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MINING ENGINEERING
LAB MANUAL

CHEMISTRY
(B.TECH)
SEMESTER II

LIST OF EXPERIMENTS

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1.	Know your lab
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3.	Determination of chloride content of water sample by argentometric method
4.	Determination of DO content of water sample by Winkler's methods.
5.	Determination of acidity of water sample
6.	To draw pH-titration curve of strong acid vs strong base
pH meter:	
7.	Determination of pH of a given sample using pH meter:
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8.	Preparation of N/10 solution
9.	Synthesis of Aspirin
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GENERAL GUIDELINES AND SAFETY INSTRUCTIONS

1. Sign in the log register as soon as you enter the lab and strictly observe your lab timings.
2. Strictly follow the written and verbal instructions given by the teacher / Lab Instructor. If you do not understand the instructions, the handouts and the procedures, ask the instructor or teacher.
3. **Never work alone!** You should be accompanied by your laboratory partner and / or the instructors / teaching assistants all the time.
4. It is mandatory to come to lab in uniform and wear your ID cards.
5. Do not wear loose-fitting clothing or jewels in the lab. Rings and necklaces are usual excellent conductors of electricity.
6. Mobile phones should be switched off in the lab. Keep bags in the bag rack.
7. Keep the labs clean at all times, no food and drinks allowed inside the lab.
8. Intentional misconduct will lead to expulsion from the lab.
9. Do not handle any equipment without reading the safety instructions. Read the handout and procedures in the Lab Manual before starting the experiments.
10. Do your wiring, setup, and a careful circuit checkout before applying power. Do not make circuit changes or perform any wiring when power is on.
11. Avoid contact with energized electrical circuits.
12. Do not insert connectors forcefully into the sockets.
13. **NEVER** try to experiment with the power from the wall plug.
14. Immediately report dangerous or exceptional conditions to the Lab instructor /teacher: Equipment that is not working as expected, wires or connectors are broken, the equipment that smells or “smokes”. If you are not sure what the problem is or what's going on, switch off the Emergency shutdown.

15. Never use damaged instruments, wires or connectors. Hand over these parts to the Lab instructor/Teacher.
16. Be sure of location of fire extinguishers and first aid kits in the laboratory.
17. After completion of Experiment, return the bread board, trainer kits, wires, CRO probes and other components to lab staff. Do not take any item from the lab without permission.
18. Observation copy and lab record should be carried to each lab. Readings of current lab experiment are to be entered in Observation copy and previous lab experiment should be written in Lab record copy. Both the copies should be corrected by the faculty in each lab.

EXPERIMENT NO. 1

Know your Lab

Laboratory Apparatus

Laboratory equipment refers to the many devices of equipment used in a laboratory to carry out specific tasks. These tools are meant for use by scientists, students, professors, and medical professionals. Some scientific lab equipment is used for weighing materials, mixing and creating solutions, and cleaning containers.

Microscope:

A microscope is a common laboratory instrument used to observe items that are too small to see with the human eye. A light microscope examines a small object by using lights and a set of magnifying lenses. An electron microscope uses electrons to magnify an image.

Test Tube:

A test tube is a lab vessel often used to carry and mix liquid chemicals. A test tube is shaped like a finger and has one open end. There are many various sizes of test tubes, but the common size is 18 x 150 mm.

Watch Glass:

A watch glass is a common kind of chemical lab tool. It is a concave piece of glass that's often used to store solids, evaporate liquids, and heat tiny amounts of a substance.

Crucible:

The crucible is a tiny container generally constructed of porcelain. Some laboratory glassware is unsuitable for heating because it may get damaged or break. A crucible is a tiny container generally constructed of porcelain. It also includes a cover that is designed to keep smoke particles inside.

Volumetric Flasks:

Volumetric flasks are another popular part of chemistry laboratory equipment. It is a form of glassware calibrated to retain certain amounts of liquid at specific temperatures. It is used in chemistry to make standard solutions and accurate dilutions. This flask comes in several sizes, and the capacity is generally specified. Depending on how many solutions you want to make, you may use a 50ml, 125ml, 250ml, 500ml, or 1000 ml volumetric flask.

Beakers:

Similar to test tubes, beakers are used to heat, mix, and store different types of materials. Beakers are cylindrical containers without rounded bottoms and have a spout and a flat base. They are also available in several sizes.

Bunsen burner:

This device is used for sterilizing and heating things. Natural gas or liquefied petroleum gas, such as methane, could be utilized.

Spatula:

A laboratory spatula is similar to a kitchen spatula, except it is considerably smaller. Spatulas are tiny, hand-held instruments for scooping and transferring solids. They can also be used to apply paste-like treatments. Most spatulas are used with various chemicals, typically resistant to heat and acid.

Magnifying Glass:

This specific piece of lab equipment creates a magnified image of an object. It is a convex lens covered with a handle-equipped frame.

Spring Balance:

A spring balance is also known as a newton meter. The tension of a spring on the scale is used to calculate the object's weight. On one side, there is a spring, and on the other, there is a hook.

Dropper:

A dropper is also known as a Pasteur pipette. It is a small glass or plastic pipe with a rubber tip on one end. Its purpose is to give little volumes of liquids one drop at a time.

Measuring Cylinder:

This typical laboratory instrument is used to determine the volume of a liquid. It is calibrated, with each marker indicating the quantity of chemical used. This glassware is cylindrical and narrow, as the name indicates.

Thermometer:

We all have used thermometers at home, so we are all familiar with them. Laboratory thermometers are almost identical in that they measure the temperature of substances (instead of body temperature) and have a high level of accuracy.

Burette:

A basic piece of chemical laboratory equipment used to dispense volumes of material. It is often used in titrations. The stopcock is located at the bottom of the long-graded tube. The burettes are 50ml, 25ml, and 10ml.

Balance:

Because certain experiments demand correct quantities of ingredients, solids are often weighed before use. A balance is a device that is used to consider materials. The most popular balance types are analytical and top loading balances.

Funnels:

Funnels are another essential kind of device. They are used to transfer chemicals into small-mouthed receptacles. Filter, thistle, and falling funnels are some of the various types, and each has a specific purpose. Buchner and Hirsch's funnels are excellent examples of organic chemistry laboratory gear.

Wash Bottle:

A wash bottle is a squeeze container used to clean and rinse glassware. The majority of wash bottles are plastic. Depending on the task, you may fill it with ethanol or deionized water.

Tongs:

You are often exposed to chemicals, heat, and other potentially harmful substances when working in a laboratory. Tongs are used to grab dangerous things and handle hot containers. Each sort of tong is intended for a particular purpose. Beaker, utility, and crucible tongs are common examples.

Ammeter:

This is a piece of equipment that is present in every physics lab. An ammeter is a tool to gauge how much electricity moves across a circuit.

Brushes for Test Tubes:

Without a cleaning tool, our laboratory equipment list would be incomplete. Test tubes, flasks, and beakers are cleaned using cleaning brushes. After usage, all equipment should be cleaned and safely stored.

EXPERIMENT NO. 2

Aim: Determine the Strength of NaOH Solution (Standard Hydrochloric acid Solution Supplied).

Theory of experiment: Titration is a technique used to determine the concentration of a given substance. A solution with a known volume and concentration (the titrant) is added to another solution with a known volume, but an unknown concentration (the titrate). An indicator added to the solution turns pink when all the unknown substance has been neutralized. By knowing the volume and concentration of the titrant, you can determine the concentration of the titrate.

Acid-Base Titration: The most common type of titration involves an acid and a base. When an acid and a base react they neutralize each other, producing salt and water. For example, one mole of hydrochloric acid will neutralize one mole of sodium hydroxide, producing salt and water.

HCl + NaOH → NaCl + H2O

(Acid) (Base) (Salt) (Water)

Molarity: The concentration of a solution is the amount of solute that is dissolved in a particular amount of solution. It can be expressed by percent volume, percent mass, or molarity. Molarity is the number of moles of solute dissolved per liter of solution.

M = moles of solute/ liters of solution

-3

The concentration of hydrochloric acid was given as 0.09756mol. dm^{3}-3

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Apparatus required:

1. Beaker
2. Conical flask
3. Burette
4. Pipette
5. Measuring cylinder
6. Dropper
7. Iron stand
8. Funnel

Chemical required

1. Hydrochloric acid
2. Sodium hydroxide
3. Phenolphthalein

Procedure:

1.10 ml of the HCl solution is pipetted out in a conical flask

2. 2-3 drops of phenolphthalein indicator is added to it.

3. Then this solution is titrated with standardized NaOH solution till the solution turns pink.

4. The titration is repeated to get a set of concordant readings. You will do at least three titrations.

5. Obtain about 100 mL of the sodium hydroxide solution in a clean beaker. This should be enough for the initial cleaning of your burette and for your first 3 trials.

6. Clean your burette: Add about 5 mL of the base solution from the beaker to the burette (use a funnel to pour). Move the funnel around while adding to ensure the sides of the burette are coated with base. Alternatively, you can remove the burette with the 5 mL of titrant from the burette stand and carefully tilt and rotate to coat all interior surfaces with the titrant. Drain the solution through the stopcock into a waste beaker. Repeat this rinse with a second 5 mL portion of base.

7. Pour more of the sodium hydroxide solution into the burette until it is near the 0.00 mL mark. Open the stopcock to allow several drops to rinse through the tip of the burette. This should eliminate any air bubbles in the burette tip. Record your initial burette reading on the data sheet for trial 1 (the volume does not need to be exactly 0.00 mL).

8. Place the flask in which you put HCl under the burette and start adding the base solution to the Erlenmeyer flask. Have one lab partner swirl the flask while the other controls the stopcock. When pink starts to develop, add the solution more slowly. At this point you should add one drop at a time followed by swirling until a very light pink color persists for at least 30 seconds. Remember, the lighter the pink the better!!!

9. Record the final reading of the burette. Wash the contents of the flask down the drain with water.

10. Refill the burette with more sodium hydroxide solution if necessary. Record the new volume under trial 2 on the data sheet. Pipette another sample of acid and add the phenolphthalein as before and titrate as before.

4. Conduct additional titrations until three of them differ by no more than 1.0%.

Observations/Data

No of observation	Volume of NaOH solution in mL	Burette reading Initial Final	Different	Constant	Indicator used
Trial-1					

Trial-2					
Trial-3					

Calculations:

V_1=Volume of HCl

$N_1 = N/10$

V_2 = Concurrent reading let X

N_2 = To be calculated

We know,

$N_1V_1= N_2V_2$

$N2=N_1V_1/V_2$

Result:

Strength of NaOH =_______g/mol

Precaution:

- Rinse burettes and pipettes with distilled water or the solution you'll be using
- Use calibrated glassware for accurate measurements
- Avoid using broken glassware
- Stir the solution gently and consistently
- Read the burette or pipette at eye level
- Control the rate at which you add the titrant
- Use a clean, dry titration flask
- Check the color of the solution against a white background
- Don't let the titrant reach 0 mL
- Rinse the area with water if any solution gets on your skin

EXPERIMENT NO. 3

Aim: Determination of chloride content of water sample by argentometric method

Theory of experiment: This method determines the chloride ion concentration of a solution by titration with silver nitrate. As the silver nitrate solution is slowly added, a precipitate of silver chloride forms.

$Ag^{+}_{(aq)} + Cl^{-}_{(aq)} \rightarrow AgCl(s) \downarrow$(White)

The end point of the titration occurs when all the chloride ions are precipitated. Then additional silver ions react with the chromate ions of the indicator, potassium chromate, to form a red-brown precipitate of silver chromate.

$2\,Ag^{+}_{(aq)} + CrO_4{}^{2-}{}_{(aq)} \rightarrow Ag_2CrO_4(s) \downarrow$(Brick red)

This method can be used to determine the chloride ion concentration of water samples from many sources such as seawater, stream water, river water and estuary water. Tap water is used as the example here. The pH of the sample solutions should be between 6.5 and 10.

Apparatus required:

1. Beakers,
2. Graduated cylinder,
3. Conical flask,
4. Burette
5. Filter paper
6. Pipette, pipette filler,
7. Volumetric flask,
8. Digital balance,
9. Titration setup (stand, clamp, and funnel) and Stirring Rod.

Chemical required:

1. Silver nitrate solution (0.05 M): Dissolve 8.4935 g of AR grade silver nitrate (Equivalent weight = 169.87 g/mol) in 1000 mL conical flask with distilled water and homogenize.

2. Potassium chromate solution (5% solution): Dissolve 5 g of AR grade potassium chromate in 100 mL of distilled water in a conical flask.

3. Conc. Sulphuric acid

Procedure:

1• Wash the burette with distilled water and rinse with standardized solution of silver nitrate and then fill the burette with the same.

2• Carefully pipette out 10 cm3 of given solution of chloride ions and transfer into a 100 cm3 conical flask. Add 1 cm3 of the potassium chromate indicator solution (it is advisable to use a 1 cm3 pipette for the purpose).

3• Titrate the solution with silver nitrate solution and ensure that the flask is constantly swirled.

4• With the progress of the titration, you would observe that the red colour formed by the addition of each drop of Silver nitrate begins to disappear more slowly; slow down the rate of addition of silver nitrate. The end point is indicated by sudden development of a faint but distinct change in colour to reddish-brown. This does not disappear on swirling the titration flask.

5• Repeat the procedure for at least three times and record your observation in Observation Table. Determination of indicator blank

6• In a clean and dry conical flask take distilled water equal to the volume of titrant + volume of the titrant and add 1 cm3 of the potassium chromate indicator solution with the help of a 1 cm3 pipette,

7• Titrate the solution with silver nitrate solution while constantly stirring the flask and determine the end point as above. (As the volume of silver nitrate used for the indicator blank is going to be 70 very small you are advised to add silver nitrate drop wise right from the beginning of the titration).

8• Note the volume of silver nitrate used for indicator blank and record it after the Observation Table

Observation:

Determination of the amount of chloride ions in the given water sample by Mohr’s method:

For sample

S.No	Volume of sample solution taken(ml)	Burette reading(ml)		Vol. of $AgNO_3$ Used(ml)
		Initial	Final	
1				
2				
3				
4				

The volume of silver nitrate used for complete precipitation of chloride ions in the given solution = A cm3 =….cm3

For Blank Correction

S.No	Volume of sample solution taken(ml)	Burette reading(ml)		Vol. of $AgNO_3$ Used(ml)
		Initial	Final	
1				
2				
3				
4				

The volume of silver nitrate used in the indicator blank titration = B cm3 =….cm3

Calculation:

The net volume of silver nitrate used for complete precipitation of chloride ions

in the given solution = (A−B) cm3 = C cm3 = ….cm3

The molarity of the chloride ions in the given solution can be determined as follows.

The reaction involved: $AgNO3 + NaCl \rightarrow AgCl(s) \downarrow + NaNO3$

Molarity equation: $M_{AgNO3}V_{AgNO3} = M_{NaCl} V_{NaCl}$

$M_{NaCl} = M_{AgNO3} . V_{AgNO3} / V_{NaCl}$

Result:

Substituting the values,

The molarity of chloride ions (as NaCl) =

The molarity of given chloride ions is = ……….M

Substituting the values, of the molarity and the volume of silver nitrate used, the molarity of chloride ions is found to be: = ...M

Precautions:

- Rinse burettes and pipettes with distilled water or the solution you'll be using
- Use calibrated glassware for accurate measurements
- Avoid using broken glassware
- Stir the solution gently and consistently
- Read the burette or pipette at eye level
- Control the rate at which you add the titrant
- Use a clean, dry titration flask
- Check the color of the solution against a white background
- Don't let the titrant reach 0 mL

EXPERIMENT NO. 4

Aim: Determination of dissolved oxygen content to water sample by Winkler's method

Theory of experiment- Oxygen is dissolved in water to the extent of 7 – 9 mgs/lit at a temperature range of 25□ - 35□ C. The estimation of dissolved oxygen in water is useful in studying corrosion effects of boiler feed water and in studying water pollution. The amount of dissolved oxygen in water is estimated using Winkler's reagent (Potassium bromide + Potassium bromate). Water sample is collected carefully avoiding aeration/deaeration in ground stoppered flask. Initially manganous sulphate and alkali-iodide reagents are added and the reactions occur as follows

$$Mn^{2+} + 2OH^{-} \rightarrow Mn\,(OH)_2\text{(white) ppt}$$

$$Mn(OH)_2 + \tfrac{1}{2}\,O_2 \rightarrow MnO(OH)_2\text{ (Yellow brown)ppt}$$

The precipitate dissolves in concentrated sulphuric acid liberating iodine and the liberated iodine is titrated against $Na_2S_2O_3$.

$MnO(OH)_2 + 2H_2SO_4 \rightarrow Mn\,(SO_4)_2 + 3H_2O$
$Mn\,(SO_4)_2 + 2KI \rightarrow MnSO_4 + K_2SO_4 + I_2$
$Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$

Chemical required:

1. Sodium thiosulphate solution
2. Std.Potassium dichromate solution
3. Dil H_2SO_4.
4. 5% KI
5. Starch indicator
6. Manganese sulphate
7. Alkali iodide mixture
8Con. H_2SO_4

Apparatus Required:

1. Burette
2. Pipette
3. Iodine flask.
4. Iron stand

Procedure:

Titration – I

Standardization of Sodium Thiosulphate

The burette is washed and rinsed with sodium thiosulphate solution. Then the burette is filled with the given sodium thiosulphate solution up to zero mark. 20 ml of potassium dichromate solution is pipetted out into a clean conical flask. To this, 5 ml sulphuric acid and 15 ml of 5% potassium iodide solution are added. This is titrated against sodium thiosulphate solution. When the solution becomes straw yellow colour, starch indicator is added and then titration is continued. The end point is disappearance of blue colour and appearance of light green colour. The titration is repeated to get concordant value.

Titration – II

Determination of Dissolved Oxygen

100-150 ml of the water sample is taken in the iodine flask, 2ml of manganese sulphate and 2 ml of alkali-iodide mixture are added. The stopper is replaced and the flak is inverted and shake several times for the rough mixing of reagents. The flask is left aside for some time. When half of the precipitate settles down, the stopper is removed and 2 ml of concentrated sulphuric acid is added.

The stopper is replaced and the flask is inverted several times for complete dissolution of the precipitate to get a clear yellow solution. 20 ml of this solution is pipetted out in a clean conical flak and titrated against standardized sodium thiosulphate solution. When the solution becomes light yellow, starch indicator is added. The titration is continued until the blue colour disappears. From the titre value the strength of dissolved oxygen is calculated and hence the amount of dissolve oxygen in the water sample is calculated.

Observation:

Titration – I

Std. Potassium dichromate Vs Sodium thiosulphate

S.No	**Volume of Potassium dichromate (mL)**	**Burette reading (mL)**		**Concordant volume of Sodium thiosulphate (mL)**	**Indicator**
		Initial	**Final**		
1	20				STARCH
2	20				
3	20				

Calculation:

Volume of Potassium dichromate V_1= 20mL

Strength of Potassium dichromate N_1=…............ N

Volume of Sodium thiosulphate V_2= .... mL

Strength of Sodium thiosulphate N_2= …………? ... N

According to the law of volumetric analysis
$V_1 N_1 = V_2 N_2$
$N_2 = V_1 \text{ x } N_1 / V_2$
= ……20……….. X …………….. / …………….
Strength of sodium thiosulphate N_2 =…................. N

Determination of dissolved oxygen
Titration – II
Water sample Vs Sodium thiosulphate

S.N	**Volume of water sample (mL)**	**Burette reading (mL)**		**Concordant volume of Sodium thiosulphate (mL)**	**Indicator**
		Initial	**Final**		
1	20				Starch
2	20				
3	20				

Calculation:

Volume of Sodium thiosulphate V_1 =................... mL

Strength of Sodium thiosulphate N_1 =…................. N

Volume of water sample V_2 = 20 mL Strength of water sample (or)
Strength of dissolved oxygen in water N_2 = …………? .... N

According to the law of volumetric analysis
$V_1 N_1 = V_2 N_2$

$N_2 = V_1 \text{ x } N_1 / V_2$
= …………….. x …………….. / 20

Strength of sodium thiosulphate N_2 =…................. N

Amount of dissolved oxygen in one liter of tap water = Normality x Eq.weight of oxygen x 1000 mgs
= .... N x 8 x 1000

Amount of dissolved oxygen in one liter of tap water = Normality x Eq.weight of oxygen x 1000 mgs

= .... N x 8 x 1000

Amount of dissolved oxygen in one liter of tap water =..........mgs/lit.

Result:

Amount of dissolved oxygen in sample water = ……mgs/lit.

Precautions:

- Rinse burettes and pipettes with distilled water or the solution you'll be using
- Use calibrated glassware for accurate measurements
- Avoid using broken glassware
- Stir the solution gently and consistently
- Read the burette or pipette at eye level
- Control the rate at which you add the titrant
- Use a clean, dry titration flask
- Check the color of the solution against a white background
- Don't let the titrant reach 0 mL

EXPERIMENT NO. 5

Aim: To determine the acidity of sample water

Theory of experiment:

Hydrogen ions present in a sample as a result of dissociation or hydrolysis of solutes reacts with additions of standard alkali (NaOH). Acidity thus depends on end point of the indicator used.

Mineral acidity: It is measured by titration to a pH of about 3.5, the methyl orange end point (also known as methyl orange acidity)

Total acidity: The colour change of phenolphthalein indicator is close to pH 8.3 at 25ºC corresponds to stoichiometric neutralization of carbonic acid to bicarbonate.

Apparatus required:

1. Beaker
2. Conical flask
3. Burette
4. Pipette
5. Measuring cylinder
6. Dropper
7. Iron stand
8. Funnel

Chemical required

1. Sodium hydroxide
2. Phenolphthalein
3. Methyl Orange
4. Ethyl alcohol
5. Distilled Water

Procedure:

1. Take 50 ml sample in a conical flask and add 2-3 drops of methyl orange indicator solution.

2. Fill the burette with 0.02 N NaOH solution and titrate till the colour of solution just changes to faint orange colour, indicating the end point. Record the volume of titrant consumed as V1 in ml.

3. For phenolphthalein acidity test, add 2-3 drops of phenolphthalein indicator solution to water sample from step 2 and continue the titration till the faint pink colour develops in the solution (i.e., the end point of titration). Record the volume of titration consumed as V2 (mL)

4. Repeat the steps 1,2and 3 for three times

Observation:

Table -1

Mineral Acidity:

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of NaOH (mL)	Concordant Reading
		Initial	Final		
1					
2					
3					

Burette Solution: Sodium Hydroxide

Pipette Solution: Sample

Indicator: Methyl Orange

End Point: Faint of Orange Color

Table -2

Total Acidity:

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of NaOH (mL)	Concordant Reading
		Initial	Final		
1					
2					
3					

Burette Solution: Sodium Hydroxide

Pipette Solution: sample solution

Indicator: Phenolphthalein

End Point: Faint Pink

Calculation:

Volume of NaOH for Mineral Acidity (V_1) = V_1 mL
Volume of NaOH for Total Acidity (V_2) = V_2 mL Normality of Sulphuric Acid= 0.02 N (let)
Volume of Sample= 100.0 mL
Equivalent weight of $CaCO_3$= 50

$$\text{Mineral Acidity} = \frac{\text{Volume of NaOH } (V_1) * N * 50 * 1000}{\text{Volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by

1,000 mL/L

Mineral Acidity as $CaCO_3$ equivalent (mg/L) =. . mg/L

$$\text{Total Acidity} = \frac{\text{Volume of NaOH } (V_2) * N * 50 * 1000}{\text{Volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L

Result:

Mineral Acidity as $CaCO_3$ equivalent (mg/L) =... mg/L

Total Acidity as $CaCO_3$ equivalent (mg/L) = ….mg/L

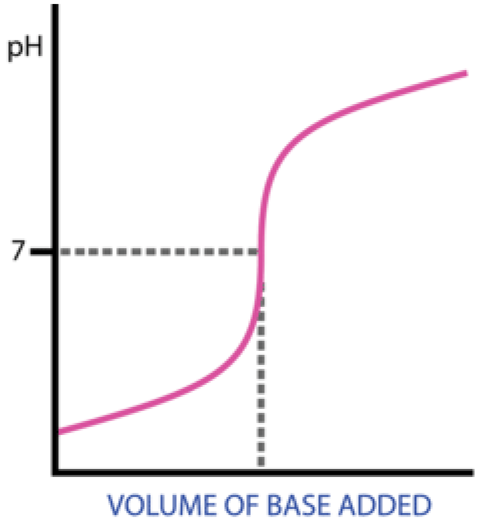
Precaution:

- Rinse burettes and pipettes with distilled water or the solution you'll be using
- Use calibrated glassware for accurate measurements
- Avoid using broken glassware
- Stir the solution gently and consistently
- Read the burette or pipette at eye level
- Control the rate at which you add the titrant
- Use a clean, dry titration flask
- Check the color of the solution against a white background
- Don't let the titrant reach 0 mL
- Rinse the area with water if any solution gets on your skin

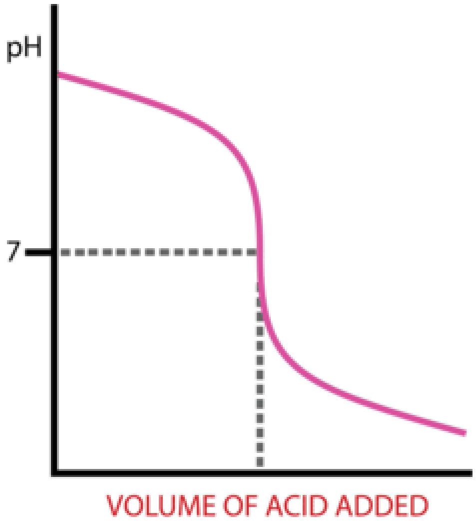
EXPERIMENT NO – 6

Aim: To draw pH-titration curve of strong acid vs strong base

Theory of experiment: A **titration curve** is a graphical representation of the pH of a solution during a titration. When an alkali is added to an acid solution, the pH of the solution increases slowly, but at vicinity of the end point, the rate of change of pH of the solution is very rapid. From the sharp break in the curve, we can find out the end point, from which the strength of HCl can be calculated.



Titration curve of a strong acid (HCl) with a strong base (NaOH)



Titration curve of a strong base (NaOH) with a strong acid (HCl)

Volume of NaOH (ml)

Procedure:

1. Standardize NaOH solution against 0.1 N oxalic acid solution using phenolphthalein as indicator. [Equiv. wt. of oxalic acid: 63]

2. Wash the glass electrode with distilled water.

3. Take 20 ml of HCl solution in a 250 ml beaker. Add sufficient distilled water (100 ml) so that the glass electrode is completely dipped.

4. Note the pH of the pure acid solution.

5. Now add 1 mL of 0.1 N NaOH from the burette in the beaker. Stir the contents well. Note the pH of the solution. Now keep on adding NaOH solution from the burette and note the pH of the solution, up to 9- 10 mL of the NaOH.

6. Near the end point add very small amount of sodium hydroxide, because change in pH will be very much appreciable when the acid is neutralized, further addition of such a small amount as 0.01 ml raises the pH about 9 to 10.

7. Plot titration curve (pH vs V) and differential titration curve **Δ**pH/**Δ**V vs Average volume (V/) and locate the end point to record the volume of the base required for neutralization of the acid of unknown strength taken into a beaker. [**Δ**pH = pH_2-pH_1; **Δ**V = V_2-V_1 & V/ is the average of two consecutive volumes i.e. V/= (V_1+V_2) / 2]

Observations:

Plot a graph between pH and Volume of NaOH added and a differential titration curve **Δ**pH/**Δ**V vs Average volume. From these two graphs, determine the volume of NaOH required for the complete neutralization of HCl.

Vol. of NaOH added (V in ml)	**pH**	**ΔV**	**ΔpH**	**ΔpH/ΔV**	**Average Vol. of NaOH added (V/ in ml)**
0		V_1-V_0	pH_1-pH_0		(V_0 + V_1) / 2
1		V_2-V_1	pH_2-pH_1		(V_1 + V2) / 2
2 contd…		contd…	contd…		contd…
3 contd…		contd…	contd…		contd…

Calculation:

For standardization of NaOH solution:

Volume of oxalic acid taken (V_1) = … ml

Strength of oxalic acid (N_1) = …. N

Volume of NaOH required (V_2) = … ml

Strength of NaOH (N_2) =? (N)

$V_1N_1 = V_2N_2$ or $N_2 = V_1N_1 / V_2$

For calculation of the strength HCl solution:

Volume of NaOH required for complete neutralization of HCl (V_1) = -------- (ml) from graph
Strength of NaOH (N_1) = ------------ N
Volume of HCl (V_2) = ------------- (ml)
Strength of HCl (N_2) =? (N)
Therefore Strength of HCl (N_2) = V_1N_1 / V_2
Amount = Normality x Equivalent Weight (HCl)
Amount of HCl present in the whole of the given solution = [N_2 x (Eq.wt) x 20] /1000 in gm
(Equivalent Weight of HCl = 36.5)

Result:

(i) Strength of the given HCl solution = ---------- N
(ii) The amount of HCl present in the whole of the given solution = --------- gm

Precautions:

- Rinse burettes and pipettes with distilled water or the solution you'll be using
- Use calibrated glassware for accurate measurements
- Avoid using broken glassware
- Stir the solution gently and consistently
- Read the burette or pipette at eye level
- Control the rate at which you add the titrant
- Use a clean, dry titration flask
- Check the color of the solution against a white background
- Don't let the titrant reach 0 ml.
- Rinse the area with water if any solution gets on your skin

EXPERIMENT NO-7

Aim:Determination of pH of a given sample using pH meter

Theory of experiment: The pH electrode used in the pH measurement is a combined glass electrode. It consists of sensing half-cell and reference half-cell, together form an electrode system. The sensing half-cell is a thin pH sensitive semi permeable membrane, separating two solutions, viz., the outer solution, the sample to be analyzed and the internal solution, enclosed inside the glass membrane and has a known pH value. An electrical potential is developed inside and another electrical potential is developed outside, the difference in the potential is measured and is given as the pH of the sample.

Apparatus required:

1. pH meter

2. Standard flasks

3. Magnetic Stirrer

4. Funnel

5. Beaker

6. Wash Bottle

7. Tissue Paper

8. Forceps

Chemicals required

1. Buffers Solutions of pH 4.01, 7.0 and 9.2

2. Potassium Chloride

3. Distilled Water

Procedure:

Three major steps are involved in the experiment. They are

1. Preparation of Reagents

2. Calibrating the Instrument

3. Testing of Sample

Preparation of reagents

Buffer Solution of pH 4.0

1. Take 100 mL standard measuring flask and place a funnel over it.
2. Using the forceps carefully transfer one buffer tablet of pH 4.0 to the funnel.
3. Add little amount of distilled water, crush the tablet and dissolved it.
4. Make up the volume to 100 mL using distilled water.

Buffer Solution of pH 7.0

1. Take 100 mL standard measuring flask and place a funnel over it.
2. Using the forceps carefully transfer one buffer tablet of pH 7.0 to the funnel.
3. Add little amount of distilled water, crush the tablet and dissolved it.
4. Make up the volume to 100 mL using distilled water.

Buffer Solution of pH 9.2

1• Take 100 mL standard measuring flask and place a funnel over it.

2• Using the forceps carefully transfer one Buffer tablet of pH 9.2 to the funnel.

3• Add little amount of distilled water, crush the tablet and dissolved it.

4• Make up the volume to 100 mL using distilled water.

Calibrating the instrument:

Using the buffer solutions calibrate the instrument.

In a 100 mL beaker take pH 9.2 buffer solution and place it in a magnetic stirrer, insert the Teflon coated stirring bar and stir well.

Now place the electrode in the beaker containing the stirred buffer and check for the reading in the pH meter.

If the instrument is not showing pH value of 9.2, using the calibration knob adjust the reading to 9.2.

Take the electrode from the buffer, wash it with distilled water and then wipe gently with soft tissue.

If the instrument is not showing pH value of 7.0, using the calibration knob adjust the reading to 7.0.

In a 100 mL beaker take pH 7.0 buffer solution and place it in a magnetic stirrer, insert the Teflon coated stirring bar and stir well.

Now place the electrode in the beaker containing the stirred buffer and check for the reading in the pH meter.

Testing of sample

In a 100 mL beaker take pH 4.0 buffer solution and place it in a magnetic stirrer, insert the teflon coated stirring bar and stir well.

Now place the electrode in the beaker containing the stirred buffer and check for the reading in the pH meter.

If the instrument is not showing pH value of 4.0, using the calibration knob adjust the reading to 4.0.

Take the electrode from the buffer, wash it with distilled water and then wipe gently with soft tissue. Now the instrument is calibrated.

In a clean dry 100 mL beaker take the water sample and place it in a magnetic stirrer, insert the teflon coated stirring bar and stir well.

Now place the electrode in the beaker containing the water sample and check for the reading in the pH meter. Wait until you get a stable reading.

The pH of the given water sample is 8.84

Take the electrode from the water sample, wash it with distilled water and then wipe gently with soft tissue.

Calculation:

To determine the value of pH of the given water sample the readings obtained are required to be tabulated.

Table

Sample No	Temperature of Sample (ºC)	pH

Result:

For sample 1 the temperature of the measurement is …° C and as obtained the value of the pH is….

For sample 2 the temperature of the measurement is …° C and as obtained the value of the pH is …..

For sample 3 the temperature of the measurement is …° C and as obtained the value of the pH is …

Precaution:

The following precautions should be observed while performing the experiment

.Temperature affects the measurement of pH at two points. The first is caused by the change in electrode output at different temperatures. This interference can be controlled by the instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second is the change of pH inherent in the sample at different temperatures. This type of error is sample dependent and cannot be controlled; hence both the pH and temperature at the time of analysis should be noted.

In general, the glass electrode, is not subject to solution interferences like color, high salinity, colloidal matter, oxidants, and turbidity or reluctant.

Oil and grease, if present in the electrode layer, should be removed by gentle wiping or detergent washing, followed by rinsing with distilled water, because it could impair the electrode response.

Before using, allow the electrode to stand in dilute hydrochloric acid solution for at least 2 hours.

Electrodes used in the pH meter are highly fragile, hence handle it carefully.

EXPERIMENT NO -8

Aim: Preparation of N/10 solution

Theory of experiment: Solutions of accurately known strength are called standard solutions. A standard solution contains a known weight of reagent in a definite volume of solution.
Normal solution: Normal solution is one, which contains one equivalent weight of the reagent in one liter of the solution. Normality is expressed as N.
Equivalent weight of acid= Molecular weight/ Molecular weight
Apparatus required:
Measuring Jar, Conical Flask, Weighing Bottle, Std. Flask, Pipette, Burette, and Electronic Balance.
Preparation of N/10 H_2SO_4
Equivalent weight of H_2SO_4 = 49 g
Specific gravity = 1.84 g/ml
So, volume of 49 g H_2SO_4= 26.6 ml
Concentrated H_2SO_4 (reagent grade) is about 97 per cent pure. Therefore, actual amount of concentrated H_2SO_4 required for 1.0 liter of N/10 H_2SO_4 solution = 100/97 x 26.6 = 27.42 ml
Thus, for 1.0 liter of N/10 H_2SO_4solution, 2.74 ml of concentrated H_2SO_4 is required.
Procedure
1. Take 2.74 ml sulphuric acid in a beaker half-filled with distilled water.
2. Transfer the contents and washings to a volumetric flask (1 liter) and make volume up to the mark.
3. Shake well and titrate this solution with 10 ml of 0.1 N Na_2CO_3 using mixed / methyl orange as an indicator.
4. Repeat the titration to get at least three concordant readings. **Standardization**

Suppose 10 ml of 0.1 N Na2CO3 = 9.5 ml of H2SO4
$V_1N_1 = V_2 N_2$
10 x 0.1 N = 9.5 x N = 0.10526
To prepare 1 liter N/10 H_2SO_4, the volume of 0.10526 N acid required is 1000 x 0.1/0.10526 = 950 ml.
Take 950 ml of 0.10526 N acid and dilute it to one liter. Check it again with N/10 Na_2CO_3 for three times.
It must neutralize equal volume of N/10 Na_2CO_3 solution. Label it as 0.1 N H_2SO_4.
Precautions:
Add $H_2SO_4$with the help of a burette.
Never add water to an acid.

Preparation of N/10 NaOH solution
Molecular weight of NaOH = 40 Acidity (number of replaceable OH group) = 1 Equivalent weight of NaOH = 40
Therefore, 4 g of NaOH dissolved in one liter of solution will give N/10 NaOH solution.
Procedure

1. Weigh quickly 4 g NaOH in a beaker (as it is hygroscopic) and dissolve it in distilled water (preferably CO -free).
2. Transfer the contents and the washings to a volumetric flask (1 liter).
3. Cool and then make volume up to the mark.
4. Shake well and standardize this solution against N/10 oxalic acid using phenolphthalein as an indicator.
5. Label it as 0.1 N NaOH solution.

Preparation of N/10 $KMnO_4$ solution

1. Dissolve 3.2 g $KMnO_4$ in one liter of distilled water.
2. Boil it for 10-15 minutes and then allow to stand for few days and then filter it through glass wool.
3. Take 10 ml of N/10 oxalic acid in a beaker. Add 5 ml dilute sulphuric acid, warm it to 60-70ºC and titrate against $KMnO_4$ from the burette till a light pinkish colour appears.

4. Take three concordant readings.

Suppose 10 ml 0.1 N oxalic acid = 9.75 ml of $KMnO_4$

$V_1N_1 = V_2N_2$

$10 \times 0.1 N = 9.75 \times N_2$

Take 10 ml of N/10 oxalic acid in a beaker. Add 5 ml dilute sulphuric acid, warm it to 60- 70ºC & titrate against $KMnO_4$ from the burette till a light pinkish colour appears. Take three concordant readings.

Suppose 10 ml 0.1 N oxalic acid = 9.75 ml of $KMnO_4$

$V_1N_1 = V_2 N_2$

$10 \times 0.1 N = 9.75 \times N_2$

10 x 0.1 N = ------------- = 0.10256 9.75

To prepare 1000 ml 0.1 N $KMnO_4$

100 x 9.75 x 0.1 --------------------- 10 x 0.1 solution the volume of $KMnO_4$ will be taken. Now take 975 ml of prepared $KMnO_4$ solution and make it 1000 ml by adding distilled water.

Precaution: Ordinary or even pure distilled water contains traces of organic matter which reduces the $KMnO_4$ solutions. That is why the solution is boiled and kept for some time before standardization.

In the absence of sufficient amount of dilute H2SO4 or due to the rapid addition of $KMnO_4$ in titration flask, brown turbidity (manganous oxide) may appear.

Preparation of N/10 Na_2CO_3 solution

Molecular weight of Na_2CO_3=106

Na_2CO_3 = 2HCl= $2NaCl+H_2O+CO_2$

So, acidity of Na_2CO_3 = Equivalent weight of Na_2CO_3 = 53 Therefore, 5.3 g $Na_2CO_3$is required for each liter of solution to make N/10 Na_2CO_3.

Na2CO3 is hygroscopic, therefore, it must be made perfectly anhydrous before it is weighed out.

Procedure

1. Take 6-7 g of Na_2CO_3 (A.R.) in a nickel crucible and heat it in a hot air oven at about 100ºC for few hours so as to drive out any moisture and to convert any moisture and to convert any preformed $NaHCO_3$ to Na_2CO_3.

2. Cool in a desiccator and weigh exactly 5.3 g dried salt and dissolve it in a little quantity of freshly boiled distilled water.

3. Transfer it to one liter measuring flask and make volume up to the mark. Shake well and label it as 0.1 N

Na_2CO_3solution.

EXPERIMENT NO. 9

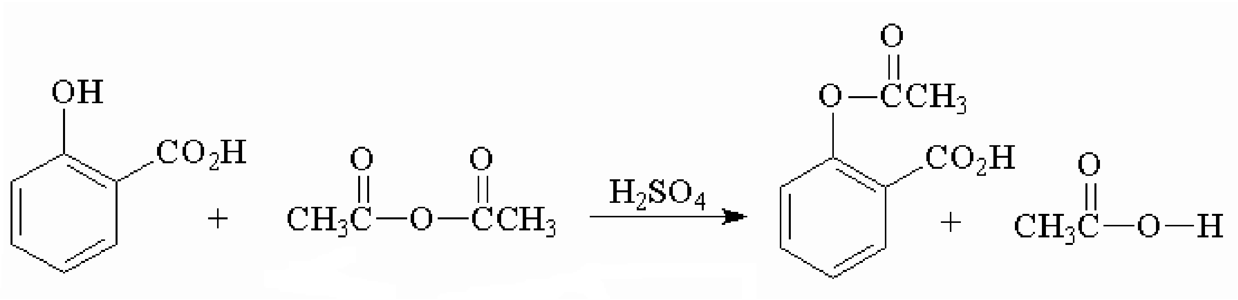
SYNTHESIS OF ASPIRIN

Aim: Synthesis aspirin from salicylic acid

Theory of experiment: Aspirin (acetylsalicylic acid) is synthesized from salicylic acid and acetic anhydride in an esterification reaction catalyzed by sulfuric acid or phosphoric acid producing Aspirin and acetic acid.

The Reaction:

Salicylic acid (C7H6O3) reacts with acetic anhydride (C4H6O3) in the presence of a catalyst (e.g., sulfuric acid, H2SO4) to form acetylsalicylic acid (aspirin, C9H8O4) and acetic acid (C2H4O2).



Salicylic acid   acetic anhydride   Aspirin   acetic acid

Apparatus required:

1.100 mL beaker

2. Filter paper

3.400 mL beaker

4. Spatula

5. Filter flask

6. Iron stand

7.10 mL graduated cylinder

8. Watch glass

9. Wash bottle

10. Buchner funnel

11. Stirring rod

Chemical Required:

1. Salicylic acid

2. Acetic anhydride

3.H_2SO_4 (concentrated) ethanol

Procedure:

1. Obtain approximately 2 g of salicylic acid and determine its mass accurately. Transfer to a 100 mL beaker.

2. Add 5 mL (0.05 mole) of acetic anhydride, followed by 5 drops of conc. H_2SO_4 (*use a dropper,* H_2SO_4 *is highly corrosive*) and swirl the flask gently until the salicylic acid dissolves.

3. Heat the flask gently on the steam bath (70–80°C) for at least 20-30 minutes. Use a stirring rod to get the reactants into solution. Allow the mixture to react.

4. Allow the flask to cool to room temperature. If acetylsalicylic acid does not begin to crystallize out, scratch the walls of the flask with a glass rod. Cool the mixture slightly in an ice bath until crystallization is completed. The product will appear as a solid mass when crystallization is completed.

5. Add 50 mL of water and cool the mixture in an ice bath. Do not add the water until crystal formation is complete.

6. Vacuum filter the product using a Buchner funnel. You can use some of the filtrate to rinse the Erlenmeyer flask if necessary.

7. Rinse the crystals several times with small portions (5 mL) of cold water and air dry the crystals on a Buchner funnel by suction until the crystals appear to be free of solvent. Test this crude product for the presence of unreacted salicylic acid using the ferric chloride test. Record the weight of the crude solid which probably contains water.

8. Stir the crude solid with 25 mL of a saturated aqueous sodium bicarbonate solution in a 150 mL beaker until all signs of reaction have ceased (evolution of CO_2 ceases).

9. Filter the solution through a Buchner funnel to remove any insoluble impurities or polymers that may have been formed. Wash the beaker and the funnel with 5 to 10 mL of water.

10. Carefully pour the filtrate with stirring, a small amount at a time, into an ice cold HCl solution (*ca* 3.5 mL of conc. HCl in 10 mL of water) in a 150-mL beaker and cool the mixture in an ice bath. Make sure that the resulting solution is acidic (blue litmus paper) and that the aspirin has completely precipitated out.

11. Filter the solid by suction and wash the crystals 3X with 5 mL of *cold* water each. Remove all the liquid from the crystals by pressing with a clean stopper or cork. Air dry the crystals and transfer them to a watch glass to dry. Test a small amount of the product for the presence of unreacted salicylic acid using the ferric chloride solution.

12. When the product is completely dry, weigh the product, determine its melting point (lit mp 135-136 °C) and calculate the percentage yield.

% yield = theoretical yield/ actual yield X 100

= amount of product recovered / theoretical amount of pro

X 100

Precaution:

Gloves and safety goggles are mandatory when anyone is performing an experiment in the lab.

H2SO4 is corrosive in nature avoid contact with eyes, body and clothes.

Recording exact amounts given in manual instead of exact approximate volume/mass used.
Crystals have to dissolving completely during recrystallization.

Always wash your hands before leaving the laboratory

EXPERIMENT NO. 10

Determination of surface tension and Viscosity

Aim: Determine the surface tension of a given liquid at room temp using stalgmometer by drop number method

Requirements: Stalgmometer, specific gravity bottle, a small rubber tube with a screw pinch cork, distilled water, experimental liquid.

Theory of experiment: In the drop number method, the number of drops formed by equal volumes of two liquid is counted. If m1 and m2 is the mass of one drop of each of the liquid having densities d1 and d2 respectively. If n1 and n2 is the number of drops formed by volume v of the two liquids, then their surface tensions are related as

$$¥_1/¥_2 = (d_1/d_2)*(n_2/n_1)$$

One of the liquid is water its surface tension and density are known. Then surface tension of the given liquid can be calculated.

Procedure:

1. Clean the stalgmometer with chromic acid mix, wash with water and dry it
2. Attach a small piece of rubber tube having a screw pinch cock at the upper end of the stalgmometer. 3. Immerse the lower end of the stalgmometer in distilled water and suck the water 1-2cm above mark A. adjust the pinch cork so that 10-15 drops fall per minute
4.Clamp the stalgmometer allow the water drops to fall and start counting the number of drops when the meniscus crosses the upper mark A and stop counting when the meniscus passes mark B
5. Repeat the exercise to take three to four readings
6. Rinse the stalgmometer with alcohol and dry it
7. Suck the given liquid in the stalgmometer and count the drops as in case of water
8. Take a clean dry weighing bottle weighs it with water as well as with liquid.
9. Note the temp of water taken in a beaker.

Observations:

Room temp=t ^{0}C

Density of water= dw

Surface tension of water= ¥ dynes/cm

No of drops From a Fixed Volume				Mean
Liquid	1	2	3	nl=
Water	1	2	3	nw=

Weight of empty specific gravity bottle=$w_1$gram

Weight of specific gravity bottle+water=w_2 gram

Weight of empty sp.gravity bottle+liquid=$w_3$gram

Weight of water= (w_2-w_1) gram
Weight of liquid= (w_3-w_1) gram

Calculations:

Density of the liquid
$D_l= (w_3-w_1)/(w_2 – w_1) *dw$
Surface tension of liquid=
¥l/¥w = $(d_l/dw)*(n_w/n_l)*$ ¥w

Result:
The surface tension of liquid is ………dynes/cm.

Precaution:

Ensure they are thoroughly cleaned with caustic soda and water to remove any contaminants that could affect surface tension measurements.
Clean and dry the stalagmometer before use.
Carefully clean the dish to avoid any dirt or residue.
Remove any dirt or residue from the metal ring used in some experiments.
Keep the capillary tube vertical while dripping in water.
Hold the stalagmometer in a vertical position throughout the drop counting process.
Maintain a constant and known temperature during the experiment.
Measure the height of the liquid column from the lowest point of the meniscus.

Observe carefully when the water and liquid are just passing the upper and lower marks of the stalagmometer.

EXPERIMENT NO. 11

Determination of the rate constant of a reaction

Aim: To study the kinetics of acid-catalyzed hydrolysis of ethyl acetate and to evaluate the value of the rate constant.

Theory of experiment:

A first order reaction is one in which the rate depends on concentration of only one of the reactants. Methyl acetate hydrolyses, in the presence of an acid, which acts as a catalyst, to give acetic acid and methyl alcohol.

$$CH_3COOCH_3 + H_2O \xrightarrow{\text{Dil. acid}} CH_3COOH + CH_3OH$$

Methyl acetate Acetic acid methyl alcohol

Rate = $k'[CH_3COOCH_3]\ [H_2O]$

Where k' is the specific reaction rate constant. Since water is present in large excess, its concentration remains practically constant throughout the reaction. As a result of this assumption, the above equation reduces to

Rate= $k'\ [CH_3COOCH_3]$

Where $k' = k\ [H_2O]$ = Constant

Hence, the rate of reaction is determined by the first power of the concentration of the ester and so the reaction is of the first order. It is however, a pseudo first order reaction which is not first order but is forced to obey the first order rate expression. Such reactions involve more than one molecule in the chemical reaction. As acetic acid is produced during the hydrolysis of methyl acetate, the reaction can be followed by titrating the reaction mixture with standard solution of an alkali.

The value of k can be calculated according to the first order rate expression which is given by,

$$k = \frac{2.303}{t} \times \log \frac{a}{a - x} \qquad [or]$$

$$k = \frac{2.303}{t} \times \log \frac{V_\infty - V_o}{V_\infty - V_t}$$

Where,

V_o = Volume of alkali used at t = 0 min,

V_t = Volume of alkali used at any time of reaction,

V_∞ = Volume of alkali used at the end of the reaction.

The values of rate constant can be calculated at different intervals of time t. It is independent of the initial concentration of the ester. It has the dimensions of $time^{-1}$. Calculate the k_t for each reading with respect to time and determine the calculated average k of the reaction. Plot a graph (straight line through origin) of log $V_\infty - V_0 / V_\infty - V_t$ against (t) and from the slope determine the rate constant k = slope x 2.303.

Apparatus required:

1. Measuring cylinder
2. Pipette
3. Burette
4. Conical Flask
5. Stop watch

Chemical required:

1. N/10 NaOH
2. N/20 HCL
3. Phenolphthalein indicator

Procedure:

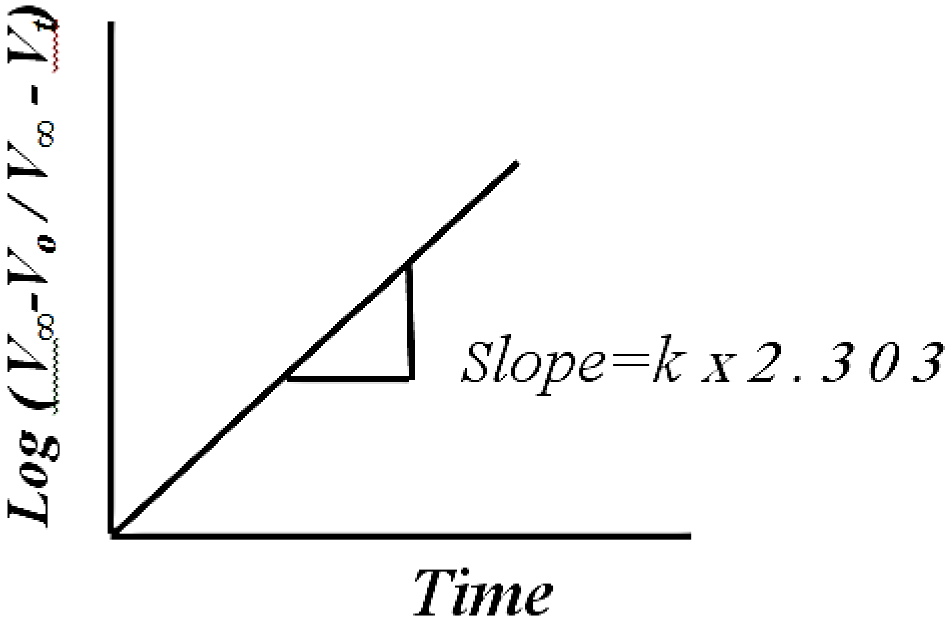
1. Take 25ml of cold water in a 250 ml conical flask. Add 4-5 drops of phenolphthalein indicator to it.
2. Take 50ml of N/20 HCL in a separate 100 ml conical flask. Add 5ml of ethyl acetate to it. Quickly shake the reaction mixture for few seconds and note the time because reaction starts. Immediately pipette out exact 5ml of reaction mixture take in the conical flask containing cold water and indicator.
3. Immediately titrate the mixture against N/10 NaOH solution in burette up to end point (pink colour) persisting for 20-30 seconds. Note down the titre value V_o.
4. Repeat the same operation and titration at every 10 minutes until 50th minute of the reaction. The titre value in such cases will be V_t.
5. Finally, heat the reaction mixture at 70^0-80^0C for 10-15 minutes to complete the reaction. And then once again take 5ml of the reaction mixture and titrate it to get V_∞.

Observations:

Sl. No.	**Time (min)**	**Burette Reading (ml)**		**Vol. of NaOH (ml)**	$\log\frac{V_\infty - V_o}{V_\infty - V_t}$	**k (min^{-1})**
		Initial	**Final**			
1	0			(value =V0)		
2	10					
3	20					
4	30					
5	40					
6	50					
7	60					
8	∞			(value =V∞)		
Calculated average rate constant (k_{av})=						

Calculation:

1. Complete the calculation as per table.
2. Draw the plot and calculate k_{plot}



Result:
From the linear nature of the plot, the order of the reaction was confirmed as 1 (pseudo first order). For the reaction the k_{av}=……… and k_{plot}=……………

Precautions:

Used fresh and accurately measured reagents.

Standardized the reagent used.

Maintained constant temperature.

Ensure rapid cooling of samples for titration to minimize further reaction.

EXPERIMENT NO. 12

Adsorption of acetic acid by charcoal

Aim: Determination of adsorption isotherm of acetic acid on activated charcoal

Theory of experiment: Adsorption is a process that occurs when a gas or liquid solute accumulates on the surface of a solid or a liquid (adsorbent), forming a molecular or atomic film (adsorbate). There are two type of adsorption:

Physisorption is a type of adsorption in which the adsorbate adheres to the surface only through Van der Waals (weak intermolecular) interactions, which are also responsible for the non-ideal behavior of real gases.

Chemisorption is a type of adsorption whereby a molecule adheres to a surface through the formation of a chemical bond, as opposed to the Van der Waals forces which cause physisorption.

Adsorption is usually described through adsorption isotherms, that is, functions which connect the amount of adsorbate on the adsorbent, with its pressure (if gas) or concentration (if liquid).

For liquids (adsorbate) adsorbed on solids (adsorbent), the Langmuir isotherm can be expressed by:

$$m = A_{max}\, kc / 1+kc \quad [\text{mol g}^{-1}, \text{resp. mol.kg}^{-1}]$$

Where:

m = substance amount of adsorbate adsorbed per gram (or kg) of the adsorbent, the unit is mol g^{-1}, resp. mol.kg^{-1}

A_{max} =maximal substance amount of adsorbate per gram (or kg) of the adsorbent, the unit is mol g^{-1}, resp. mol.kg^{-1}

k = adsorption constant ($mol^{-1}\ dm^3$)

c = concentration of adsorbate in liquid (mol dm^{-3})

Apparatus required:

1. Boiling flasks

2. Erlenmeyer's flasks

3. Funnels

4. Burettes

5. Titrimetric flasks

6. Pipettes,

7. Holders for funnel,

8. Holders for burettes,

9. Filtering paper,

10. Wessel for weighing coal,

11. Rubber stoppers,

Material required:

1. Solution of acetic acid

2. Solution of NaOH,

3. Activated charcoal,

4. Phenolphthalein.

Procedure:

1. Prepare aqueous solutions of acetic acid into numbered flasks following the scheme given in the table. The total volume of each solution is 60 ml. Use flasks fitted with stoppers.

Table 1 Scheme for acetic acid dilution

flask No	V(acetic acid)(cm^3)	V(distilled water)(cm^3)
1	6	54
2	12	48
3	18	42
4	30	30
5	42	18
6	60	0

2 Transfer 10 ml of the solution from each flask into numbered titrimetric flask. Final volume of acetic acid solution is VA = 50 ml per flask.

3 Determine the actual concentration of acetic acid in flasks by titration in this way: For titration, modify the volume in each titrimetric flask. Take away defined volume of the solution, to obtain in each flask the volume as given in the Table 2.

Table 2 Volumes of the acetic acid solutions (V) used for titration before and after adsorption

titrimetric flask No.	1	2	3	4	5	6
volume solution - V [ml]	10	10	5	5	5	2

4 Add 1-2 drops of phenolphthalein and titrate by NaOH.

5 Once the endpoint has been reached, read the burette. The volume of the base (X_i^o) - ml that was required to reach the endpoint write down to the Table 3.

6 Calculate the actual concentration of acetic acid before adsorption (C_i^o) in the flask No.1-6, respectively according Equation 2:

$C_i^o = X_i^o C_T / V$ [mol dm^{-3}]

Where,

X_i^o =is the volume of the titrant before adsorption (NaOH)

C_T =is the volume of the titrant (NaOH)

V =is the volume of the analyte

7 Using practical balance and Wessel for weighing coal, weigh 6 portions of activated charcoal, each portion 5 g. The accuracy of weighing must be with accuracy 0.01 g.

8 Put activated charcoal into numbered flasks with stoppers (1 portion per flask).

9 Plug up the flasks, and shake them. Wait for 20 minutes, the process of adsorption is in progress. Mix the mixtures for several times by flasks shaking within this period. Remark: The process of adsorption is a function of time too. It is important to put char- coal into flasks at the same time, to provide adsorption for the same period in each flask.

10 Filter the mixtures into clean and dry flasks. To avoid disturbing effect of adsorption of acetic acid into filtering paper, remove away the first portion of filtration, app. 5 ml.

11 Determine the final concentration of acetic acid (C_i) in each of the flasks after adsorption: From each solution, transfer the asked volume into clean and dry titrimetric flask, again following

Table 2.

12 Repeat points 4-6, and from the consumed base (X_i) from Table 3 determine the concentration of acetic acid (C_i) after adsorption according equation:

$C_i = X_i C_T / V$ [mol dm^{-3}]

Where

X_i = the volume of the titrant after adsorption (NaOH)

C_T & V = the same values as in Equation 2

Table 3

Experimental data

flask No	X_i^o(ml)	C_i^o[mol dm^{-3}]	X_i(ml)	C_i [mol dm^{-3}]	m_i [m mol g^{-1}]	1/ C_i [dm3 mol-1]	1/mi [g mmol-1]

Data treatment

Determination of the substance amount of acetic acid adsorbed per gram of the charcoal mi (m mol g^{-1}) in individual flask:

$$m_i = (C_i^o - C_i)V_A / g$$

Where,

C_i^o= concentration of acetic acid before adsorption

C_i = concentration of acetic acid after adsorption

V_A= volume of the liquid phase used for adsorption

g = the mass of the adsorbent – charcoal

i= 1-6 the number of flask

Determination of k and Amax

The Equation 1 one can rearrange into a form:

$1/m_i = 1/A_{max} + 1/ k A_{max}. C_i$

$1/m_i = f(1/C_i)$ should be a straight line

The slope represents the value of $1/ k A_{max}$

Intercept corresponds to $1/A_{max}$

Result:

$1/ k A_{max} =$

$1/A_{max} =$

EXPERIMENT NO -13

Saponification/acid value of an oil

Aim: Determination of Saponification Value of Fat or Oil

Theory of experiment: Saponification is the process by which the fatty acids in the glycerides of the oil are hydrolyzed by an alkali. The resultant salts of fatty acids are called soaps. Saponification value is the amount (mg) of alkali required to saponify a definite quantity (1g) of an oil or fat.

$C_3H_5 (C_{I7}H_{35}COO)_3 + 3KOH = C_3H_5(OH)_3 + 3C_{I7}H_{35}\cdot COOK$

Stearin Glycerol Potassium stearate

A known amount of fat or oil is refluxed with an excess amount of alcoholic KOH. After saponification, the amount of KOH is estimated by titrating against standard acid. This will give the amount of KOH utilized for saponification.

Apparatus required:

Conical flask
Few pieces of pumice
Burette
Pipette
Reflux condenser

Chemical required:

0.5N HCl
0.5N alcoholic KOH solution
1% phenolphthalein indicator

Procedure:

1• Melt the sample if it is not already liquid and filter through paper to remove any impurities and traces of moisture. The sample must be completely moisture-free.

2• Accurately weigh 2-5g of oil in 250ml conical flask.

3• Add 25-50ml of alcoholic KOH from burette to dissolve the fat completely.

4• Prepare a blank also by taking only alcoholic KOH same quantity used in the sample and allowing it to drain at the same duration of time.

5• Attach the reflux condenser with the flask.

6• Reflux the content by gentle boiling in a water bath for 1 hour.

7• Cool the flask to room temperature.

8• Add few drops of phenolphthalein indicator.

9• Titrate the content with 0.5N HCl.

Observation:

S. No	Weight of Sample (g)	Blank titre value (b)	Sample titre value (a)
1			
2			
3			

Calculation:

Saponification Value of oil (S.V.) = (b-a) x 28.05 / Wt. of sample (g)

1ml of 0.5N HCl = 28.06 mg KOH

Result:

Saponification value =

Precaution:

Please ensure that all glassware like is Pipette, Burette, Conical flask etc. are clean and free of any impurities.

Carry out the titration most accurately.