times 4.008 / 5 \times 1000 \mathrm{ppm}$ of calcium ions

## In the case of $\mathbf{~ M g}$ Estimation

The procedure is same

## Calculation

1 ml of $0.01 \mathrm{MEDTA}=0.243 \mathrm{mg}$ of Mg
As V ml of 0.01 M EDTA= $\mathrm{V} \times 0.243 \mathrm{mg}$ of Mg
So 5.0 ml of $\mathrm{Mg} \mathrm{SO} \mathrm{S}_{4}$ solution $=\mathrm{V} \times 0.243 \mathrm{mg}$ of Mg
Hence 1000 ml Mg SO 4 SOLUTION $=\mathrm{V} \times 0.243 / 5 \times 1000 \mathrm{mg} / \mathrm{L}$ of Mg
$=\mathrm{V}$ x 0243/5 x 1000ppm of Magnesium

## Result:

i) The calcium hardness of the given water sample $=$ $\qquad$ ppm.
ii) The Mg hardness of the given water sample $=$ $\qquad$ ppm.

Experiment No...6.
Date:
Objective: Determination of heat of neutralization of hydrochloric acid with sodium hydroxide.
Requirements: Thermos flask, glass stirrer, thermometer, stop watch, beaker, measuring cylinder $\left(50 \mathrm{~cm}^{3}\right)$, Sodium hydroxide $=(0.5 \mathrm{M})$, Hydrochloric acid $=(0.5 \mathrm{M})$

Principle: The heat of neutralization of an acid at a given temperature is defined as the amount of heat evolved when one-gram equivalent of an acid is neutralized by one-gram equivalent of a base in dilute solution at that temperature.

It is found that heat of neutralization for all the strong acids and strong base is practically a constant quantity. This can be explained on the basis of theory of ionization. Strong acids and strong bases are assumed to be completely ionized in dilute solutions. Moreover the salts they form on mixing are also completely ionized in dilute aqueous solution. The reaction between a strong acid like HCl and a strong base like NaOH can be written as:

$$
\begin{array}{ll}
\mathrm{H}_{(\text {aq) }}^{+}+\mathrm{Cl}^{-(\mathrm{aq})} \\
\text { or } \\
\mathrm{H}^{+}{ }_{(\mathrm{aq})}+\mathrm{Na}^{+}{ }_{(\text {aq) }}+\mathrm{OH}_{(\text {aq) }}^{-} & \\
& \Delta \mathrm{H}=-57.3 \mathrm{~kJ}
\end{array}
$$

Thus Neutralization is simply a reaction between the $\mathrm{H}^{+}$ions given by the acid and OH ions given by the base. In case of strong acids and strong bases the number of $\mathrm{H}^{+}$ions and $\mathrm{OH}^{-}$ions produced by one gram of any strong acid or strong base is always the same. Hence the enthalpy of neutralization of a strong acid with a strong base is always the same.

A known volume of HCl solution of known concentration is allowed to react completely with a strong alkali in dilute solution. The rise in temperature is noted. Knowing the heat capacity of the calorimeter, masses of acid and base and; their specific heats in dilute solutions, the heat of neutralization can be calculated.
$\Delta \mathrm{H}=-[$ Heat gained by the calorimeter + Heat gained by the solution $]$

## Procedure:

1. Take a thermos flask and determine its heat capacity.
2. Now take $50 \mathrm{~cm}^{3}$ of HCl in this thermos flask and note down the temperature reading after every half-minute for five minutes.
3. Similarly take $50 \mathrm{~cm}^{3}$ of NaOH in the thermos flask after removing the acid in a beaker. Note down the temperature of NaOH every half-minute for five minutes.
4. Pour the HCl solution in the NaOH already placed in the thermos flask and note down the time of mixing accurately.
5. Note the temperature of the mixture after every half a minute for five minutes.
6. Record the observations as given below.

## Observation:

Molarity of the acid $=0.5 \mathrm{M}$
Molarity of the base $=0.5 \mathrm{M}$
Volume of the acid taken $=50 \mathrm{~cm}^{3}$
Volume of the base taken $=50 \mathrm{~cm}^{3}$

| Time (Sec.) | Temperature ${ }^{\circ} \mathrm{C}$ |  | Time (Sec.) | Temperature ${ }^{\circ} \mathrm{C}$ |
| :--- | :--- | :--- | :--- | :--- |
|  | Acid | Base |  |  |
|  |  |  |  |  |
|  |  |  |  |  |
|  |  |  |  |  |

## Calculations:

Plot a graph between temperature and time (as shown in the Fig.1) From the Fig., calculate, $\mathrm{T}_{\mathrm{a}^{\prime}} \mathrm{T}_{\mathrm{b}}, \mathrm{T}_{\mathrm{m}^{\prime}}$ at the time of mixing.

Where,
$\mathrm{T}_{\mathrm{a}}=$ Temperature of the acid at the time of mixing
$\mathrm{T}_{\mathrm{b}}=$ Temperature of the base at the time of mixing
$\mathrm{T}_{\mathrm{m}}=$ Temperature of the mixture at the time of mixing
This can be done by drawing a vertical line at the time of mixing. Extend the curves to this vertical line. The points of intersection will give the respective temperatures.

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The heat evolved during neutralization will raise the temperature of the solution and that of thermos flask, i.e.
$\Delta \mathrm{H}=-[$ Heat gained by the calorimeter + Heat gained by the solution $]$
$=-\left[\mathrm{C}_{\mathrm{p}(\text { Calorimeter) })}\left(\mathrm{T}_{\mathrm{m}}-\mathrm{T}_{\mathrm{b}}\right)+\left[\mathrm{C}_{\mathrm{p}(\text { Base })}\left(\mathrm{T}_{\mathrm{m}}-\mathrm{T}_{\mathrm{b}}\right)\right]+\left[\mathrm{C}_{\mathrm{p} \text { (acid) }}\left(\mathrm{T}_{\mathrm{m}}-\mathrm{T}_{\mathrm{a}}\right)\right]\right.$
$=-\left[\mathrm{C}_{\mathrm{p} \text { (Calorimeter) }}\left(\mathrm{T}_{\mathrm{m}}-\mathrm{T}_{\mathrm{b}}\right)\right]+\left[\mathrm{V}_{\text {base }} \mathrm{x}\right.$ specific heat of the base $\left.\mathrm{x}\left(\mathrm{T}_{\mathrm{m}}-\mathrm{T}_{\mathrm{b}}\right)\right]+\left[\mathrm{V}_{\text {acid }} \mathrm{x}\right.$ specific heat of the acid $x\left(T_{m}-T_{a}\right)$ ]
In the case of dilute solutions, the specific heat of the acid and base can be taken as that of water $=4.185 \mathrm{JK}^{-1}$. Hence,

Specific heat of water $=$ specific heat of acid $=$ specific heat of base $=\mathrm{s}$
Thus, $\Delta H=-\left[C_{p(\text { calorimeter) }}\left(T_{m}-T_{b}\right)+V_{\text {base }}\right.$. s. $\left(T_{m}-T_{b}\right)+V_{\text {acid }} . s .\left(T_{m}-T_{a}\right)$
Substituting the values of $\mathrm{C}_{\mathrm{p} \text { calorimeter' }}$ specific heat of water, $\mathrm{V}_{\text {base }}, \mathrm{V}_{\text {acid }} \mathrm{T}_{\mathrm{m}}$
$\mathrm{T}_{\mathrm{a}}$ and $\mathrm{T}_{\mathrm{b}}$ calculate $\Delta \mathrm{H} .\left(5=4.185 \mathrm{~J} / \mathrm{g} /{ }^{\circ} \mathrm{C}\right)$
This is the enthalpy when $50 \mathrm{~cm}^{3}$ of 0.5 M HCl is neutralized by $50 \mathrm{~cm}^{3}$ of 0.5 NaOH .
Number of moles in $50 \mathrm{~cm}^{3}$ of $0.5 \mathrm{M} \mathrm{HCl}=0.5 / 1000 \times 50=0.025$ moles
$\therefore \Delta \mathrm{H}$ for the neutralization of 1 mole of $\mathrm{HCl}=-\Delta \mathrm{H} / 0.025=\ldots$ Jmole $^{-1}$


## Result:

The heat of neutralization of hydrochloric acid with sodium hydroxide is ... $\mathrm{Jmol}^{-1}$.

## Questions:

1. What is the difference between heat capacity and specific heat?
2. What do you mean heat capacity of a substance?

Experiment No. .7...

Date.
Objective To determine the surface tension of given liquids with the stalagmometer
Apparatus required: Stalagmometer, Beaker, weighing bottle with a lid,
Chemical required: Distilled water, liquid whose surface tension to be measured

## Principle:

## Surface tension of liquids

The molecules of liquids attract each other by cohesive forces resulting into small distances between the molecules (on the order of 0.1 nm ). Thus the compressibility of liquids is lower then that of gas, while the density is much higher. On the other hand, these cohesive forces are not strong enough to result into the fixed position of molecules that can be seen in solid matter. Liquids do not keep a fixed shape, but adapt the shape of a container. Attractive cohesive forces are short-range forces, which are based on the electronic interactions. They affect molecules in their close vicinity only (zone of molecular interaction). In the bulk of the liquid, each molecule is attracted equally in all directions by the neighboring molecules, ence zero net force (Fig. 1). However, the molecules at the surface do not have other like olecules on all sides around them and they are pulled inwards the liquid core by non-zero net force Fig. 1). Consequently, they cohere more strongly to those associated with them directly on the surface and form a surface "film". Nevertheless, these surface molecules are in the energetically unfavorable state, which forces liquid to minimize the surface area. The geometrical requirement of smallest surface area at the fixed volume is satisfied by the sphere. It is the reason why the free drops of water form spherical droplets.


Figure 1: Cohesive forces in a case of the molecule at the liquid surface and in the bulk.

Surface area increase, $\Delta S$, is balanced out by the increase of energy, $\Delta E$, where
$\Delta E=\sigma \cdot \Delta S$
The coefficient $\sigma$ defines the surface tension in the units of $\mathrm{J} / \mathrm{m}_{2}=\mathrm{N} / \mathrm{m}$. Each liquid is characterized by its own surface tension, which decreases with an increasing temperature (See. Table 1).
The energetic definition of the surface tension (equation 1) is not the only way todescribe it. We can derive it also from the simple experiment shown in Figure 2. It involves the wire rim with the AB side being able to move, and a soap film spanning the space inside the rim. We can observe that the movable wire AB is being pulled towards the soap film, as its area is shrinking down. There is a force in a plane of soap film acting in a direction perpendicular to the wire. It is called the surface force, and is expressed as
$\mathrm{F}=\sigma \cdot 1$
where 1 is the length of $A B$ wire and $\sigma$ is the surface tension. According to this, surface tension is defined as a ratio of surface force to a length of rim that is pulled by this force
$\sigma=\Phi / \mathrm{I}$
Equation (4) defines the surface tension again in units of $\mathrm{N} / \mathrm{m}$.


Figure 2: Soap bubble spanning the space inside the wire rim with a movable side AB .
The determination of the surface tension by the drop counting method
There are two line marks on the stalagmometer: top line above the wide part and bottom line bellow it. The volume between these two lines is $\mathbf{V}$, and liquid with density $r$ contained in this volume has a mass $\mathbf{m}$

$$
\begin{equation*}
\mathrm{m}=\mathrm{V} \cdot \rho \tag{8}
\end{equation*}
$$

Such a volume $\mathbf{V}$ corresponds to $\mathbf{n}$ drops, which are released from the stalgmometer upon
the decrease of liquid level from top to bottom line mark. Here, the average mass of one drop
is $\frac{\mathrm{m}}{\mathrm{n}}=\frac{\mathrm{V} \cdot \rho}{\mathrm{n}}$
Substituting the mass $m$ in the equation (7) with the mass of one drop then yields
$\sigma=\sigma_{\mathrm{H} 2 \mathrm{O}} \cdot \frac{\rho}{\rho_{\mathrm{H} 2 \mathrm{O}}} \cdot \frac{\mathrm{n}_{\mathrm{H} 2 \mathrm{O}}}{\mathrm{n}}$

## Experimental procedure:

1. Fill the stalagmometer up to the top mark with distilled water. Release water to the weighing bottle and count how many drops it takes to decrease the water level in stalagmometer down to bottom mark. Write down the number of drops $\mathrm{nH}_{2} 0$.
2. Empty and dry the weighing bottle and stalagmometer, and prepare them for the next measurement.
3. Repeat steps 1 and 2 for liquids with the unknown surface tension.
4. Write down the densities of studied liquids according to the notes on bottles, and density of distilled water at the actual temperature in laboratory from the table 1.Using equation (10), calculate the surface tension for all the studied liquids.

Observations:
Tab: The determination of the surface tension by the drop counting method

|  | number of drops | $\rho\left[\mathrm{kg} / \mathrm{m}^{3}\right]$ | $\sigma[\mathrm{N} / \mathrm{m}]$ |
| :---: | :---: | :---: | :---: |
| $\mathrm{H}_{2} \mathrm{O}$ |  |  |  |
| liquid 1 |  |  |  |
| liquid 2 |  |  |  |
| liquid 3 |  |  |  |

Tab. 1: The temperature dependence of the surface tension and distilled water density.

| $\mathrm{t}\left[{ }^{\circ} \mathrm{C}\right]$ | $\sigma[\mathrm{N} / \mathrm{m}]$ | $\rho_{\mathrm{H} 2 \mathrm{O}}\left[\mathrm{kg} / \mathrm{m}^{3}\right]$ |
| :---: | :---: | :---: |
| 15 | 0.07349 | 999.96 |
| 16 | 0.07334 | 999.94 |
| 17 | 0.07319 | 999.90 |
| 18 | 0.07305 | 999.85 |
| 19 | 0.07290 | 999.78 |
| 20 | 0.07275 | 998.20 |
| 21 | 0.07259 | 997.99 |
| 22 | 0.07244 | 997.77 |
| 23 | 0.07228 | 997.54 |
| 24 | 0.07213 | 997.30 |
| 25 | 0.07197 | 997.05 |

Weight of empty specific gravity bottle $=\mathrm{w}_{1} \mathrm{~g}$
Weight of empty specific gravity bottle with water $=w_{2} g$
Weight of empty specific gravity bottle with liquid $=w_{3} g$
$\therefore \quad$ Weight of water $=\left(\mathrm{w}_{2}-\mathrm{w}_{1}\right) \mathrm{g}$
Weight of liquid $=\left(\mathrm{w}_{3}-\mathrm{w}_{1}\right) \mathrm{g}$
Relative density of the liquid $=\mathrm{d}_{1} / \mathrm{d}_{\mathrm{w}}=\mathrm{w}_{3}-\mathrm{w}_{1} / \mathrm{w}_{2}-\mathrm{w}_{1}$
$\gamma_{1} / \gamma_{\mathrm{w}}=\mathrm{d}_{\mathrm{l}} / \mathrm{d}_{\mathrm{w}} \mathrm{X}_{\mathrm{w}} / \mathrm{n}_{\mathrm{l}}$
where,
$\gamma_{1} d_{1} n_{1}=$ surface tension, density and number of drops of liquids.

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$\gamma_{w} d_{w} n_{w}=$ surface tension, density and number of drops of water.
By putting the respective values, the surface tension of the liquid relative to water is found out.

Absolute surface tension of the liquid.
$\gamma_{1}=n_{w} / n_{1} \times d_{l} / d_{w} x \gamma_{\mathrm{w}}=\ldots \ldots \ldots \ldots . . . \mathrm{Nm}^{-1}$.

Result: The surface tension of the given liquid is $\qquad$ dune/cm

## Questions:

1. What are the effects of Surface tension?
2. Give some application of Surface Tension.
3. What is the procedure to measure surface tension by stalagmometer?
4. Why we use narrow capillary in the stalagmometer?
5. Define surface energy.
6. Explain 'the drops of liquid or bubbles of a gas are spherical in shape'.

Experiment No... 8 $\qquad$ Date:
Objective: To find out the viscosity of a given liquid using Ostwalds viscometer.
Requirements: Ostwald's viscometer, stop watch, specific gravity bottle, 10 cm 3 pipette, distilled water, rubber tubing.
Principle: The Ostwald's Viscometer method is based on Poiseuille's equation. This relates the rate of flow of a liquid through a capillary tube with the coefficient of viscosity and is expressed by the following equation-

$$
\eta=\pi \mathrm{r} 4 \mathrm{tP} / 8 \mathrm{Vl}
$$ 1

where,
$\mathrm{V}=$ Volume of the liquid of viscosity $\eta$ flowing in time $t$, through a capillary tube of radius $r$ and length 1
$\mathrm{P}=$ Hydrostatic pressure of the liquid.
Thus the determination of the absolute viscosity by means by Poiseuille's expression involves the determination of $\mathrm{v}, \mathrm{r}, \mathrm{t}, \mathrm{l}$ and P .

The method is however tedious and laborious one. Hence, a simpler method is used wherein we compare the viscosities of the two liquids. If the coefficient of viscosity of one liquid is known, then that of the other can be calculated.
If t 1 and t 2 are the flow times required to flow for equal volumes of two liquids through the same length of a capillary tube then from equation (1) we have
$\eta_{1}=\pi \mathrm{r}_{\mathrm{t}_{1}} \mathrm{P}_{1} / 8 \mathrm{Vl}$
$\eta_{2}=\pi \mathrm{r} 4 \mathrm{t} 2 \mathrm{P}_{2} / 8 \mathrm{Vl}$
or

$$
\begin{aligned}
& \eta_{1} / \eta_{2}=\mathrm{t}_{1} \mathrm{P}_{1} / \mathrm{t} 2 \mathrm{P}_{2} \\
& \text {. } 2
\end{aligned}
$$

Where,
$\mathrm{H}=$ height of the liquid column (constant for all the liquids if taken for identical points and liquids are taken in equal volumes for a particular set of observations.)
$\mathrm{g}=$ acceleration due to gravity
$\mathrm{d}=$ density of the liquid

Since in this case for two liquids $h$ and $g$ are same hence,
$\eta_{1} / \eta_{2}=t_{1} d_{1} / t_{2} d_{2}$
This will give us the relative viscosity of the given liquid. Relative viscosity has no units. The absolute viscosity
$\boldsymbol{\eta}=\mathrm{d}_{1} / \mathbf{d}_{2} \mathbf{X} \mathrm{t}_{1} / \mathrm{t}_{2} \mathbf{X} \boldsymbol{\eta}_{\mathbf{2}}$


## Procedure:

1. Wash the viscometer with chromic acid $\left(\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}+\right.$ Conc. $\left.\mathrm{H}_{2} \mathrm{SO}_{4}\right)$ and then rinse it 2-3 times with distilled water. It is finally washed with alcohol and ether and then dried.
2. Attach a piece of clean rubber tube to the end of viscometer and clamp the viscometer vertically in air.
3. Now introduce a sufficient volume of the given liquid with the help of a pipette in bulb so that the bend portion of the U-tube and more than half of bulb are filled up.
4. Through the rubber tube, suck up the liquid until it rises above the mark. Make sure that there is no air bubble inside the liquid.
5. Now, allow the liquid to fall freely through the capillary upto the mark. Start the stopwatch and note the time $\mathrm{t}_{1}$ for the flow of the liquid from mark X to mark Y .

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6. Repeat the experiment thrice. The values should be concordant.
7. Remove the liquid and clean and dry the viscometer again.
8. Repeat the experiment by taking the same volume of the distilled water and note down the time taken t 2 for flow of water from mark X to y Repeat thrice.
9. Weigh the relative density bottle and note down its weight.
10. Fill it with the given liquid and weigh it again.
11. Remove the liquid. Wash it with chromic acid and then distilled water. Dry in the oven. Now fill it with the distilled water and weigh it.

## Observations:

Room temperature =. $\qquad$ ${ }^{0} \mathrm{C}$

| Sr. No. | Given Liquid |  |  | Water |
| :--- | :--- | :--- | :--- | :--- |
|  | Time of Flow | Mean $\left(\mathrm{t}_{1}\right)$ | Time of Flow | Mean $\left(\mathrm{t}_{2}\right)$ |
| 1. |  |  |  |  |
| 2. |  |  |  |  |
| 3. |  |  |  |  |

Weight of empty specific gravity bottle=.... $\mathrm{w}_{1} \mathrm{~g}$
Weight of empty specific gravity bottle + given liquid= $\qquad$ $\mathrm{w}_{2} \mathrm{~g}$

Weight of empty specific gravity bottle + distilled water= $\qquad$ $\mathrm{w}_{3} \mathrm{~g}$

## Calculation:

Density of solution/Density of water $=\mathrm{d}_{1} / \mathrm{d}_{2}=\mathrm{w}_{2}-\mathrm{w}_{1} / \mathrm{w}_{3}-\mathrm{w}_{1}$
Relative viscosity of the given liquid $=\eta_{1} / \eta_{2}=d_{1} / d_{2} X t_{1} / t_{2}$
Absolute viscosity $=\mathrm{d}_{1} / \mathrm{d}_{2} \mathrm{X} \mathrm{t}_{1} / \mathrm{t}_{2} \mathrm{X} \eta_{2}$
Result: The relative viscosity of the given solution is $\qquad$ at $\qquad$ ${ }^{0} \mathrm{C}$.

The absolute viscosity of the given solution is $\qquad$ Pas at $\qquad$ ${ }^{0} \mathrm{C}$.

## Questions:

1. What is the unit of coefficient of viscosity?
2. Define viscosity of a liquid.
3. How are the viscosity is measured and fluidity of a liquid related to each other?
4. What is the effect of temperature on viscosity of liquid?

Experiment No. $\qquad$ 9.....

## Date:

$\qquad$
Objective:To Study the kinetics of hydrolysis of ester and to calculate the rate constant.
Requirements: Ethyl/methyl acetate, dil. $\mathrm{HCl}, \mathrm{NaOH}$ solution, phenolphthalein indicator, burette, pipette, conical flask, stopwatch.

Principle: The hydrolysis of methyl acetate and ethyl acetate in presence of an acid may be represented as:

$$
\begin{aligned}
& \mathrm{CH}_{3} \mathrm{COOCH}_{3}+\mathrm{HOH} \longrightarrow \mathrm{CH}_{3} \mathrm{COOH}+\mathrm{CH}_{3} \mathrm{OH} \\
& \mathrm{CH}_{3} \mathrm{COOC}_{2} \mathrm{H}_{5}+\mathrm{HOH} \longrightarrow \mathrm{CH}_{3} \mathrm{COOH}+\mathrm{C}_{2} \mathrm{H}_{5} \mathrm{OH}
\end{aligned}
$$

The reaction is an example of a pseudo unimolecular reaction; the concentration of water is high and practically remains constant during the reaction. Concentration of the $\mathrm{H}^{+}$ions also remains constant. Thus the rate of reaction,
$K=\frac{2.303}{t} \log 10 \frac{a}{a-x}$
where, $\mathrm{a}=$ initial concentration.
and $(a-x)=$ concentration after time $t$
The values of $a,(a-x)$ and $t$ are taken from the experimentally determined values.
When a definite volume of the reaction mixture is titrated against standard alkali, then the volume of the alkali used at different time interval will be as :
$V_{0} \propto$ Initial acid added
$V_{\infty} \propto$ Initial acid added $+\mathrm{CH}_{3} \mathrm{COOH}$ formed at infinite time
$V_{\infty}-V_{0} \propto a$
$V_{\infty}-V_{t} \propto(a-x)$

## Procedure:

1. Take four conical flask of 150 ml each and add 5 ml of methyl/ ethyl acetate.
2. Fill the burette with standard NaOH solution.
3. Add 20 ml of standard HCl solution in the each conical flask and note down the exact time of addition for every conical flask separately.
4. Add about 10 to 20 ml ice cold water or few pieces of ice and two drops of phenolphthalein indicator in the fourth flask immediately after adding HCl solution, and titrate the mixture against NaOH solution till it become light pink in colour. Note the burette reading as $\mathrm{V}_{0}$
5. Titrate the second flask after 20 min . from the noted time, similarly and note the burette reading as $\mathrm{V}_{20}$.
6. Titrate the third flask after 40 minutes from the noted time and note the burette reading as $\mathrm{V}_{40}$.
7. Heat the fourth flask over hot plate at $50^{\circ} \mathrm{C}$ for 10 minutes and titrate the same against NaOH solution using phenolphthalein indicator. Note down the burette reading as $\mathrm{V}_{\infty}$.

## Observation table:

| Vol. of ester and acid | $\mathrm{V}_{0}$ |  |  | $\mathrm{V}_{20}$ |  |  | $\mathrm{V}_{40}$ |  |  | $\mathrm{V}_{\infty}$ |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | Initial | Fina <br> 1 | Diff | $\begin{aligned} & \hline \text { Initi } \\ & \text { al } \end{aligned}$ | Final | Diff | Initial | Final | Diff | Initi <br> al | Fina $1$ | Diff |
| 25 ml |  |  |  |  |  |  |  |  |  |  |  |  |

## Calculations:

As $\quad K=\frac{2.303}{t} \log \frac{a}{(a-x)}=\frac{2.303}{t} \log \frac{V_{\infty}-V_{0}}{V_{\infty}-V_{t}}$

Calculate the Value of $\mathrm{K}_{20}$ and $\mathrm{K}_{40}$ using the above formula.
Result: The value of $\mathrm{K}=$ $\qquad$ Minutes ${ }^{-1}$

## Precautions:

1. Titrate the first mixture just immediately after adding HCl .
2. Ice-cold water should be added during the titration.

Experiment No...10........

## Date:

$\qquad$
Objective: To find the cell constant of conductivity cell.
Requirements: Conductivity bridge (A.C. mains operated), conductivity cell, thermometer, beaker, burette, pipette, funnel, 0.01 N KCl Solution, distilled water.

## Description of Conductivity Bridge:

Power switch: This switch is on the back of the instrument. When it is switched on, the display on the front panel glows.

Cell control: This is at the back of the instrument. The display is adjusted to 1.000 with the help of CAL control.

Range Switch: This is on the front panel of the instrument. It has positions marked $0.2,2$, 20. These numbers stand for the full-scale meter values in millisiemens. For example when the switch is at 2 , the meter reads between O and 2 ms .

Temperature Knob. This is provided in the front panel of the instrument. This is to set the temperature control to the actual temperature of the solution (measured by the ordinary thermometer) under test.
Function Switch. This control is also on the front panel of the instrument. It has positions marked check, cell constant and conductivity. This is used to set the instrument in any of the above positions.
Display window. This window on the front panel displays the readings (cell constant or conductivity)

Cell constant knob: This knob, provided at the front panel is adjusted to the known value of the solution of KC1.


Conductivity Meter

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Principle: It is evident from the proceeding discussion that specific conductance of a solution is the conductivity of a solution contained between two parallel electrodes which have cross sectional area one $\mathrm{cm}^{2}$ and which are kept one cm apart. If R is the resistance of a solution measured by a cell having electrodes of cross sectional area ' $a$ ' $\mathrm{cm}^{2}$ and ' l ' cm apart, the specific conductance K of the solution is given by,

Where,

$$
\begin{aligned}
& \mathrm{C}=\text { observed conductance and } \\
& l / \mathrm{a}=\text { cell constant. }
\end{aligned}
$$

Thus to obtain the value of specific conductance, the observed conductance has to be multiplied by a certain factor known as cell constant. (The values of a and $l$ are constant for a particular cell so $l / a$ is constant and hence the name cell constant).

The cell constant i.e. the ratio $l / a$ can not be determined from the geometrical dimensions of the cell because the exact area of cross section of electrode or exact distance between them cannot be measured accurately. Thus cell constant is determined by an indirect method. From equation (ii)

$$
\begin{equation*}
l / \mathrm{a}=\mathrm{K} / \mathrm{C} . \tag{iii}
\end{equation*}
$$

or Cell constant $=\quad$ Specific conductance $/$ Observed conductance
For the determination of cell constant, a solution of known specific conductance is taken whose conductance is determined using the given cell. Thus cell con- stant can be determined using equation (iii). Solution of KCl of known concentration and known specific conductance are used for this purpose because KCl is strong electrolyte.

The mobility of $\mathrm{K}^{+}$and $\mathrm{Cl}^{-}$is quite comparable. Following Table gives the specific conductance values of $\mathrm{KCl}(0.01 \mathrm{M})$ at different temperatures.

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| Temp ( ${ }^{\circ} \mathrm{C}$ ) | 25 | 26 | 27 | 28 | 29 | 30 | 31 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Sp.conductance <br> ( $\mathrm{m} \mathrm{mhocm}^{-1}$ ) | 1.408 | 1.458 | 1.487 | 1.531 | 1.561 | 1.642 | 1.683 |
| Temp( ${ }^{\circ} \mathrm{C}$ ) | 32 | 33 | 34 | 35 | 36 | 38 |  |
| Sp.Conductance <br> ( $\mathrm{m} \mathrm{mhocm}^{-1 \text { ) }}$ | 1.731 | 1.777 | 1.818 | 1.862 | 1.916 | 1.968 |  |

## Procedure:

1. Wash the conductivity cell thoroughly with distilled water.
2. Rinse the conductivity cell repeatedly with KCl solution.
3. Take sufficient volume of KCl solution in a beaker and note down its temperature.
4. Connect the instrument to the mains and switch on the instrument using the power switch. Connect the electrode leads in the input sockets at the rear of the instrument.
5. Set the 'Function' switch to 'check' position and adjust the display to 1.000 with CAL control at the back panel.
6. Dip the conductivity cell in KCl solution and adjust the temperature knob of the conductivity bridge at the temperature of the KCl Solution.
7. Move the 'Function' switch to 'conductance' position and range position to appropriate range.
8. Adjust the cell constant knob so that the display reads the known value of the KCl solution at that temperature.
9. Bring the 'Function switch' to 'cell constant' position and read the value of the cell constant from the display window.

## Result:

The cell constant of the given cell is $\qquad$ $\mathrm{cm}^{-1}$

## Questions:

1. Define cell constant.
2. What is the effect of temperature on conductance?
3. How specific conductance and equivalent conductance vary with dilution?

Experiment No. :
11
1......

Date:
Objective: To find out the strength of a given strong acid solution by titrating it against strong base solution Conductometrically
Equipment Required: Conductometer, Conductivity cell, Magnetic stirrer/ Glass stirrer,
Beaker, Burette and Pipette.
Chemical required: NaOH solution, HCl solution, Distilled water
Principle: According to Kohlrausch the electrical conductance of a solution depends upon the number and mobality of ions. Let us consider the titration of a strong acid like HCl against a strong base like NaOH . Before the NaOH solution is added the acid solution has a high conductance due to the highly mobile hydrogen ion. As alkali solution is added the hydrogen ions are removed due to combination with the hydroxyl ions forming feebly ionized water $\left(\mathrm{H}_{2} \mathrm{O}\right)$ molecules and their place is taken by the much slower moving $\mathrm{Na}+$ ions
$\left[\mathrm{H}^{+}+\mathrm{Cl}^{-}\right]+\left[\mathrm{Na}^{+}+\mathrm{OH}^{-}\right] \longrightarrow \mathrm{Na}^{+}+\mathrm{Cl}^{-}+\mathrm{H}_{2} \mathrm{O}$ (feebly ionized)
Consequently the conductance of the solution decreases and continues to decrease on adding sodium hydroxide solution until equivalence point is reached. Any further addition of alkali means increase of $\mathrm{Na}+$ and fast moving hydroxyl ions and thus the conductance begins to increase If we plot the conductance measured against the volume of NaOH added the point of intersection will give the neutralization point.


Volume of NaOH added

## Significance:

Titration's involving coloured liquids are difficult to be performed using ordinary indicators. But these can be easily carried out by conductometric titration method.
Moreover, conductometric titration can be performed accurately even with dilute solutions

## Procedure:

1. Wash the conductivity cell thoroughly with distilled water.
2. Fit the electrode leads to the input sockets at the rear of the instrument
3. Calibrate the instrument as per the process mentioned in the instrument Manual.
4. Take 20 ml of the given acid solution whose strength is to be determined in a 100 ml beaker and dilute it by adding equal volume of water.
5. Clamp the conductivity cell in the beaker containing the acid solution.
6. Set the function switch to Cond. position.
7. Fill the NaOH solution in the burette.
8. Note down the conductance from the conductometer.
9. Keep on adding NaOH from the burette, 0.1 ml at a time. After each addition stir the
10. Mixture with magnetic stirrer or a glass rod and measure the conductance.

## Observation:

$$
\text { Volume of unknown acid solution taken } \quad=20 \mathrm{ml}
$$

## Calculation:

The values of observed conductance are plotted along Y axis against the volume of alkali added along the X axis. The point of intersection gives the amount of alkali required for neutralization of the acid

Suppose at the end point (or neutralization point) $\mathrm{V}_{2} \mathrm{ml}$ of 0.5 N NaOH is consumed, then:

| $\mathrm{N}_{1} \mathrm{~V}_{1}=$ | $\mathrm{N}_{2} \mathrm{~V}_{2}$ |
| :--- | :--- |
| $(\mathrm{HCl})$ | $(\mathrm{NaOH})$ |
| $\mathrm{N}_{1} \mathrm{X} \mathrm{20}$ | $=\quad 0.5 \times \mathrm{V}_{2}$ |

Strength $=$ Normality X Equivalent wt.
Equivalent wt. of $\mathrm{HCl}=36.5$

## Result:

Strength of the given $\mathrm{HCl}=$ $\qquad$ .gm/liter

## Questions:

1. Define specific conductance?
2. What is cell constant?
3. What do you mean by conductometric titration?
4. What are the advantages of conductometric titration over normal titration?
5. Explain the titration graph of $\mathrm{CH}_{3} \mathrm{COOH}$ against NaOH .

## Experiment No. :...12...

## Date:

Objective:To Perform a conductometric titration between a weak acid and strong base and to find out the strength of the acid.
Requirements: Burette, stand, pipette, glass rod, conductometer device, Standard acid and base.

Principle: When a weak acid is titrated against a strong base the initial conductance is very low due to the presence of less number of $\mathrm{H}^{+}$ions. The conductance of solution increases on addition of NaOH ( Strong Base) slowly due to the formation of salt which suppresses the ionization of weak acid As more of NaOH is added, highly ionized salt, sodium acetate is formed thereby increasing the conduction.

| $\mathrm{CH}_{3} \mathrm{COOH}$ |
| :--- | :--- |
| Weak Acid |$+$| $\mathrm{NaOH} \longrightarrow$ |
| :--- |
| Strong Base |$\quad$| $\mathrm{CH}_{3} \mathrm{COONa}^{+}$ |
| :--- |
| Highly ionized Salt |$+$| $\mathrm{H}_{2} \mathrm{O}$ |
| :--- |
| Unionized. |

When the acid is neutralized, further addition of NaOH causes a sharp rise in conductance due to addition of conducting $\mathrm{OH}^{-}$ions.


## Procedure:

## Step-I: Calibration of the instrument

1. Switch on the instrument and connect the conductivity cell with the socket available at the rear of the instrument.
2. Select the appropriate range of cell constant whatever written over the conductivity cell by pushing the appropriate knob in the front of the conductometer.
3. Wash the cell with distilled water and dip in the beaker containing Standard solution ( KCl ) select the calibration function to "calibrate".
4. Set the TEMP. control to 25 and adjust CAL control to get 1000 display on the readout.
5. Measure the temperature of the Standard solution and set the Temp control to the value of measured temperature.
6. Push the CAL/MEAS switch to MEAS position.
7. Adjust CELL ADJ. Control (at the rear panel) to set the value of the Conductivity of standard solution on the read out.

NOTE: Once the CELL ADJ. Control is set with a particular conductivity cell, it should not be disturbed.

## Step-II: Measurement of Conductance

1. Take $20-30 \mathrm{ml}$ of the acid and add an equal or known amount of distilled water in a beaker and deep the conductivity cell. Measure the temperature of the solution and adjust the same in the instrument by temp control knob.
2. The display in the instrument is the conductivity of the solution, note it down
3. Start adding base in the acid from the burette 0.5 to 1 ml at a time; stir the solution with a glass rod without disturbing the cell.
4. Note down the display reading when it becomes constant after every addition of base.

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5. Carry on the process of titration till sufficient number of readings has been taken well after the equivalence point.
6. Plot a graph between the conductance readings and volume of base added.

## Observation Table:

| S. No. | Volume of base added | Conductance |
| :--- | :--- | :--- |
| 1 |  |  |
| 2 |  |  |
| 3 |  |  |
| 4 |  |  |
| 5 |  |  |
| 6 |  |  |
| 7 |  |  |
| 8 |  |  |
| 9 |  |  |

## Calculations:

The Normality of the acid can be determined by using normality equation
$N_{1} V_{1}=N_{2} V_{2}$
where, $\quad \mathrm{N}_{1}=$ Normality of the acid
$\mathrm{N}_{2}=$ Normality of base.
$\mathrm{V}_{1}=$ Volume of acid taken for the titration
$\mathrm{V}_{2}=$ Volume of base added till the equivalence point ( Taken from the plotted Graph).

$$
N_{1}=\frac{N_{2} V_{2}}{V_{1}}
$$

Strength of the acid $=N_{1} X$ Equivalent weight of the acid gm/l

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## Result:

The strength of the given acid is $\qquad$ gm/l.

## Questions:

1. What is the advantage of adding water with acid in the step-II?
2. Why does the conductivity of the acid starts increasing on addition of base?
3. Discuss the pattern of graph obtained during this titration.
4. Discuss the conductometric titration of strong acid against weak base.

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## List of Content of Notebook

## Objective:

Materials:
Procedure:
Data:
Other Measurements:
Formulas:
Calculations/Results:
Simplified/Rounded Calculations:
Conclusion:
Partners:
References:

