ENGINEERING CHEMISTRY

LAB MANUAL

Year : **2019 - 2020**

Course Code : **AHSB09**
Regulations : **IARE - R18**
Class : **B.Tech I Semester**
Branch : **CSE**

Prepared by

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Assistant Professor

FRESHMAN ENGINEERING

INSTITUTE OF AERONAUTICAL ENGINEERING
(Autonomous)
Dundigal, Hyderabad - 500 043

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Vision

To bring forth professionally competent and socially sensitive engineers, capable of working across cultures meeting the global standards ethically.

Mission

To provide students with an extensive and exceptional education that prepares them to excel in their profession, guided by dynamic intellectual community and be able to face the technically complex world with creative leadership qualities.

Further, be instrumental in emanating new knowledge through innovative research that emboldens entrepreneurship and economic development for the benefit of wide spread community.

Quality Policy

Our policy is to nurture and build diligent and dedicated community of engineers providing a professional and unprejudiced environment, thus justifying the purpose of teaching and satisfying the stake holders.

A team of well qualified and experienced professionals ensure quality education with its practical application in all areas of the Institute.

Philosophy

The essence of learning lies in pursuing the truth that liberates one from the darkness of ignorance and Institute of Aeronautical Engineering firmly believes that education is for liberation.

Contained therein is the notion that engineering education includes all fields of science that plays a pivotal role in the development of world-wide community contributing to the progress of civilization. This institute, adhering to the above understanding, is committed to the development of science and technology in congruence with the natural environs. It lays great emphasis on intensive research and education that blends professional skills and high moral standards with a sense of individuality and humanity. We thus promote ties with local communities and encourage transnational interactions in order to be socially accountable. This accelerates the process of transfiguring the students into complete human beings making the learning process relevant to life, instilling in them a sense of courtesy and responsibility.

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Program Outcomes	
PO1	**Engineering knowledge**: Apply the knowledge of mathematics, science, engineering fundamentals, and an engineering specialization to the solution of complex engineering problems.
PO2	**Problem analysis**: Identify, formulate, review research literature, and analyze complex engineering problems reaching substantiated conclusions using first principles of mathematics, natural sciences, and engineering sciences.
PO3	**Design/development of solutions**: Design solutions for complex engineering problems and design system components or processes that meet the specified needs with appropriate consideration for the public health and safety, and the cultural, societal, and environmental considerations.
PO4	**Conduct investigations of complex problems**: Use research-based knowledge and research methods including design of experiments, analysis and interpretation of data, and synthesis of the information to provide valid conclusions.
PO5	**Modern tool usage**: Create, select, and apply appropriate techniques, resources, and modern engineering and IT tools including prediction and modeling to complex engineering activities with an understanding of the limitations.
PO6	**The engineer and society**: Apply reasoning informed by the contextual knowledge to assess societal, health, safety, legal and cultural issues and the consequent responsibilities relevant to the professional engineering practice.
PO7	**Environment and sustainability**: Understand the impact of the professional engineering solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.
PO8	**Ethics**: Apply ethical principles and commit to professional ethics and responsibilities and norms of the engineering practice.
PO9	**Individual and team work**: Function effectively as an individual, and as a member or leader in diverse teams, and in multidisciplinary settings.
PO10	**Communication**: Communicate effectively on complex engineering activities with the engineering community and with society at large, such as, being able to comprehend and write effective reports and design documentation, make effective presentations, and give and receive clear instructions.
PO11	**Project management and finance**: Demonstrate knowledge and understanding of the engineering and management principles and apply these to one’s own work, as a member and leader in a team, to manage projects and in multidisciplinary environments.
PO12	**Life-long learning**: Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change.

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Program Specific Outcomes – Computer Science and Engineering	
PSO1	**Professional Skills:** The ability to understand, analyze and develop computer programs in the areas related to algorithms, system software, multimedia, web design, big data analytics, and networking for efficient design of computer-based systems of varying complexity.
PSO2	**Problem-Solving Skills:** The ability to apply standard practices and strategies in software project development using open-ended programming environments to deliver a quality product for business success.
PSO3	**Successful Career and Entrepreneurship:** The ability to employ modern computer languages, environments, and platforms in creating innovative career paths to be an entrepreneur, and a zest for higher studies.

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ATTAINMENT OF PROGRAM OUTCOMES & PROGRAM SPECIFIC OUTCOMES

EXPT.No.	CSE	
	Program Outcomes Attained	Program Specific Outcomes Attained
I	PO1, PO2, PO3	PSO2, PSO3
II	PO1, PO2, PO3	PSO2, PSO3
III	PO1, PO2, PO3	PSO2, PSO3
IV	PO1, PO2, PO3	PSO2, PSO3
V	PO1, PO2, PO3	PSO2, PSO3
VI	PO1, PO2, PO3	PSO2, PSO3
VII	PO1, PO2, PO3, PO5	PSO2, PSO3
VIII	PO1, PO2, PO3	PSO2, PSO3
IX	PO1, PO2, PO3, PO5	PSO2, PSO3
X	PO1, PO2, PO3, PO5	PSO2 ,PSO3
XI	PO1, PO2, PO3, PO5	PSO2, PSO3
XII	PO1, PO2, PO3, PO5	PSO2, PSO3

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Certificate

This is to certify that it is a bonafied record of Practical work done by Sri/Kum. ____ bearing the Roll No. ________________________ of _____________ class ____ branch in the ______________________________ laboratory during the academic year ____________________ under our supervision.

Head of the Department

Lecture In-Charge

External Examiner

Internal Examiner

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II	Estimation Of Total Hardness Of Water By Complexometric Method Using EDTA			
III	Estimation Of Hcl By Conductometric Titrations			
IV	Estimation Of Hcl By Potentiometric Titrations.			
V	Estimation Of Acetic Acid By Conductometric Titrations			
VI	Estimation Of Fe^{+2} By Potentiometry Using $Kmno_4$			
VII	Determination Of Chloride Content Of Water By Argentometry			
VIII	Determination Of Surface Tension Of A Given Liquid Using Stalagmometer			
IX	Determination Of Viscosity Of Liquids By Ostwald's Viscometer			
X	Determination of Rate constant of Ester catalysed by an acid			
XI	Verification Of Freundlich Adsorption Isotherm-Adsorption Of Acetic Acid On Charcoal			
XII	Thin Layer Chromatography Calculation Of R_f Values. Eg:Ortho And Para Nitro Phenols			

Chemistry Lab Do’s and Don’ts

The chemistry laboratory must be a safe place in which to work and learn about chemistry. Most of these involve just using common sense.

1. Wear chemical splash goggles at all times while you are in the laboratory.
2. Wear a chemical-resistant apron.
3. Be familiar with your lab assignment **before** you come to lab. Follow all written and verbal instructions carefully. Observe the safety alerts in the laboratory directions. If you do not understand a direction or part of a procedure, ask the teacher before proceeding.
4. When entering the lab/classroom, do not touch any equipment, chemicals, or other materials without being instructed to do so. Perform only those experiments authorized by the instructor.
5. No student may work in the laboratory without an instructor present. Work only with your lab partner(s). Do not venture to other lab stations for any reason.
6. Do not wear bulky or dangling clothing.
7. Never eat or drink in the laboratory. Don't chew on the end of a pen which was lying on the lab bench.
8. Wash acid, base, or any chemical spill off of yourself immediately with large amounts of water. Notify your teacher of the spill.
9. Clean up spills immediately. If you spill a very reactive substance such as an acid or base, notify the people in the area and then obtain assistance from your teacher. Acid spills should be neutralized with baking soda, base spills with vinegar before cleaning them up.
10. If chemical substances get in your eye, wash the eye out for 15 minutes. Hold your eye open with your fingers while washing it out.
11. If you take more of a chemical substance from a container than you need, you should not return the excess to the container. This might cause contamination of the substance remaining. Dispose of the excess as your teacher directs.
12. When weighing never place chemicals directly on the balance pan. **Never weigh a hot object**.
13. Never smell anything in the laboratory unless your teacher tells you it is safe. Do not smell a substance by putting your nose directly over the container and inhaling. Instead, waft the vapors toward your nose by gently fanning the vapors toward yourself.
14. Do not directly touch any chemical with your hands. Never taste materials in the laboratory.
15. If you burn yourself on a hot object, immediately hold the burned area under cold water for 15 minutes. Inform your teacher.
16. Observe good housekeeping practices. Work areas should be kept clean and tidy at all times. Only lab notebooks or lab handouts should be out on the table while performing an Experiment. Books and book bags should not be on the lab table. Passageways need to be clear at all times.
17. Always replace lids or caps on bottles and jars.
18. If your Bunsen burner goes out, turn the gas off immediately.

19. Constantly move a test tube when heating it. Never heat a test tube that is not labeled Pyrex and never point the open end at anyone.
20. Always add acid to water and stir the solution while adding the acid. Never add water to an acid.
21. Report all accidents to your teacher.
22. Absolutely no running, practical jokes, or horseplay is allowed in the Laboratory.
23. Thoroughly clean your laboratory work space at the end of the laboratory Session. Make sure that all equipment is clean, and returned to its original place.

Importance of Engineering Chemistry Laboratory:

The experiments to be performed in the Engineering Chemistry Laboratory as per the curriculum of **AUTONOMOUS** regulations are very useful to the students of I B.Tech of different disciplines. The various **experiments included in the syllabus are the basics of Engineering Chemistry that are useful for different** industries. To mention, Complexometric titrations to find Hardness of water, understanding the basic concepts of Electrochemistry like Conductometry and Potentiometry, importantly preparation of a pain killer Aspirin, preparation of a industrially useful polymer (Thiokol) rubber. In all the above experiments, the students would learn about handling of the equipment and conceptualization of certain fundamental aspects of Chemistry.

EXPERIMENT-I

SYNTHESIS OF ASPIRIN

AIM:

To prepare Aspirin from salicylic acid.

APPARATUS:

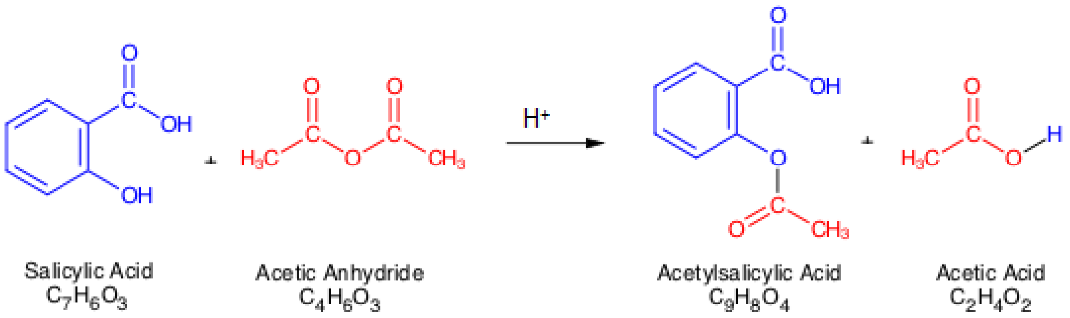
Conical flask, Beakers, glass rod, funnel etc.

CHEMICALS REQUIRED:

1.Salicylic acid - 2.5gm (MW= 138)
2.Acetic anhydride - 3.5ml
3.Con.H_2SO_4 - 3-4drops

PRINCIPLE:

Salicylic acid is a phenolic acid. The phenolic group can be easily acetylated using acetic anhydride. This is an example of Nucleophilic substitution reaction. Phenolic hydroxyl group of salicylic acid acts as a Nucleophile and lone pair of electrons on the Oxygen atom attacks the carbonyl group of acetic anhydride to form Aspirin.



PROCEDURE:

Take all the three chemical compounds in a given 250ml conical flask. Shake the mixture thoroughly and warm the reaction mixture at 50-60^{o}C for 15 min. on a water bath with continuous stirring with glass rod and allow the reaction mixture to cool. Add nearly 100ml of distilled water. Stir thoroughly and filter the product.

RECRYSTALIZATION:

Dissolve the product in 20ml alcohol and pour the solution into warm water, If solids separate warm it to dissolve the solids and clear the solution and allow it to cool slowly to get beautiful needles of Aspirin(MW=180) is formed.

RESULT:

Percentage yield of product ______%

VIVA QUESTIONS:

1) Define Nucleophilic substitution reaction?
2) What is molecular weight of aspirin?
3) What is the another name of aspirin?
4) What are applications of aspirin?
5) Write the chemical equation for synthesis of aspirin?

EXPERIMENT-II

ESTIMATION OF TOTAL HARDNESS OF WATER BY COMPLEXOMETRIC METHOD USING EDTA

AIM :

To estimate the total hardness, permanent hardness and temporary hardness of water by using standard solution of EDTA

APPARATUS :

Burette, pipette, Conical flask, Beakers, Standard flask, Burette stand and funnel etc.,

CHEMICALS REQUIRED :

Magnesium sulphate, Buffer, Disodium salt of EDTA, Eriochrome black-T or Solochrome balck -T etc.

PRINCIPLE :

Hard water which contains Ca^{2+} andMg^{2+} ions which forms wine red color complex with the indicator

Ca^{2+} (or) Mg^{2+} + EBT ———> Ca-EBT (or) Mg-EBT
(Wine red colour complex)

EDTA forms a colour less complex with the metal ions (Ca^{2+} andMg^{2+})

Ca-EBT (or) Mg-EBT + EDTA———> Ca-EDTA (or) Mg-EDTA + EBT
(Wine red colour complex) Colorless stable complex) (Blue)

When free ions are not available, EDTA extracts the metal from (ion) metal ion indicator complex, there by releasing the free indicator.

PROCEDURE :

STEP-I

PREPARATION OF STANDARD SOLUTION OF $MgSO_4$:

Weigh the approx 0.25gm of $MgSO_4$ and transfer into 100ml standard flask through the funnel and dissolve in minimum quantity of distilled water. Make up the solution up to the mark with distilled water and shake the flask well for uniform concentration then calculate the Molarity of $MgSO_4$

Molecular Weight of $MgSO_4$ = 246.48gm
Molarity of $MgSO_4$=0.01M

STEP-II

STANDARDISATION OF EDTA SOLUTION:

Pipette out 20ml of $MgSO_4$ solution into a clean conical flask. Add 2ml of buffer solution and add 2 to 3 drops of EBT indicator and it gets wine red color solution Take EDTA solution in a burette after titrate with EDTA solution till wine red color changes to blue color. Note the burette reading and repeat the titration to get concurrent values.

S. No	**Volume of $MgSO_4$ in ml**	**Burette Reading (ml)**		**Volume of EDTA consumed (ml)**
		Initial	**Final**	
1	20			
2	20			
3	20			

M_1 = $MgSO_4$ molarity M_2 = EDTA molarity
V_1 = volume of $MgSO_4$ V_2 = volume of EDTA consumed

$$M_1V_1 = M_2V_2$$

$$M_2= M_1V_1/ V_2$$

STEP-III

STANDARDISATION OF HARD WATER:

Pipette out 20ml of tap water into a 250ml conical flask add 2 ml of buffer solution and add 2-3drops of EBT indicator. Titrate the wine red color solution with EDTA taken in burette, till a blue color end point is obtained. Repeat the titration to get concurrent values.

S. No	Volume of Hard water in(ml)	Burette Reading (ml)		Volume of EDTA consumed (ml)
		Initial	Final	
1	20			
2	20			
3	20			

M_3 = molarity of hard water M_2 = EDTA molarity
V_3= volume of Hard water V_2^l = volume of EDTA consumed

$$M_3V_3 = M_2V_2^l$$

$$M_3 = M_2V_2^l / V_3$$

Total hardness= M_3X100X1000 = -----------PPM

STEP-IV

STANDARDISATION OF PERMANENT HARDNESS OF WATER :

Pipette out 100ml of hard water sample into a beaker containing 250ml and boil the water till volume reduces to 50ml (all the bicarbonates of Ca^{2+}, Mg^{2+} decomposes to$CaCO_3$and $Mg(OH)_2$ respectively). Cool the solution and filter the water into beaker then pipette out 20ml of this cool water sample in to 250ml conical flask add 2ml of buffer solution and 2-3 drops of EBT indicator. Titrate the wine red color solution with EDTA taken in the burette, till a blue colored solution end point is obtained. Repeat the titration to get concurrent values.

S. No	**Volume of Hard water in (ml)**	**Burette Reading(ml)**		**Volume of EDTA consumed(ml)**
		Initial	**Final**	
1	20			
2	20			
3	20			

M_4 = molarity of hard water M_2 = EDTA molarity
V_4 = volume of hard water V_2^{ll} = volume of EDTA consumed

$$M_4V_4 = M_2\, V_2^{ll}$$

$$M_4 = M_2\, V_2^{ll}/\, V_4$$

Permanent hardness = M4X100X1000 = --------------PPM

RESULT:

1) Total hardness in _______ PPM
2) Permanent hardness in _______ PPM
3) Temporary hardness in _______ PPM (Total hardness – Permanent hardness)

VIVA QUESTIONS:

1. How many types of Hardness are there?
2. What is the indicator used in this experiment?
3. What is the name of the buffer solution used in EDTA titration?
4. What are the units of Hardness?
5. Why hardness is expressed in equivalents of calcium carbonate?

EXPERIMENT-III

ESTIMATION OF HCL BY CONDUCTOMETRIC TITRATIONS.

AIM :

To determine the strength of the strong acid by titration with strong base Conductometrically.

APPARATUS :

Digital Conductivity meter, Conductivity cell, Burette, Beakers, Measuring Cylinder, Burette Stand etc.

CHEMICALS REQUIRED :

Sodium hydroxide (NaOH), Hydrochloric acid (HCl)

PRINCIPLE :

At first solution contain H^+ and Cl^- ions. Since H^+ ions posses greater mobility it follows that the conductivity is mainly due to H^+ ions. The addition of NaOH is represented by the equation.

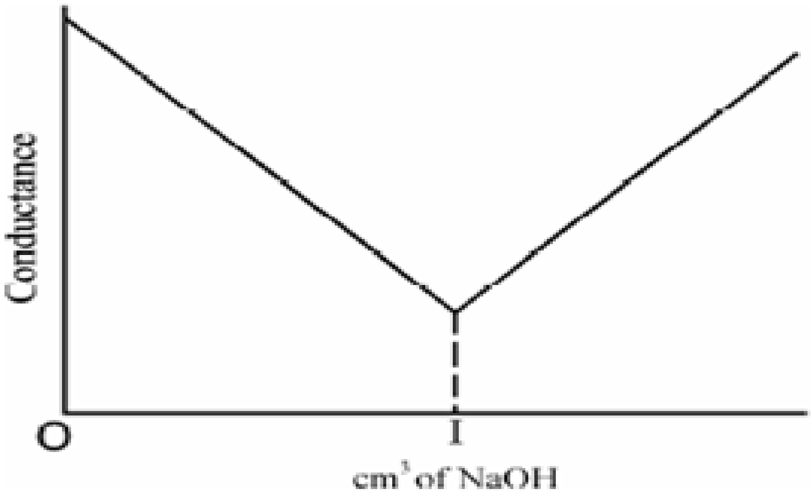
$$HCl + NaOH \longrightarrow NaCl + H_2O$$

As NaOH is added the H^+ ions are removed.The conductivity decreases as Na^+ ions do not process much mobility.
As the neutralization point and solutions contains Na+ ions and Cl- ions and will have minimum conductance value.
If NaOH is further added this will add OH- ions and so the conductivity increases.

PROCEDURE :

A standard solution of 0.2N NaOH is prepared. Similarly 0.1N HCl is prepared. 20 ml of HCl is taken in a 100 ml beaker and to it 20 ml of distilled water is added and kept in a thermostat. The conductivity cell is washed with distilled water and rinsed with acid soln. The cell is kept in acid containing beaker and it is connected to the bridge.The conductivity of the sol^n is measured by adjusting the reading. NaOH sol^n is taken into burette and add 1 ml of sol^n to acid, stirred well and conductance is measured. Each time 1 ml of base is added to acid stirred well and the conductance is measured. For every instance. Equal numbers of values are taken on either side of the point of maximum. Repeat the procedure of addition of 1 ml NaOH and noting the conductivity of the resulting solution. Take 20-25 readings

GRAPH:



CALCULATIONS :

S. No	Volume of NaOH (ml)	Observed conductance (ms)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		

FORMULA:

$N_1V_1 = N_2V_2$

RESULT:

The normality of strong acid (HCl) determined by titrating against a strong base (NaOH) =________N

VIVA QUESTIONS:

1. When strong acid combines with a strong base what type of reaction occurs?
2. Why conductance decreases on addition of NaOH to HCI ?
3. What is the unit for conductance?
4. How the end point for a particular reaction is calculated using this, titration method?
5. How conductance is related to the concentration of the ions?

EXPERIMENT-IV

ESTIMATION OF HCL BY POTENTIOMETRIC TITRATIONS.

AIM:

To determine the strength of the strong acid by titration with strong base Potentiometrically.

APPARATUS:

Potentiometer, Platinum electrode, Calomel electrode, Burette, Beakers, Standard flask, pipette, Burette Stand etc.

CHEMICALS REQUIRED:

Sodium hydroxide, Hydrochloric acid and Quinhydrone powder etc.

PRINCIPLE:

The cell will have certain emf depending upon pH value of the solution, on adding small portions of alkali to an acid. The acid potential changes slowly at first since the change in electrode depends on the fraction of hydrogen ions removed. After addition of certain amount of alkali the titration of hydrogen ions removed by alkali increases, correspondingly there is a rapid decrease in emf on addition of excess of alkali. The emf again shows a flow change. If a graph is plotted by taking volume of alkali added on X- axis and change in emf by point of intersection on Y-axis, a curve is obtained.

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

The cell can be represented as

H_2, Pt/Acid sol^n // KCl (aq) / Calomel electrode

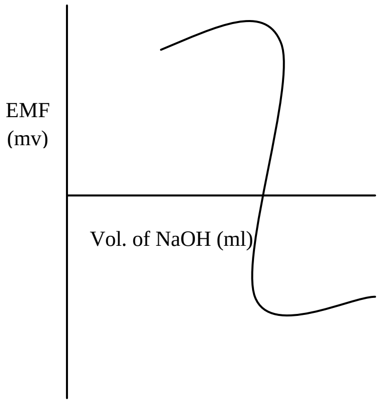
PROCEDURE:

Calibrate the instrument before starting the experiment. Approximately 0.1N HCl is prepared and standard decinormal solution of NaOH is prepared. Exactly 20 ml of the acid is pipette out into a clean 100ml of beaker and a pinch of Quinhydrone is added which acts as indicator. Platinum electrode and calomel electrodes are dipped in the solution.

The alkali (NaOH) against which the acid is being titrated is taken in burette. The solution is stirred well with a glass rod. The end reading is taken after adding definite amount of alkali. Finally after knowing the range in which the end point can be located, the whole experiment is repeatedly adding in steps of 1 ml in the end point.

GRAPH:

Two graphs are plotted of which one is between volume of alkali and observed emf and other is between volume of alkali and $\Delta E/\Delta V$ Sigmoid curve



CALCULATIONS:

S. No	**Volume of NaOH (ml)**	**Observed EMF (mv)**
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		

FORMULA:

$N_1V_1 = N_2V_2$

RESULT:

The normality of strong acid (HCl) determined by titrating against a strong base (NaOH) =________N

VIVA QUESTIONS:

1. What are the electrodes used in the potentiometric titrations?
2. What is calomel electrode?
3. What is the advantage of potentiometric titrations?
4. What is reference electrode? Give examples?
5. What is the indicator electrode?

EXPERIMENT- V

ESTIMATION OF ACETIC ACID BY CONDUCTOMETRIC TITRATIONS

AIM:

To determine the strength of the mixture of acids by titration with strong base Conductometrically.

APPARATUS:

Digital Conductivity meter, Conductivity cell, Burette, Beakers, Measuring cylinder, Burette Stand etc.

CHEMICALS REQUIRED:

Sodium hydroxide (NaOH), Hydrochloric acid (HCl) and Acetic acid (CH_3COOH) etc…

PRINCIPLE:

The conductivity of the solution is related to the mobility of ions which in turn related with the size of the ions.When a mixture of acids like a strong acid (HCl) and weak acid (acetic acid) are titrated against a strong base (NaOH), strong acid reacts first followed by a weak acid.When the titration of strong acid and strong base are carried out, there is a decrease in conductivity as highly mobilized hydrogen ions are replaced by sodium ions.

NaOH + HCl -------> NaCl + H_2O

When the whole strong acid is consumed, base reacts with weak acid and conductivity increases as unionized weak acid becomes the ionized salt.

CH_3COOH + NaOH----------> CH_3COONa + H_2O

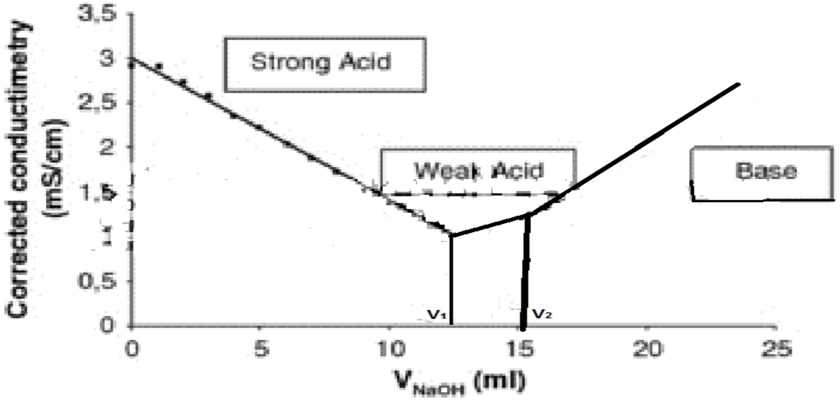
After both the acids are consumed, there is a steep increase in conductivity which gives the end point and this increase in conductivity is due to the fast moving hydroxyl ions from the base. From this, amount of base consumed for acid and in turn, the amount of acids present is calculated.

Let V_1 and V_2 ml be the volumes of alkaline corresponding beaker resp. Then V_1 ml of NaOH equivalent to HCl, $(V_2-V_1)_S$ ml of NaOH equivalent to CH_3COOH

FORMULA:

$$N_1V_1 = N_2V_2$$

GRAPH:



PROCEDURE :

1) Fill the burette with standard 0.2N NaOH solution
2) Take the given mixture (25 ml of CH_3COOH + 25 ml of HCl) in a small beaker and titrate against NaOH Conducto metrically.
3) Add 1ml of NaOH from burette and stir well and note down the conductance of solution.
4) Repeat the determination by stirring well after each addition of 1ml of NaOH until add up to 25-30 ml of NaOH.
5) Plot a graph between volume of NaOH Vs Conductance.

OBSERVATIONS AND CALCULATIONS:

S. No	Volume of NaOH (ml)	Observed conductance (ms)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		

The volume of NaOH corresponding to the first point of inflectionsis $V_1 = X_1$ ml and that corresponding to the second point of inflectionis $V_2 = X_2$ ml.

$V_2 - V_1 = X_3$ ml

Hence the HCl and CH_3COOH in their mixture of volume (50ml) consume respectively X_1ml and X_3 ml
There fore

(a). Strength of HCL = $\dfrac{X_1\text{ml x con. of NaOH}}{20\text{ml}}$ = ----------N

(b). Strength of CH_3COOH = $\dfrac{X_3\text{ml x con. of NaOH}}{20\text{ml}}$ = --------N

RESULT:

1) Strength of given HCl = ________________N
2) Strength of given CH_3COOH = ______________N

VIVA QUESTIONS:

1. When acetic acid combines with a strong base what type of reactionoccurs?
2. Why conductance decreases on addition of NaOH to CH_3COOH ?
3. What is the unit for conductance?
4. How the end point for a particular reaction is calculated using this, titration method?
5. How conductance is related to the concentration of the ions?

EXPERIMENT-VI

ESTIMATION OF Fe^{+2} BY POTENTIOMETRY USING $KMNO_4$

AIM:

To estimate the amount of ferrous iron present in the whole of the given solution by potentiometry.

PRINCIPLE:

The total reaction is

$2KmnO_4 + 8H_2So_4 + 10FeSO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 8H_2O$

PREPARATION OF SOLUTIONS:

1. Preparation of standard $KMnO_4$:

Dissolve 1.58gm of $KMnO_4$ in 1000 ml of distilled water and shake the flask well for uniform concentration. Because $KMnO_4$ is a secondary standard, it is standardized by titrating with standard oxalic acid.

2. Preparation of standard oxalic acid solution:

Weigh out accurately 0.315gms of oxalic acid crystals into a 100ml standard flask, dissolve the sample in little distilled water and make up the solution to the mark distilled water and shake the flask well for uniform concentration.

3. Preparation of 2N H_2SO4 solution:

56ml of conc.H_2So_4 is added Drop by Drop to 1000ml of water taken in a beaker by keeping it in a trough of water. This reaction is highly exothermic.

Note: H_2SO4 must not be added to water.

PROCEDURE:

Standardization of $KMnO_4$ solution:

Pipette out 20ml of the oxalic solution into a 250ml conical flask and add 20ml of 2N H_2SO4. Heat the solution to 70 – 80 °C. (till fumes come out of the solution) and titrate the warm solution with $KMnO_4$ taken in a burette. The end point is pale pink color. Note the burette reading and repeat the titration for concurrent values.

Let the titre value be xml.

$V_1 N_1 = V_2 N_2$

V_1 = Volume of oxalic acid

V_2 = Volume of $KMnO_4$

N_1 = Normality of oxalic acid

N_2 = Normality of $KMnO_4$

$$(N_2)\ \text{Normality of } KMnO_4 = \frac{20 * Normality\ \ of\ Oxalic\ acid}{x}$$

Potentiometric Titration:

The given Fe^{+2} solutions is made up to the mark of the given 100 ml standard flask and shake the flask well for uniform concentration. Pipette out 20ml of the Fe^{+2} solution into a clean 250ml beaker and add equal volume(20 ml) of dil. H_2SO_4 along with 100ml of distilled water to enable the electrodes to immerse well in the solution. A platinum electrode (indicator electrode) and a standard calomel electrode (reference electrode) from the potentiometer are dipped in to the beaker. The solution in the beaker is stirred using a magnetic stirrer. The initial EMF is noted 0.5 ml of $KMnO_4$ solution is added from the burette at regular intervals of time, while stirring the solution and the EMF is measured. The volume of $KMnO_4$ added and the corresponding EMF readings are noted. At the end point there is a sharp increase in EMF due to the complete oxidation of the Fe^{+2} to Fe^{+3}. The addition of $KMnO_4$ is continued till the equivalent point is crossed by at least 5ml.

From the observed values:

$\Delta E = E_2 - E_1$

$\Delta V = V_2 - V_1$

$\Delta E / \Delta V$ is calculated for each addition of $KMnO_4$ and a graph is drawn by pointing $\Delta E / \Delta V$ (y-axis) against volume of $KMnO_4$ added (x axis). A smooth curve is drawn by joining all the points. The peak in the graph indicates the end point as shown below. From the amount of Fe^{+2} is calculated.

CALCULATIONS:

$V_2 N_2 = V_3 N_3$

V_2 = Volume of $KMnO_4$

N_2 = Normality of $KMnO_4$

V_3 = Volume of Fe^{+2}

N_3 = Normality of Fe^{+2}

$$N_3 = \frac{y\ (20 * \text{Normality of } KMnO_4)}{20}$$

RESULT:

Amount of Fe^{+2} present in the given solution $= \underline{N_3 * \text{equivulent wt}}$

$= \underline{N_3 * 55.85}$ gms/100ml

VIVA QUESTIONS:

1. What is redox reaction?
2. What is the example for strong oxidizing agent?
3. What is the reaction occurs between ferrous ion and ferric ion?
4. What is relation between Molarity and Normality?
5. Define Normality?

EXPERIMENT-VII

DETERMINATION OF CHLORIDE CONTENT OF WATER BY ARGENTOMETRY.

AIM:

To estimate the chloride concentration in the given water sample

APPARATUS:

Burette, Burette stand, pipette, Conical flask, Beakers, Standard flask, and funnel etc.,

CHEMICALS REQUIRED:

Silver Nitrate($AgNO_3$), Potassium chromate(K_2CrO_4), Distilled Water, Raw Water

PRINCIPLE:

The sample of water containing chlorides is titrated with silver nitrate solution.Chlorides are precipitated as white silver chlorides the potassium chromate used as indicator ,which supplies chromate ions. As the concentration of chloride ions opposes extinction the silver ion concentration increases to a level at which reddish brow precipitate of silver chromate is formed indicating the end point.

FORMULAE:

Amount of chloride content present in water sample = (V2-V1) X 35.5 X 1000/ Volume of Sample

Where N = Normality of $AgNO_3$ = 0.01 N

PROCEDURE:

1. Wash all the apparatus with water thoroughly
2. Fill the burette with silver nitrate and fix it to the burette stand
3. Pipette out the 20ml of distilled water from beaker into a conical flask
4. Add about 1ml of potassium chromate indicator to a solution in conical flask
5. Titrate the solution with silvernitrate until the solution becomes reddish brown
6. Tabulate all the readings and calculate the volume of $AgNO_3$ consumed by using the formula.
7. Amout of chloride content is obtained in mg/lit (or) ppm.

PRECAUTIONS:

1. The glass apparatus used in the experiment should be washed with distilled water
2. All the reagents should be freshly prepared
3. The end point of the titration should be observed carefully
4. The volume of the indicator should be small in all titrations.

Tabular column:

S.NO	Volume of Sample	Name Of the Sample	Burette Reading		Volume Of $AgNO_3$ Consumed
			Initial	Final	
1					
2					
3					

Caliculations:

Amount of chloride content present in water sample

= (V2-V1) X 35.5 X 1000/ Volume of ample

RESULT:

The amount of total chloride content present in water sample

VIVA QUESTIONS:

1) Define argentometry?
2) What is the indicator used in argentometry?
3) What are the units for Chloride content?
4) Define Standard solution?
5) Give the examples for Primary and Secondary standard solutions?

EXPERIMENT-VIII

DETERMINATION OF SURFACE TENSION OF A GIVEN LIQUID USING STALAGMOMETER

AIM:

To determine surface tension of liquids by using stalagmometer.

APPARATUS:

Stalagmometer, Density bottle, Thermometer, Beakers, Burette Stand etc.

CHEMICALS REQUIRED:

Distilled water, Acetone, Test liquid etc.

PRINCIPLE:

The force in dynes acting on a surface at right angles at any line of unit length is called Surface tension.

$$\text{Surface tension } (\gamma) = \frac{F}{L}$$

THEORY:

DROP NUMBER METHOD:

It is the simplest method for determination of surface tension of liquids in the laboratory. This method is based on the principle that a fixed volume (weight of liquid W) of the liquid is delivered as freely falling from Capillary tube held vertically. Surface tension is directly proportional to weight i.e. $W \alpha \gamma$

Circumference of drop = 2π r is equal to length of the bar .Hence, the equation is represented as

$$\gamma = F/2\pi r$$

$$\gamma = mg/2\pi r$$

If ‘V’ volume contains 'n' drops then weight of a single drop is $\gamma 2 \pi r = \rho vg/n$

If density of two liquids ρ_1 and ρ_2 the no. of drops of two liquids be n_1 and n_2 of the same volume of liquids from two fixed points respectively. Applying equation and radius of tube is same.

$$\gamma = v \rho g/2 \pi r n$$

$$\gamma_1 = v \rho_1 g/2 \pi r n_1 \text{-----------} (1)$$

$$\gamma_2 = v \rho_2 g/2 \pi r n_2 \text{-----------} (2)$$

$$\boxed{\text{Formula } \gamma_1 / \gamma_2 = \rho_1 n_2 / \rho_2 n_1}$$

$\rho_1 \Rightarrow$ density of water (0.998 at 25^oC)

$\rho_1 \Rightarrow$ density of given sample

$\gamma_1 \Rightarrow$ Surface tension of water at room temp. I.e. 72.8 dynes/cm

$\gamma_2 \Rightarrow$ Surface tension of liquid to be determined

$n_1, n_2 \Rightarrow$ no. of drops of water and liquid by stalagmometer.

PROCEDURE:

a) Thoroughly clean the density bottle and stalagmometer using chromic acid and purified water
b) Stalagmometer must be mounted in vertical plane using burette stand
c) Fill the purified water in the instrument and count the number of drops falling between two points of instrument then Repeat this step at least three times
d) Rinse the stalagmometer using the same liquid whose surface tension is to be determined
e) Fill the stalagmometer by liquid and count the number of drops falling down between the two points as in step (c) and Repeat step (e) at least three times
f) Density of water and liquid is determined using density bottles

CALCULATIONS:

S. No	SAMPLE	NUMBER OF DROPS				DENSITY	SURFACE TENSION
		I	II	III	MEAN		

Weight of empty density bottle (W_1) = __________ gm

Weight of empty bottle + water (W_2) = __________ gm

Weight of empty bottle +liquid (W_3) = __________ gm

Density of water (ρ_{1}) = ________

Density of liquid (ρ_{2}) = ________

$$\gamma_2 = \frac{\rho_2 n_1}{\rho_1 n_2} \times \gamma_1$$

RESULT:

Surface tension of given liquid at room temperature = _________ dynes/cm

VIVA QUESTIONS:

1) Define Surface Tension?
2) What is the need of Surface Tension?
3) What is the formula for Surface Tension?
4) What are the units of Surface Tension?
5) What is the effect of Temperature on Surface Tension?

EXPERIMENT-IX

DETERMINATION OF VISCOSITY OF LIQUIDS BY OSTWALD'S VISCOMETER

AIM:

To determine the absolute viscosity of a liquid by using Oswald’s viscometer.

APPARATUS:

Oswald’s viscometer, stop watch, density bottle, rubber bulbs, Beakers, etc.

CHEMICALS REQUIRED:

Standard liquid (water), test liquid etc.

PRINCIPLE: (POISEUILLE'S PRINCIPLE)

If a liquid flows with in a uniform velocity at a rate of 'V' in 't' seconds through a capillary tube of radius 'r' and length 1cm under a driving pressure 'p' dynes/ cm^2. Then,The co-efficient of viscosity is given as =

$$\boxed{\eta = \pi\ r^4\ t\ \triangle P/\ 8VL}$$

$\eta \Rightarrow$ Viscosity of liquid in poise
$\triangle P \Rightarrow$ pressure head i.e. dynes/ cm^2.
$r \Rightarrow$ radius of inner layer of capillary tube
$L \Rightarrow$ length of capillary tube
$V \Rightarrow$ volume of capillary tube
$t \Rightarrow$ flow time in seconds

The poiseulles law is applicable only to linear flow or stream line flow. For a given Oswald's viscometer the length, radius and volume of liquids are constants and at end are combined to a single constant. The above equation can be written as

$\eta = k\,t\,\Delta P$ in this equation ΔP depends on

I. Density of liquid to be measured
II. Acceleration due to gravity
III. the difference due to gravity is constant Then

The viscosity of liquid may be expressed as
$\eta_1 = kt_1\,\rho_1$ (viscosity Standard liquid (water))
$\eta_2 = kt_2\,\rho_2$(viscosity of test liquid)
Relative viscosity $\Rightarrow$ $\eta_1/\eta_2 = t_1\,\rho_1/\,t_2\,\rho_2$

Units: (CGS) $\Rightarrow$ dynes-sec/cm^2 or centi poise (CPS)

PROCEDURE:

Clean thoroughly and dry the Oswald's viscometer, a definite volume of standard liquid is allow to flow into 'A ' arm such that it raises above the values X and Y. The same procedure is repeated with the test liquid and note the time by stop clock

CALCULATIONS:

S. No	Standard liquid (t_1)	Test liquid (t_2)
TRIAL-I		
TRIAL-II		
TRIAL-III		

Weight of empty density bottle (W_1) = __________ gm

Weight of empty bottle + water (W_2) = __________ gm

Weight of empty bottle +liquid (W_3) = __________ gm

Density of water ($\rho_{1)}$ = ________

Density of liquid (ρ_2) = ________

Viscosity Standard liquid (η_1) at 25^oC= 1.0019cps

$$\eta_2 = \frac{t_2 \rho_2}{t_1 \rho_1} \times \eta_1$$

RESULT:

Absolute viscosity of a given liquid (η_2) = _________ cps

VIVA QUESTIONS:

1) Define Viscosity?
2) What is the need of Viscosity?
3) What is the formula for Viscosity?
4) What are the units of Viscosity?
5) What is the effect of Temperature on Viscosity?

EXPERIMENT-X

DETERMINATION OF RATE CONSTANT OF ESTER CATALYSED BY AN ACID

AIM:

To determine the rate constant of hydrolysis of an ester such as methyl acetate catalyzed by an acid (1M HCl)

APPARATUS:

Burette, pipette, Conical flask, Beakers, Burette stand,water bath, stop watch etc.

CHEMICALS REQUIRED:

Methyl acetate, 1M HCl,0.5M NaOH,phenolphthalein indicator.

PRINCIPLE:

$$CH_3COOCH_3+H_2O \xrightarrow{HCl} CH_3COOH+CH_3OH$$

FORMULA:

$$K=2.303/t \log V_{\dot{\alpha}}-V_0 /V_{\dot{\alpha}}-V_t$$

Ester gets hydrollysed in aqueous medium.the reaction is very slow and can be enhanced in presence of strong acid like hcl.the reaction can be expressed by the above equation.the reaction can be followed by volumetric estimation of acetic acid formed in the course of reaction by strong base.the reaction is first order with respect to methyl acetate.since the concentration of water is taken in large excess.hence the reaction is a pseudo first order reaction.

Where

K= first order rate constant

V_0= Volume of NaOH req.by the Rxn.Mixture initially

V_t= Volume of NaOH req.by the Rxn.Mixture at the particular time interval

$V_{\dot{\alpha}=}$ Infinite Volume of NaOH

PROCEDURE:

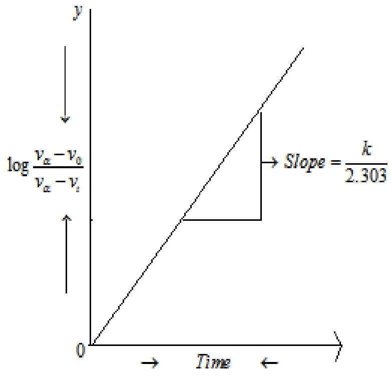
Take 100ml of 1M HCl in a clean reagent bottle and place it in a water bath to attain room temp.fill the burette with 0.5M NaOH soln. Now add 10ml of pure methyl acetate to the reagent bottele containing hcl sol.and shake the sol gently.Immediately noe the time and pipette out 10 ml of the reaction mixture in to a clen conical flask containing 50ml of ice cold water(see that there remains pieces of ice till the end of the titration).in order to quench the reaction, now add 2-3 drops of phenolphthalein indicator and titrate against 0.5M NaOH sol.until the pale pink color just persist(appears). Repeat the titration with 10ml of reaction mixture for every 10 minutes of the interval up to 60 minutes and tabulate the results.

Heat the remaining reaction mixture in a water bath at 60^0c for 20min.cool it to room temperature and pipette out 10ml of reaction mixturte in to a clean conical flask containing 50ml of distilled water and add 2-3 drops of phenolphthalein indicator. Titrate this against 0.5M NaOH and note the titer value as $V_{\acute{\alpha}}$.

GRAPH:

Plot graph by taking $\log V_{\acute{\alpha}}-V_0 / V_{\acute{\alpha}}-V_t$ on Y-axis,time on X-axis A straight line passing through origin is obtained.And its slope = K=2.303
Therefore K = slope = X = 2.303

Units: min-^{1}



CALCULATIONS:

TABLE:

S.NO	Time in mins	Vol. of NaOH	$V_{ά}-V_{t}$	$V_{ά}-V_{0}/V_{ά}-V_{t}$	$\log V_{ά}-V_{0}/V_{ά}-V_{t}$	$K=2.303/t \log V_{ά}-V_{0}/V_{ά}-V_{t}$
1	0					
2	10					
3	20					
4	30					
5	40					
6	50					
7	60					
8	90					

RESULT:

The rate constant for the hydrolysis of methyl acetate using 1M HCl

(a). Experimentally = min^{-1}

(b). By graph = min^{-1}

VIVA QUESTIONS:

1) Define Rate Constant?
2) Define Hydrolysis?
3) What is the importance of Rate Constant?
4) What is the Formula for First order reaction?
5) What are the units for First order reaction?

EXPERIMENT-XI

VERIFICATION OF FREUNDLICH ADSORPTION ISOTHERM- ADSORPTION OF ACETIC ACID ON CHARCOAL

AIM:

Determination of adsorption of acetic acid from solution on activated charcoal and to examine the validity of Freundlich isotherm

APPARATUS:

Reagent bottles,Burette ,Burette stand,Waterbath,Conicalflask etc.

CHEMICALS REQUIRED:

Acetic acid,Charcoal,Phenophthalein indicator,Distilled water

PREPARATION OF 0.25M acetic acid:

Weigh out exactly 3 gms of acetic acid into a 1000 ml standard flask and make up the solution to the mark with distilled water after dissolving the salt in little distilled water.shake the flask well for uniform concentration.

PREPARATION OF 0.1M NaOH solution:

Dissolve 4 gms of NaOH in 1000 ml of water and stir the solution well for uniform concentration.

PHENOPHTHALEIN INDICATOR:

Dissolve 1 gm of Phenophthalein indicator Iin 100 ml of ethanol.

PROCEDURE:

Take well cleaned and dried 6 stoppered reagent bottles and label them.With the help of two burettes transfer the acetic acid and distilled water into these six bottles as shown below.Mix the solution well and keep all the six bottles in a water bath for sometime to acquire the temperature of the water bath.Weigh exactly 2gms of activated charcoal on six glazed papers and add the same to each bottle.Shake the bottle well and keep in the water bath for1/2 an hour time While shaking the bottles from time to time.Filter the contents of all the six bottles into six different labeled,dried conical flasks.Titrate the six samples with M/10 naoh taken in the burette using phenolphthalein indicator till pale pink coloured end point is obtained.Let the titre value be V3,V4,V5,V6,V7,V8 respectively for bottle numbers 1,2,3,4,5,6.Take 20ml of the stock solution into a clean 250ml conical flask,add 2 drops of phenolphthalein indicator and titrate against 0.1M NaOH solution till pale pink colour is obtained as end point.Repeat the titration to get concurrent values.Let the titre value be x ml.

Reagent bottle no.	1	2	3	4	5	6
Acetic acid(0.25M)(ml)	50	40	30	20	10	0
Distilled water(ml)	50	60	70	80	90	100

CALCULATIONS:

Molarity of acetic acid solution prepared(M_1)= wt of acetic acid.10/126

Molarity of NaOH solution (M_2)=$V_1M_1/n_1=V_2M_2/n_2$

V_1= volume of acetic acid (20ml)

M_1= molarity of acetic acid(calculated in the previous step)

n=number of moles of acetic acid reacted=1

V_2=volume of NaOH

n=number of moles of NaOH=2

$M_2 = V_1M_1/n_1 \text{ x } n_2/V_2 = \frac{20 \text{ x } M_1 \text{ x } 2}{x}$

The initial concentration of the acetic acid solutions in 1 to 6 conical flasks.

Reagent bottle no.	Initial concentration(C)
1	$\frac{50 \text{ x } M_2}{100} = a$
2	$\frac{40 \text{ x } M_2}{100} = b$
3	$\frac{30 \text{ x } M_2}{100} = c$
4	$\frac{20 \text{ x } M_2}{100} = d$
5	$\frac{10 \text{ x } M_2}{100} = e$
6	------------------------ zero

Concentration of the acetic acid solutions after adsorption

Bottle no.1 = $M_{\text{(acetic acid)}} = \frac{2 \text{ x } M_{\text{NaOH}} \text{ x } V_3}{50} = P$

Bottle no.2 = $M_{\text{(acetic acid)}} = \frac{2 \text{ x } M_{\text{NaOH}} \text{ x } V_4}{40} = q$

Bottle no.3 = $M_{(\text{acetic acid})} = \frac{2 \times M_{NaOH} \times V_5}{30} = r$

Bottle no.4 = $M_{(\text{acetic acid})} = \frac{2 \times M_{NaOH} \times V_6}{20} = s$

Bottle no.5 = $M_{(\text{acetic acid})} = \frac{2 \times M_{NaOH} \times V_7}{10} = t$

Bottle no.6 = zero

Amount of acetic acid adsorbed $x = \frac{(C_1 - C_2) \times 100}{1000} = \frac{C_1 - C_2}{10}$

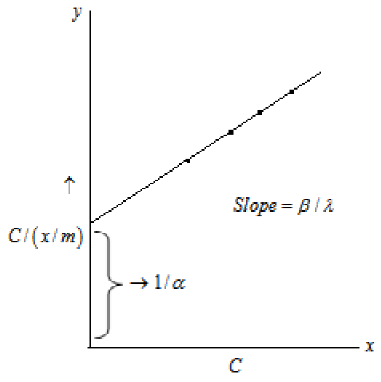
Where, C_1 = initial concentration C_2 = conc.after adsorption

Reagent bottle no.	Initial conc C_1	Conc.of acetic acid after adsorption	Amount of acetic acid adsorbed $(C_1-C_2)/10 = x$
1	a	p	$a - p/10$
2	b	q	$b - q/10$
3	c	r	$c - r/10$
4	d	s	$d - s/10$
5	e	t	$e - t/10$

Amount of the adsorbant (acetic acid) m = 2 gms

Initial concentration of six bottles © = a,b,c,d,e,f respectively.

Plot a graph taking c/(x/m) on ordinate and c on abscissa.



The straight line will prove the validity of langmueir isotherm.

RESULT:

Amount of acetic acid adsorbed by charcoal =

VIVA QUESTIONS:

1) Define Adsorption Isotherm?
2) Define Adsorption?
3) What is the P^H range of Phenapthelin indicator?
4) What is the indicator used in this experiment?
5) What is the colour change of indicator used in this experiment?

EXPERIMENT-XII

THIN LAYER CHROMATOGRAPHY CALCULATION OF R_f VALUES OF ORTHO AND PARA-NITROPHENOLS

AIM:

Calculation of R_f values of ortho and para-nitrophenols by using Thin layer chromatography

APPARATUS:

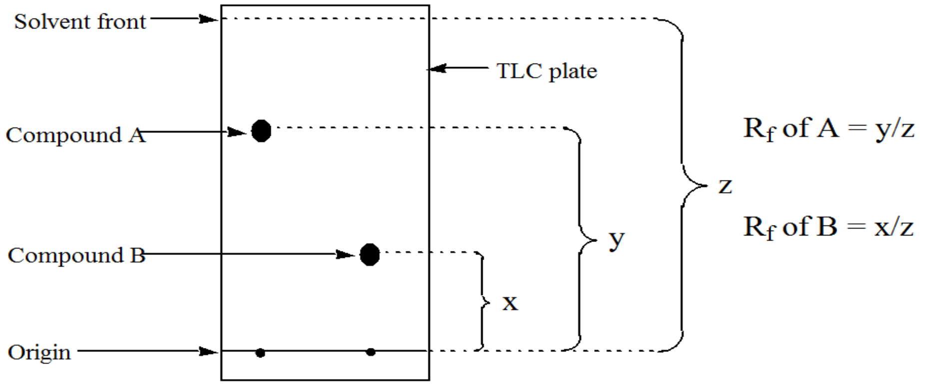
UV chamber,TLC plates

CHEMICALS REQUIRED:

Ortho and Para-Nitro phenol,silica gel.

PROCEDURE:

1. The thin layer chromatography(TLC) plates were prepared on microscopic slides,which were suspended in a slurry of silica gel G.
2. The slurry of silica gel G was prepared by adding 40ml water to 60 gms of dry gel.
3. The mixture was agitated continuously with an electrical shaker to maintain uniformity of the slurry.
4. All slides were dipped for 30 seconds and no binder is added.the slides were removed after 30 seconds and dried.
5. Draw a pencil line above the bottom of TLC plate and mark spots for each p-nitrophenol and o-nitrophenol,equally spaced along line.
6. Use a capillary tube and add a tiny drop of each solution to a different spot and allow the plate to dry.
7. Add the solvent toluene to a chamber or a beaker with a tight lid so that the solvent is not more than 1 cm depth.
8. Place the tlc plate in the chamber.Make sure that the level of the solvent is below the pencil line.
9. Keep the lid when the solvent level reaches about 1 cm from the top of the plate, remove the plate and mark the solvent level with a pencil.
10. Allow the plate to dry in a fume cupboard.place the plate under a U.V lamp in order to see the spots.Draw around the spots lightly in pencil asshown below.



Thin layer chromatogram

CALCULATIONS:

Calculate the rf value of the samples.

$$R_f \text{ value} = \frac{\text{distance moved by the sample}}{\text{distance moved by the solvent}}$$

R_f value of o-nitrophenol = y/x

R_f value of P-nitrophenol = y^1/x

RESULT:

R_f value of o-nitrophenol and P-nitrophenol =

VIVA QUESTIONS:

1) Define R_f value?
2) What is the formula for o-para phenol?
3) What is the formula for o-nitro phenol?
4) What is the indicator used in this experiment?
5) What is the colour change of indicator used in this experiment?