

I SEM Chemical Engineering Branch

# Chemistry Practical Manual

Science and Humanities Deptt.  
LEC Morbi

## LEC Morbi

**Subject: Chemistry 3110001**  
**Academic Year: 2020-21 (Odd)**

List of experiments

Sr. No.	Title	COs	Marks
1	Acid base titration	CO1	2
2	Validation of Beer and Lambert law	CO1, CO6	2
3	Fractional crystallization $K_2Cr_2O_7$ and NaCl	CO1, CO6	2
4	Wet-corrosion	CO1,	2
5	Gravimetric analysis-Chloride ion	CO1, CO6	2
6	Aspirin synthesis	CO1	2
7	Bakelite synthesis	CO1	2
8	Thin layer chromatography	CO1, CO6	2
9	Estimation of saponification value	CO1, CO6	2
10	Turbidity measurement	CO6	2
11.	A Visit to lab	-	-

As per GTU guideline minimum 8 to be performed

# GENERAL PRECAUTIONS TO BE TAKEN IN THE LABORATORY

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1. Never work in the laboratory unless a demonstrator or teaching assistant is present.
2. Do not throw waste such as match stems filter papers etc. into the sink. They must be thrown into the waste jars.
3. Keep the water and gas taps closed except when these utilities are needed.
4. Never taste any chemical unless instructed to do so and don't allow chemicals to come in contact with your skin.
5. While working with gases, conduct the experiment in a fume hood.
6. Keep all the doors and windows open while working in the laboratory.
7. You should know about the hazards and properties of every chemical which you are going to use for the experiment. Many chemicals encountered in analysis are poisonous and must be carefully handled.
8. Sulphuric acid must be diluted only when it is cold .This should be done by adding it slowly to cold water with stirring ,and not vice versa.
9. Reagent bottles must never be allowed to accumulate on the work bench. They should be placed back in the shelves as and when used.
10. Containers in which reaction to be performed a little later should be labeled. Working space should be cleaned immediately.

# CHEMICAL HAZARD SYMBOLS

Chemical hazard symbols are found on some home products, as well as bottles of chemical reagents in the lab. Here, we take a look at European hazard symbols and the various dangers that they warn of.



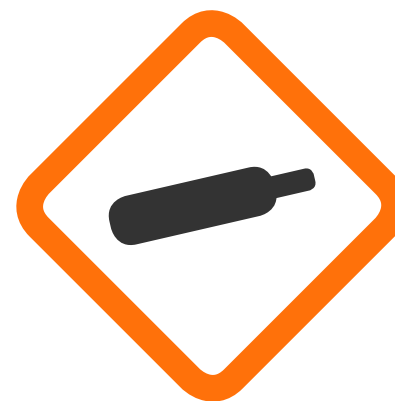
## ENVIRONMENTAL HAZARD

Indicates substances that are toxic to aquatic organisms, or may cause long lasting environmental effects. They should be disposed of responsibly.



## TOXIC

Indicates life-threatening effects, in some cases even after limited exposure. Any form of ingestion and skin contact should be avoided.



## GAS UNDER PRESSURE

Container contains pressurised gas. This may be cold when released, and explosive when heated. Containers should not be heated.



## CORROSIVE

May cause burns to skin and damage to eyes. May also corrode metals. Avoid skin & eye contact, and do not breathe vapours.



## EXPLOSIVE

May explode as a consequence of fire, heat, shock or friction. Chemicals with this label should be kept away from potential ignition sources.



## FLAMMABLE

Flammable when exposed to heat, fire or sparks, or give off flammable gases when reacting with water. Ignition sources should be avoided.



## CAUTION

May irritate the skin, or exhibit minor toxicity. The chemical should be kept away from the skin and the eyes as a precaution.



## OXIDISING

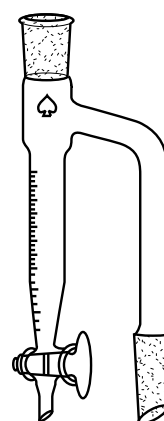
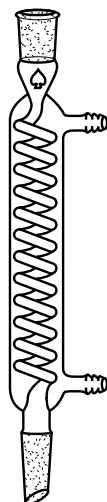
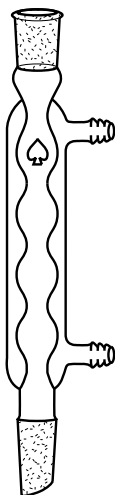
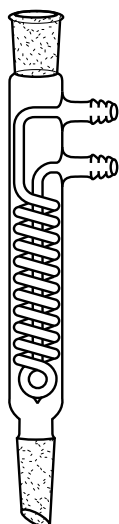
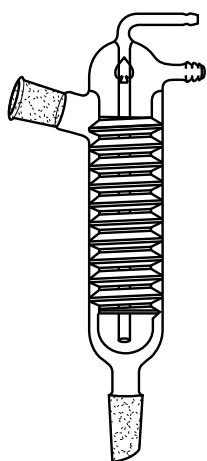
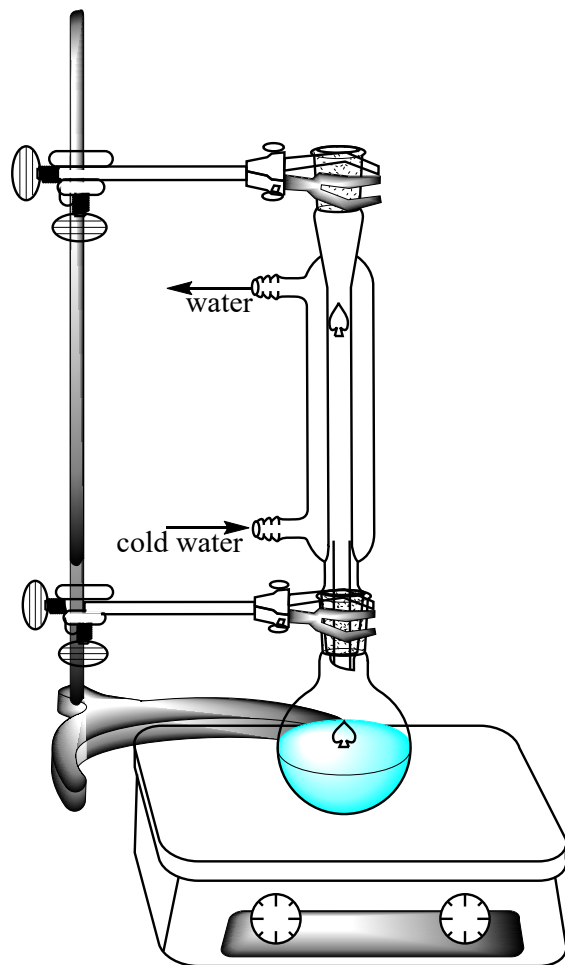
Burns even in the absence of air, and can intensify fires in combustible materials. Should be kept away from ignition sources.

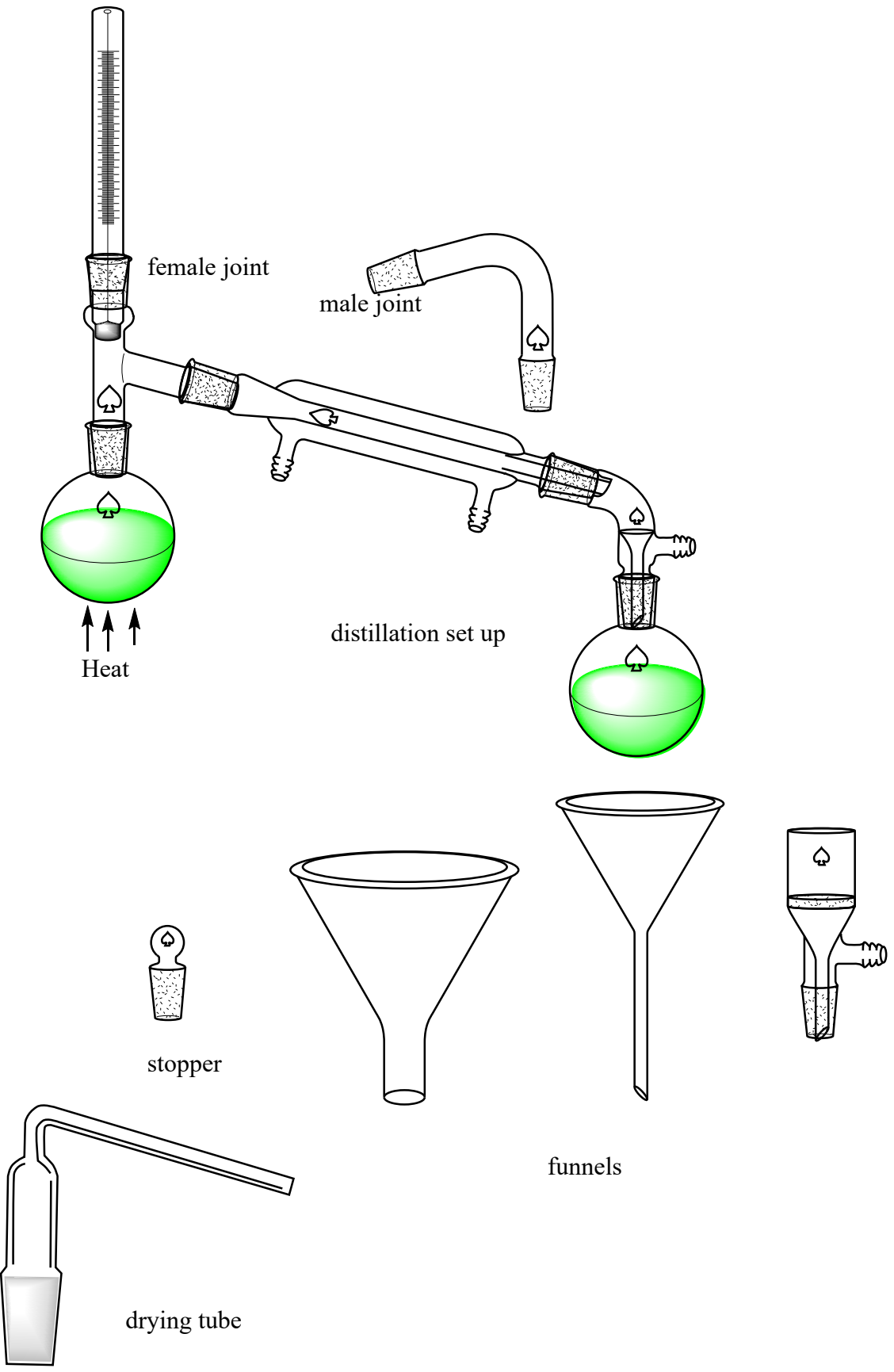


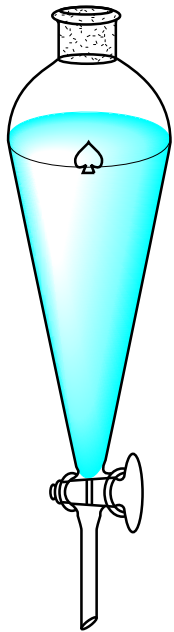
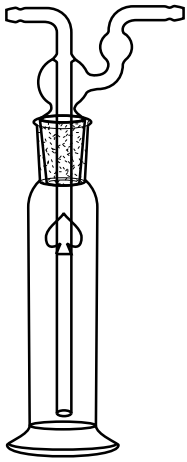
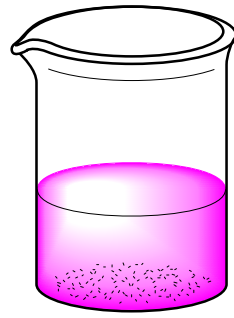
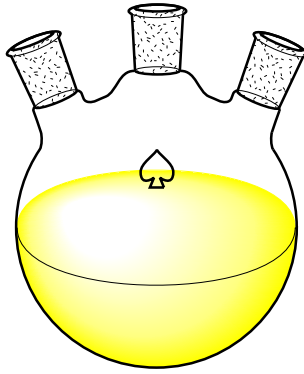
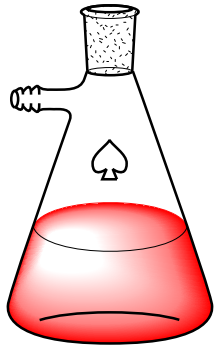
## HEALTH HAZARD

Short or long term exposure could cause serious long term health effects. Skin contact and ingestion of this chemical should be avoided.









## Activity-01 (A Visit to chemistry Laboratory)



Erlenmeyer flask



Two-neck round-bottom boiling flask



Round-bottom boiling flask



Beaker



500 ml filter flask



Glass funnel (short stem)



Powder funnel



Separatory funnel



Hirsch funnel



Büchner funnel\*



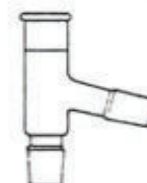
West Condenser



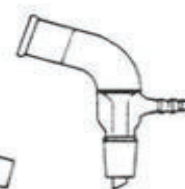
Distilling Column  
(shown with packing)



Claisen adapter



Distillation adapter



Vacuum adapter



filter adapter



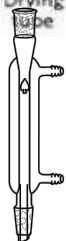
Drying tube



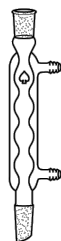
Clamp Holder



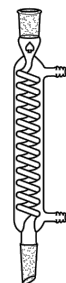
Clamp, extension



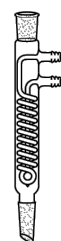
Liebig



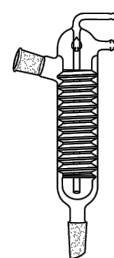
Allihn



Graham



Dimroth



Friedrichs



### Activity-02

Identify the following equipments/glass-wares in your chemistry laboratory-





### Activity

Write the name of other equipments those are not listed in this book-

- 1
- 2
- 3
- 4
- 5
- 6
- 7
- 8
- 9
- 10

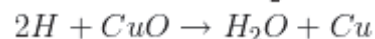
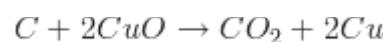
### Activity-03

#### Identification of elements in organic compound

Element	Order of abundance
Carbon	Always present
Hydrogen	Nearly always present
Oxygen	Generally present
Nitrogen, halogens and sulphur	Less commonly present
Phosphorus and metals	Rarely present

**1. Detection of Carbon and Hydrogen:** If the compound under investigation is organic then there is no need. These elements are confirmed by the following common tests.

The organic substance is mixed with about three times its weight of dry copper oxide. The mixture is then placed in a hard glass test tube fitted with bent delivery tube. The other end of this tube is dipped into lime water in another test tube. On heating the mixture strongly the following reactions take place:

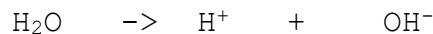


Thus, if carbon is present it is oxidized to carbon-dioxide and turns lime water milky. If hydrogen is also present, it will be oxidized to water which condenses in small droplets on the cooler walls of the test tube and inside the bulb, the formation of water can be further confirmed with anhydrous copper sulphate (white) which turns blue.

# Experiment 01

## Acid-Base Titration Introduction:

Water dissociates (self-ionizes) into  $H^+$  (hydrogen ion) and  $OH^-$  (hydroxide ion) ions naturally to a very small extent:



In a neutral solution the concentration of  $[H^+]$  and  $[OH^-]$  are equal at  $1.0 \times 10^{-7}$  M. An acidic solution is one in which the concentration of  $[H^+] > [OH^-]$ , and in an basic solution the  $[H^+] < [OH^-]$ . pH is a measure of the concentration of  $[H^+]$  and is defined as :

$$pH = -\log[H^+]$$

In a neutral solution  $pH = 7$ . A  $pH < 7$  indicates an acidic solution and  $pH > 7$  signifies a basic solution.

An acid-base reaction is one in which  $H^+$  ion are transferred from an acid to a base:



In this experiment we will explore the use of titration, adding small quantities of a base to an acid and recording the rise of pH. We can plot the pH against the amount of base added producing a titration curve. The steepest point on the curve occurs at the equivalence point, when the acid is exactly neutralized. Because the titration curve is so steep near equivalence only a small amount of base can result in a large change in pH.

This point is the end point of our reaction and can be signified by the color change of an indicator. An indicator, in an acid base reaction, is a substance whose color changes over a particular pH range. Phenolphthalein is an example of an indicator which changes from colorless to pink as pH goes from 8 to 10.

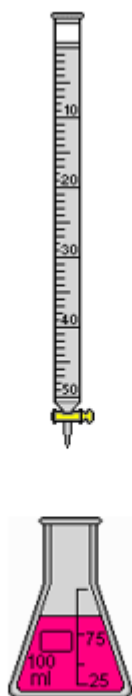
In this lab we will be using a strong acid and a strong base to perform our acid base titration. Strong acids and bases can be assumed to dissociate in water completely. We will be adding NaOH, a strong base, to HCl, a strong acid. The NaOH will neutralize the HCl in a reaction that produces sodium chloride (salt) and water:



Since the stoichiometric ratio of hydrogen ions to hydroxide ion is 1:1 in this reaction, the number of moles of NaOH added to HCl will be equal at the end point or:

$$(\text{Molarity acid}) \times (\text{Volume of acid}) = (\text{Molarity of base}) \times (\text{Volume of base added})$$

## Procedure



Step 2: Add an indicator to the acid, select the flask and add 2 drops of phenolphthalein indicator. The indicator menu is available under the chemicals main menu (Chemicals→Indicators) or the context menu.

Step 3: Display pH, select flask and add a pH meter to the acid solution using the equipment main menu (Equipment→pH meter) or the context menu.

Step 4: Turn on the collection of titration data, select the flask and turn on collection of titration data using "Collect Titration Data" menu from the procedures menu or from the right-mouse context menu.

Step 5: Open the titration data window by selecting the "View Titration Data" from the Procedures menu. (Note: this window will plot a titration curve once you start titrating)

Step 6: Fill burette with NaOH, obtain a 50 ml burette and fill with .2M NaOH solution.

Step 7: Titrate NaOH into HCl until end point, record initial burette volume and add NaOH (quickly at first then slowly) until the HCl solution turns pink and record the final burette volume of NaOH in burette.

## Observations:

Initial burette volume: 50

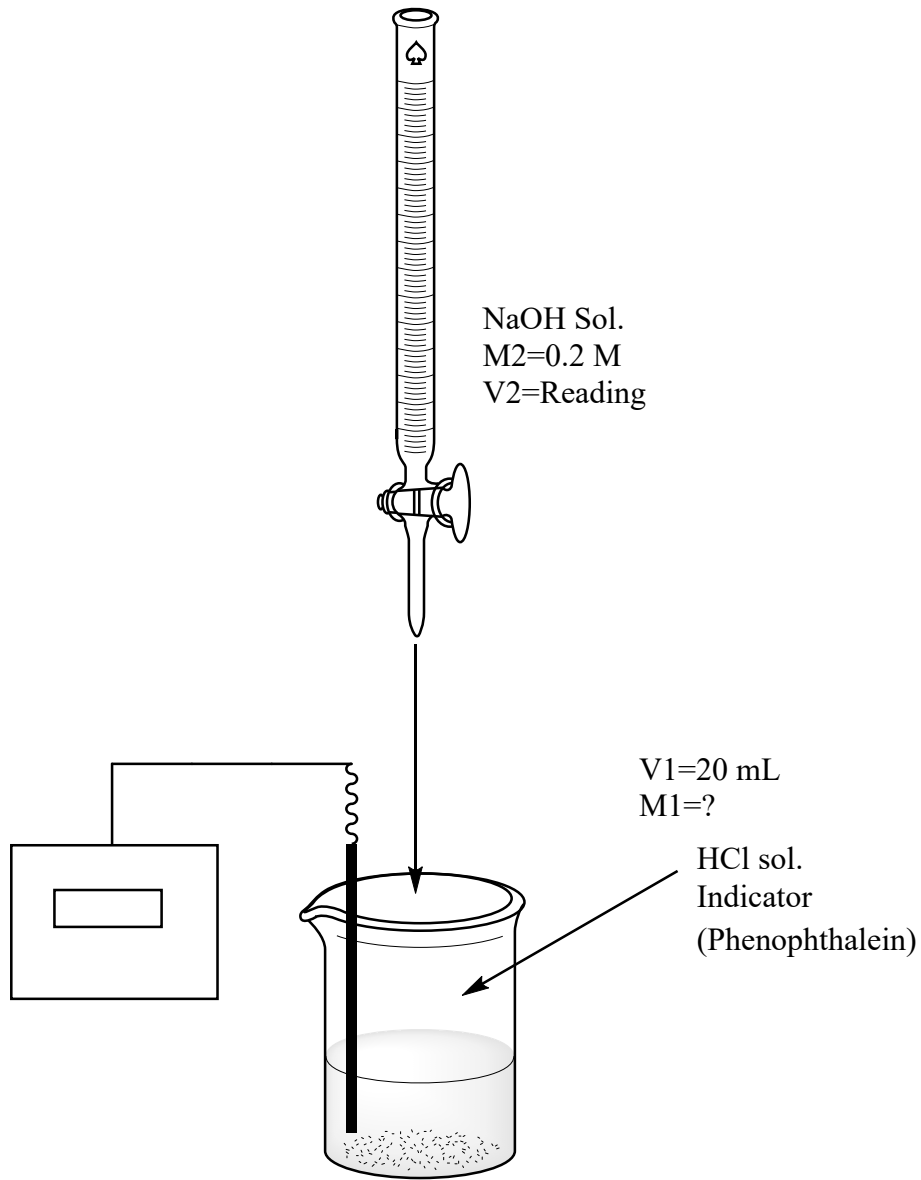
Final burette volume: 30

Total volume titrated to reach end-point: 20

What happened to the rise in pH as the end-point was approached?

Ans. rapidly increased

Prepare a titration curve with pH on the y-axis and volume of base added on x-axis.  
Determine the equivalence point from the curve (steepest point of curve).



V <sub>2</sub>	pH
0	0.1
3	0.12
6	0.15
9	0.7
12	1.5
15	2.5
18	4
21	12
24	13
27	13.5

$$M_1V_1 = M_2V_2$$

$$M_1 \times 20 = 0.2 \times 19$$

$$M_1 = 0.19 \text{ M}$$

## Experiment 02

### Beer and Lambert Law-V-Lab Simulator

#### Determining the concentration of copper (ii) sulfate

##### INTRODUCTION

In this experiment, we will use photometry to determine the concentration of an unknown solution (of food coloring). In order to accomplish this goal, we must first determine what wavelength of light the solution best absorbs (the wavelength of maximum absorbance). Then we will prepare solutions of known concentration, measure their absorbances, and create a calibration plot. We can then use information from that plot to determine the concentration of our unknown.

##### BACKGROUND

Spectrophotometry is the study of light as a function of its wavelength. Light is energy, in the form of electromagnetic radiation. That light has the capability to do work. Plastic left out in the sun degrades and becomes brittle. If you put a bleaching agent in your hair and go sit in the sun, sunlight causes your hair to bleach. Solar cells change light energy into electricity. We can understand the energy contained in light by understanding the light wave. If we look at the wave, we can define the length of the repeat units (wavelength), how many waves repeat per second (frequency) and the height of the wave (amplitude). We can relate frequency and wavelength for an electromagnetic wave by:  $c = \lambda \cdot \nu$ , or the speed of light is equal to the frequency of the wave times the wavelength. This equation works for the whole electromagnetic wave series. Refer to your textbook for a more complete description.

We are looking at a small portion of this spectrum, between the ultra violet and infrared sections, which we call visible light. We see visible light. We can feel infrared radiation as heat. We cannot visually see ultraviolet light but we feel the sunburn caused by overexposure to UV light. Violet light, at a wavelength of about 400 nm has higher energy than red light, at about 700 nm. This visible light can cause chemical changes or reactions in compounds. The compounds absorb specific wavelengths of light. These reactions can be permanent or reversible. Different molecules absorb different wavelengths of light. The color of an object is due to the combination of those wavelengths that an object did not absorb. Plants appear green because they absorb the wavelengths in the blue and red regions, leaving the greens, to be seen. The absorbed light energy is used in photosynthesis, the conversion of CO<sub>2</sub> and water into sugars and carbohydrates.

We can differentiate compounds based on their light absorption. We can look at blue or green mineral samples and predict that there are copper compounds present. Dark red/orange rocks often have iron compounds. A red ruby can be identified as chromium doped alumina. We can more precisely understand these compounds by burning a small amount and examining the light spectra given off by



the flame. This is called flame emission spectroscopy. This information is tabulated into tables. You can estimate the wavelength of the color absorbed visually by samples by use of the following table.

The image displays two screenshots of a virtual lab interface for Beer's Law. The top screenshot shows a red light source at 508 nm, a pink solution, and a detector showing 0% transmittance. The bottom screenshot shows a red light source at 780 nm, a blue solution, and a detector showing 5.63% transmittance. Both screenshots include a ruler, a concentration slider, and a solution dropdown menu.

**Top Screenshot:**

- Wavelength: 508 nm (preset)
- Solution: Drink mix
- Concentration: 100 mM
- Detector: 0% (Absorbance selected)

**Bottom Screenshot:**

- Wavelength: 780 nm (preset)
- Solution:  $\text{CuSO}_4$ : Copper(II) sulfate
- Concentration: 100 mM
- Detector: 5.63% (Absorbance selected)

5. Plot your calibration data, [conc] (on the x-axis) vs. **Absorbance** in your lab notebook. Use nearly a full page to do this. Follow the guidelines for good graphing, including drawing a *best fit* line.

6. Calculate the slope (**k**) by choosing the two points from your plot that would best estimate the slope of your best fit line and plug them into the slope equation.
7. Express the relationship in Beer's law form, substituting your slope for **k**.
8. Add your unknown data point to the best fit line based on its absorbance and clearly label it. Drop a dotted line to the concentration axis to estimate the concentration of your unknown.
9. Use the Beer's law equation to calculate the concentration of your unknown. It should be close to the estimate in the step above.

	Conc	T%
1	100	63.12
2	200	39.87
3	300	25.18
4	400	15.9

## Experiment 03

### Fractional Crystallization Introduction:

The technique of fractional crystallization is used by chemists to separate a mixture of dissolved substances (salts) in a solvent into its purified components (salts). This is accomplished by taking advantage of the variation of solubility of different salts in a given solvent with temperature. The solution containing the mixture is evaporated until the least soluble substance (salt) crystallizes out.

In this experiment you will be given a sample containing potassium dichromate  $K_2Cr_2O_7$  and sodium chloride  $NaCl$ , both of which are water soluble ionic substances, with different solubilities at different temperatures. Sodium Chloride exhibits little change in solubility between the range of  $0\text{ }^\circ\text{C}$  to  $100\text{ }^\circ\text{C}$ , while potassium dichromate solubility increases 16-fold over the same temperature range. This property can be used to separate a mixture of the two salts from a solution.

#### Procedure:

Step One: Prepare a solution of the two salts - First obtain a 100 ml beaker then add 60 ml of distilled water at room temperature. Next add a 12 gram sample of Sodium Chloride ( $NaCl$ ) and a 15 gram sample of Potassium dichromate ( $K_2Cr_2O_7$ ), mix and if necessary heat the solution with a bunsen burner until all the solid has dissolved.

Step Two: Cool solution - Obtain a 600 ml beaker from the equipment menu, then fill with 300 ml of ice water. Ice water may be obtained using the distilled water dialog box from the chemical menu. Place the 100ml beaker into the 600ml beaker by selecting the combine menu option, from the arrange menu. Cool until all the potassium dichromate has come out of solution ( $0\text{ }^\circ\text{C}$ ).

Step Three: Remove solid potassium dichromate from solution - First obtain a filtering flask with Buchner funnel, this is done by picking a 100 ml Erlenmeyer flask and then adding a buchner funnel to it by selecting the Buchner funnel menu item from the equipment menu, while the flask is picked. Then pour the solution from the beaker into the filtering flask with buchner funnel, until beaker is empty.

Step Four: Removing sample from the filter - To remove sample from filter pick the flask and again select the buchner funnel option from the equipment menu. This time a dialog box will appear indicating the presence of solid in the filter. Select the piece of lab equipment you wish the sample to be transferred to and press OK.

Step Five : Removing sodium chloride from solution - to finally remove sodium chloride from solution heat the solution and boil away the remaining water. Check the final product to ensure that you can account for all of the salts.

#### Observations:

Weight of  $\text{K}_2\text{Cr}_2\text{O}_7$  recovered: 15 g

Weight of  $\text{NaCl}$  recovered: 12 g

## **Experiment 04**

### **To study Wet corrosion loss of Metal by weight loss Method.**

#### ***Introduction:***

Industrial revolution that is ever expanding within different parts of the world has several advantages and disadvantages in the quality of environment. Most industries utilize metals or their ores (such as mild steel, aluminum, zinc, and copper) in the fabrication of their installations. In most cases, these metals are exposed to aggressive medium/media and are prone to corrosion. Corrosion is an electrochemical process that gradually returns the metal to its natural state in the environment. Corrosion in industries is often activated by processes such as acid wash, etching, pickling and others.

The cost of replacing metals due to corrosion is often exorbitant and economically unbearable. Therefore, industries have adopted several options to control corrosion of metals including anodic/cathodic protection, painting, electroplating and galvanizing. However, the use of corrosion inhibitors has proven to be one of the most effective methods.

#### **Theory of corrosion**

Metal corrosion is the decay of metals as they combine with oxygen to form metallic oxides. Corrosion is a chemical process that is the reverse of the process of smelting the metals from their ores. Very few metals are found in their pure state in nature. Most are found as metallic oxides. These oxides have other undesirable impurities in them. The refining process involves the extraction of the base metal from the ore. The base metal is then mixed with other elements (either metallic or nonmetallic) to form alloys. Alloying elements are added to base metals to develop a variety of useful properties. For instance, in aircraft structural applications, high strength-to-weight ratios are the most desirable properties of an alloy. After the base metals are refined, whether alloyed or not, they have a potential to return to their natural state. However, potential is not sufficient in itself to begin and promote this reversion; a corrosive environment must also exist. The significant element of the corrosive environment is oxygen. The process of oxidation (combining with oxygen) causes wood to rot or burn and metals to corrode. Control of corrosion depends upon maintaining a separation between susceptible alloys and the corrosive environment. This separation is accomplished in various ways.

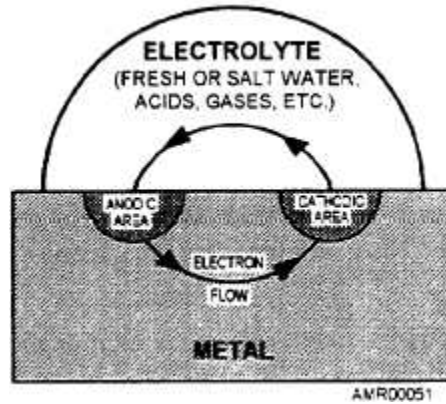
A good intact coat of paint provides most of the corrosion protection on naval aircraft. Sealants used at seams and joints prevent entry of moisture into the metal. Preservatives are used on unpainted areas of working parts. Finally, shrouds, covers, caps, and other mechanical equipment provide varying degrees of protection from corrosive mediums. However, none of these procedures

will provide 100-percent protection. Weathering causes paint to oxidize and decay. Sealants may be worked out by vibration or be eroded by rain and windblast. Preservatives offer only temporary protection when used on operating aircraft. The mechanical covering can be installed improperly or negligently. Control of corrosion begins with an understanding of the causes and the nature of corrosion. Corrosion Theory in its most familiar form is a reaction between metal and water, and is electrochemical in nature. the process of electrochemical or direct chemical attack on metals. The reaction is similar to that which occurs when acid is applied to bare metal. Corrosion Define the theory of corrosion and its process.

Identify the publications and materials used in the prevention of corrosion. Metal corrosion is the decay of metals as they combine with oxygen to form metallic oxides. Corrosion is a chemical process that is the reverse of the process of smelting the metals from their ores. Very few metals are found in their pure state in nature. Most are found as metallic oxides. These oxides have other undesirable impurities in them. The refining process involves the extraction of the base metal from the ore. The base metal is then mixed with other elements (either metallic or nonmetallic) to form alloys. Alloying elements are added to base metals to develop a variety of useful properties. For instance. in aircraft structural applications, high strength-to-weight ratios are the most desirable properties of an alloy. After the base metals are refined, whether alloyed or not, they have a potential to return to their natural state. However, potential is not sufficient in itself to begin and promote this reversion; a corrosive environment must also exist. The significant element of the corrosive environment is oxygen. The process of oxidation (combining with oxygen) causes wood to rot or burn and metals to corrode. Control of corrosion depends upon maintaining a separation between susceptible alloys and the corrosive environment. This separation is accomplished in various ways.

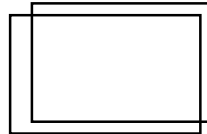
A good intact coat of paint provides most of the corrosion protection on naval aircraft. Sealants used at seams and joints prevent entry of moisture into the metal. Preservatives are used. The electrochemical attack involves metals of different electrical potential. These metals do not have to be in direct contact. If one metal contains positively charged ions and the other negatively charged ions, all that is needed is an electrical conductor. When the conductor is present, current will flow between the two metals, as in the discharge of a dry-cell battery. In electrochemical corrosion, the electrical conductor may be any foreign material, such as water, dirt, grease, or any debris that is capable of acting as an electrolyte. The presence of salt in any of the foregoing mediums accelerates the current flow and increases the rate of corrosive attack. Once an electrical connection is made, the electron flow is established in the direction of the negatively charged metal (cathode).

This action eventually destroys the positively charged metal (anode). Preventive measures include avoiding the establishment of the electrical circuit and removing corrosion as soon as possible to avoid serious damage. Figure shows the electron flow in a corrosive



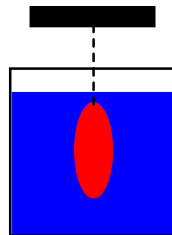
### Procedure:

1. Metal coupons of dimension  $5.0 \times 4.0 \times 0.15 \text{ cm}$  ( $41.35 \text{ cm}^2$ ) were cut and wet-abraded with silicon carbide abrasive paper (sand paper) (from grade #1000 to #1200), rinsed with distilled water and in acetone before they were dried in the air.



**W=10 mg**

2. The pre-cleaned and weighed coupons were suspended in beakers containing the test solutions using glass hooks and rods.



3. Tests were conducted under total immersion conditions in 150 mL of the aerated and unstirred test solutions.
4. Immersion time was varied from 1 hour to 5 days (120 h) in 0.1 M HCl.
5. The coupons were retrieved from test solutions after every 12 h, appropriately cleaned, dried and re-weighed.
6. The weight loss was taken to be the difference between the weight of the coupons at a given time and its initial weight.

7. The effect of temperature on metal corrosion and corrosion inhibition was investigated by repeating the experiments at 303 K.
8. All tests were run in duplicate and the data obtained showed good reproducibility.

**Observations:**

The corrosion rate, CR g cm<sup>-2</sup> h<sup>-1</sup> and inhibition efficiency, IE%, as functions of concentration in the acid media were calculated using the equation:

Time of Immersion: .....

Temperature at which the experiment is carried out :.....

Time	Weight of Coupon before experiment (in mg)	Weight of coupon after experiment (in mg)	Weight loss (in mg)
12 h	10	9.24	
24 h	10	9.23	
36 h	10	9.22	
48 h	10	9.20	

$$\text{Corrosion Rate (mg h}^{-1}\text{cm}^{-2}) = \frac{dW}{dt \times A} \quad 0.16/12 \times 41.35 \quad (1)$$

Where ΔW is the weight loss and dt is the time of immersion.



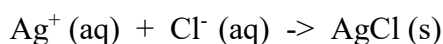
# Experiment 05

## Gravimetric Analysis of Chloride

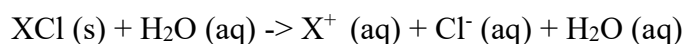
### Introduction

Gravimetric analysis is a method for determining the amount of a chemical present by converting it, through a chemical reaction, into another of known chemical composition, which can be isolated and weighed.

In this experiment we will determine the composition of an unknown chloride salt (XCl), by reacting chloride and silver ions to produce a silver chloride precipitate. The silver chloride is then filtered out and weighed. Based on the known stoichiometric ratios of the silver chloride reaction 1:1 we can determine the amount of chloride initially present in our metal sample.



We will begin our experiment by first adding a weighed sample of our unknown XCl sample to water, the XCl will disassociate in the water into  $\text{Cl}^-$  and  $\text{X}^+$  ions:



A 1M solution of  $\text{AgNO}_3$  is added to serve as the source for our  $\text{Ag}^+$  ions, as  $\text{AgNO}_3$  is added  $\text{AgCl}$  will precipitate out of the solution.  $\text{AgNO}_3$  is added until no more  $\text{AgCl}$  precipitates out of solution, the initial quantity of  $\text{Cl}^-$  ions present serves as the limiting agent to our reaction. The amount of excess  $\text{AgNO}_3$  added is not important, once all of the  $\text{AgCl}$  has precipitated out of solution. We then filter out our  $\text{AgCl}$  precipitate, and weigh it.

From the mass of our  $\text{AgCl}$  precipitate we can determine how many moles of  $\text{AgCl}$  were produced:

$$\text{Moles of AgCl} = \text{weight of AgCl} / \text{GMW of AgCl} = \text{weight of AgCl} / 143.32$$

We can determine the % of  $\text{Cl}^-$  present in our unknown:

$$\text{Moles of Cl}^- = \text{moles of AgCl}$$

$$\text{Weight of Cl}^- \text{ in XCl sample} = (\text{moles of Cl}^-) \times (\text{atomic weight of Cl}^- = 35.4527)$$

$$\% \text{ Cl}^- \text{ in sample} = (\text{weight of Cl}^- \text{ in XCl sample} / \text{weight of XCl sample}) \times 100 \%$$

### Procedure

Step 1: Obtain unknown, in a 250 ml Beaker add 5 g of unknown chloride XCl.

Step 2: Add water to XCl, fill beaker to 100ml level by adding water, stir solution until XCl is completely dissolved.

Step 3: Add Concentrated Nitric Acid, add 1 ml of concentrated acid to XCl solution.

Step 4: Add 1M AgNO<sub>3</sub>: Obtain a 100ml graduated cylinder and fill with 1M AgNO<sub>3</sub>, add AgNO<sub>3</sub> to XCl solution in 5 to 25 ml increments. A precipitate of AgCl will form and gradually settle. Continue to add AgNO<sub>3</sub> until no more precipitate forms. To verify that the reaction is complete check the chemical properties of the beaker (by double-clicking on it) and confirm that all of the XCl (in solution) has been consumed.

Step 5: Filter and weigh AgCl : Obtain a 250ml Erlenmeyer flask and add a filter (Buchner funnel), pour the contents of the beaker into the flask. Remove the filter from the Erlenmeyer flask (by again selecting the filter menu or button) and save the solid contents in a watch glass. Weigh the sample and record the result. \*

\*Note that in an actual lab the AgCl filtered precipitate would need to be dried to remove excess water, however in this simulation the filtered precipitate is free of water.

### Observations:

Initial unknown chloride sample weight (g): 5

Weight of AgCl precipitate: 12.262 g

Approximate volume of AgNO<sub>3</sub> solution added (ml): 100 mL

Moles of AgCl = ( AgCl precipitate mass/143.32 ):

Weight of Cl<sup>-</sup> in XCl sample = (moles of AgCl)x(atomic weight of Cl<sup>-</sup> = 35.4527) :

% Cl<sup>-</sup> in sample = (weight of Cl<sup>-</sup> in XCl sample / weight of XCl sample) x 100 % :

What happened as excess AgNO<sub>3</sub> solution was added to the XCl solution?

Ans- no further ppt

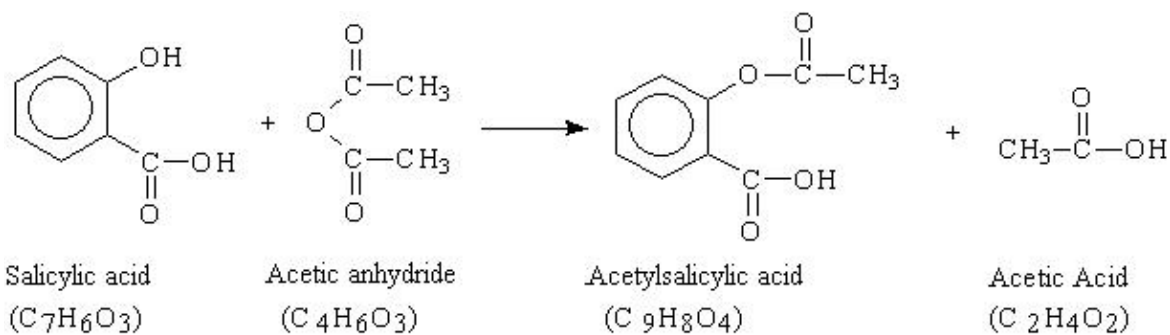
## Experiment 06

# Synthesis of Aspirin

### Chemicals & Equipment

Bunsen burner or hot plate  
Boiling stones  
Buchner funnel  
500-mL filter flask  
filter paper  
250-mL flask & beaker  
melting point apparatus

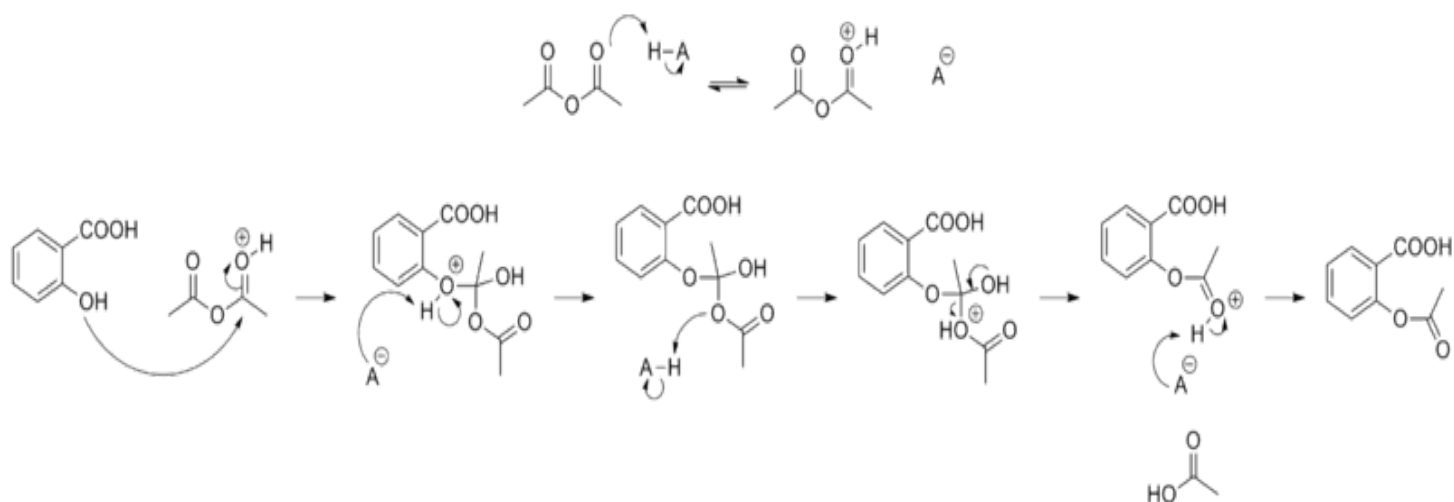
salicylic acid  
commercial aspirin  
1% FeCl<sub>3</sub>  
acetic anhydride  
18M H<sub>2</sub>SO<sub>4</sub>  
95% ethanol  
capillary tubes



### Introduction



Aspirin is most widely sold over-the-counter drug. It has the ability to reduce fever (an antipyretic), to reduce pain (an analgesic), and to reduce swelling, soreness, and redness (an anti-inflammatory agent). One of the first recorded accounts for the discovery of aspirin appeared in England, in 1763, crediting the bark of willow trees with a beneficial effect in alleviating distress due to fevers, aches, and pains.



## Part I Synthesis of Aspirin

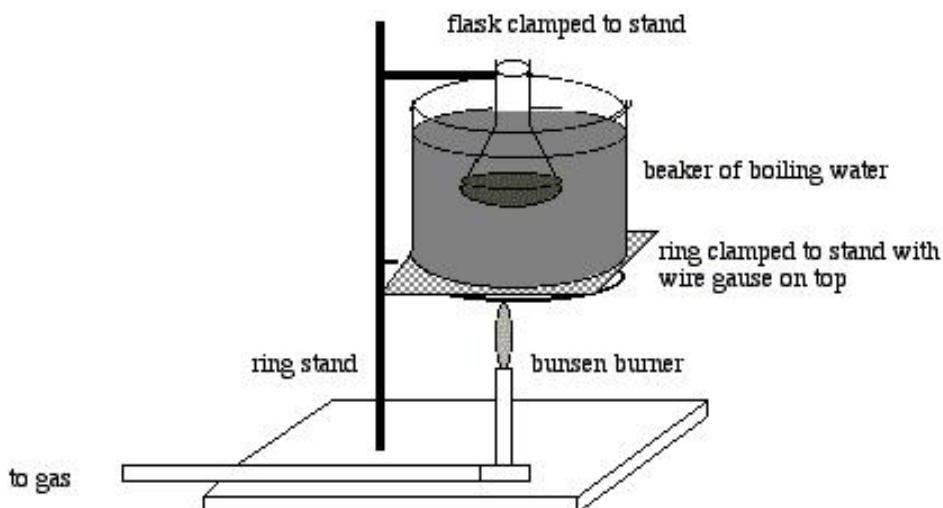
- Caution! The preparation of aspirin involves the use of two hazardous materials - concentrated sulfuric acid and acetic anhydride. Proceed only if you have a fume hood to work in, and after you have listened carefully to the instructor's safety directions.

### Procedure

1. Weigh 4.0 g (0.030 mole) of salicylic acid in a 125 mL Erlenmeyer flask. Using this quantity of salicylic acid to calculate the theoretical yield of aspirin
2. Carefully add 6 mL (0.051 mole) of acetic anhydride to the flask.
3. **Using extreme caution**, add 5 drops of concentrated sulfuric acid to the flask, swirl gently, and place the flask in a beaker of boiling water.
4. Clamp the flask to a ring stand and heat for 20 minutes. Constantly stir with a glass rod; the entire solid must completely dissolve.
5. Remove the flask from the boiling water bath and allow to cool to room temperature. Crystallization should occur during cooling. If crystals begin to grow, let the flask sit undisturbed until crystals stop growing then add the 40 mL of ice water.
6. If crystals do not grow, slowly pour the solution into a 250-mL beaker containing 40 mL of ice water, mix thoroughly, and place the beaker in ice water and let sit undisturbed until crystals have grown. The water destroys any unreacted acetic anhydride and will cause the insoluble aspirin to precipitate out of solution.
7. Collect the crystals by vacuum filtration.
8. Wash the crystals with two 10-mL portions of cold water followed by one 10- mL portion of cold ethanol. Allow the crude product to dry, then weigh it on the rough balance.
9. Weigh a watch glass. Add the crystals and re-weigh. Calculate the weight of crude aspirin.

Determine the percent yield. Test a small amount of this crude product for its melting point as described in Part II. Test the freshly made product for purity. Aspirin naturally decomposes into

acetic acid over time so the purity test should be done the day the aspirin is prepared. Save some of your aspirin for testing.



### Recrystallization

*The crude aspirin needs to be further purified. The crude products obtained from most preparations of organic compounds are contaminated with unreacted starting materials and substances from side-reactions. These can often be eliminated by a simple process known as RECRYSTALLIZATION. The next phase of this experiment involves the recrystallization, and thus, purification, of your crude aspirin sample.*

10. Dissolve about 2-4 g of your crude product in about 20 mL ethyl alcohol in a 125 mL Erlenmeyer flask, warming the alcohol in a water bath to effect dissolution. **Caution: Do not use a flame to heat ethyl alcohol. As it is a flammable compound.** If you obtained less than 6 g of crude product, use proportionately less alcohol.
11. If any solid material remains undissolved, filter the solution.
12. Add 50 mL of warm water (about 50°C) to the clear alcohol solution. If any crystals appear at this point, heat the contents of the flask until they dissolve.
13. Set the flask aside to cool, observing it carefully.
14. When crystals start to form, cool the flask by surrounding it with cold water. The crystallization process will then go to completion.
15. Collect the crystals by vacuum filtration.
16. Allow the crystals to dry.
17. Save your sample of the aspirin for a melting point determination and further analysis.

Results:

Weight of dry aspirin: 4 g

## Experiment 07

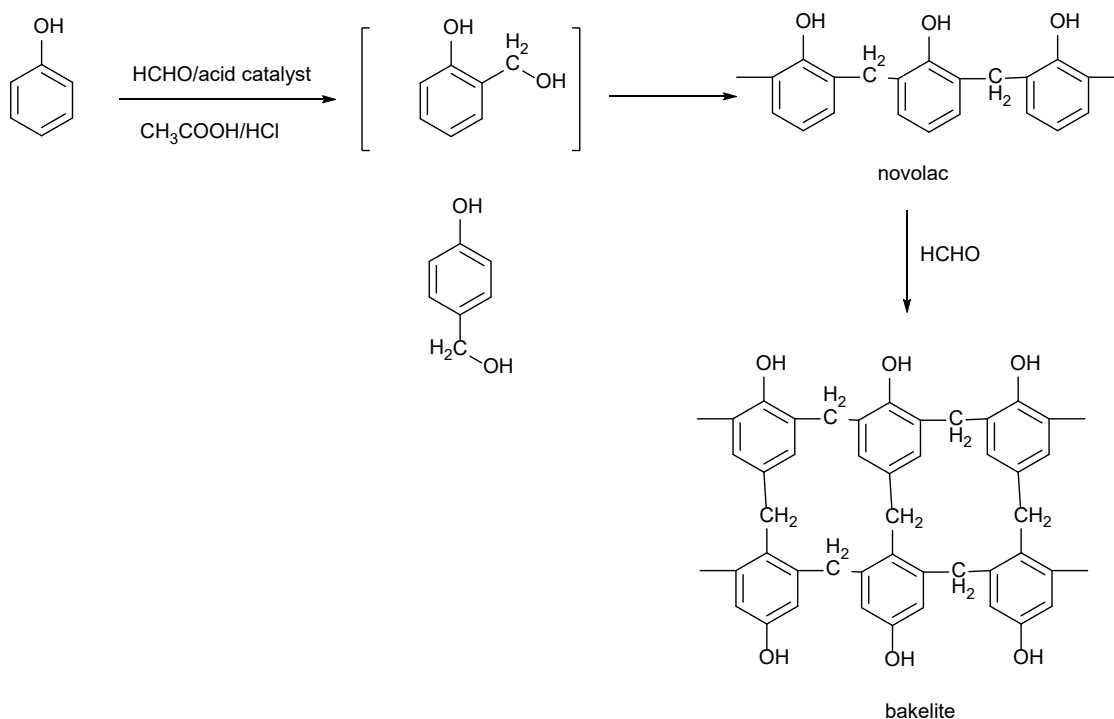
**Aim:** To prepare the phenol-formaldehyde resin.

**Chemicals required:** Phenol (2g), 40% aq formaldehyde solution or formalin (2.5 mL), glacial acetic acid (5 mL) and conc. HCl (8mL).

**Theory:** Phenol formaldehyde resin or P-F resin or phenolic resins (also called phenoplasts) are important class of polymers which are formed by condensation polymerization of phenol and formaldehyde in acidic or alkaline medium. Following steps are involved:

### Step 1: Formation of methylol phenol derivative

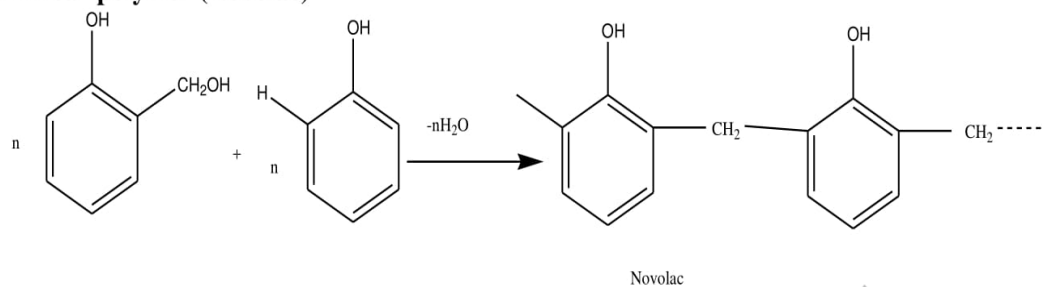
Initially the monomers combine to form methylol phenol derivative depending upon phenol to formaldehyde ratio.



**Step 2:** The phenol formaldehyde derivatives react among themselves or with phenol to give a linear polymer or a higher cross linked polymer.

#### (a) Linear polymer (Novolac)

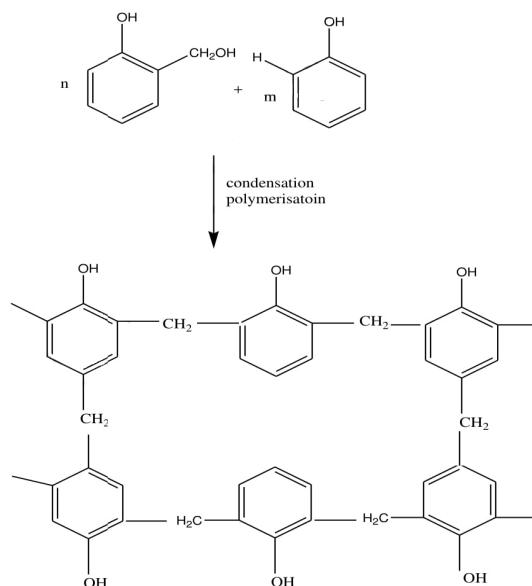
**(a) Linear polymer (Novolac)**



**(b) Cross linked polymer (Bakelite)**

A highly cross linked thermosetting polymer called Bakelite may be formed by further condensation of novolac or methylol derivative.

It was first prepared by Backeland. It is easily formed if curing agent hexamethylene tetramine is added during synthesis.



**Procedure**

1. Place 5 mL of glacial acetic acid and 2.5 mL of 40 % aq formaldehyde solution in a 100 mL beaker. Add 2 g phenol safely.
2. Wrap the beaker with a wet cloth or place it in a 250 mL beaker having small amount of water in it.

3. Add conc. HCl drop wise with vigorous stirring by a glass rod till a pink coloured gummy mass appears.
4. Wash the pink residue several times with to make it free from acid.
5. Filter the product and weigh it after drying in folds of a filter or in an oven. Report the yield of polymer formed.

### **Observation**

Weight of empty watch glass =  $W_1$  g

Weight of watch glass + polymer formed =  $W_2$  g

Weight of polymer formed (W) =  $W_2 - W_1$  g

### **Result**

Weight of phenol formaldehyde resin = 3.1 g

<https://www.youtube.com/watch?v=f-oNngiG9ek>



**LEC, Chemical Engineering Department**

**Practical Sheet 8**

Name of Student-----Enl. No.-----

Teacher Sign-----

Date-----

**Experiment No.8**

**Back Ground of experiment**

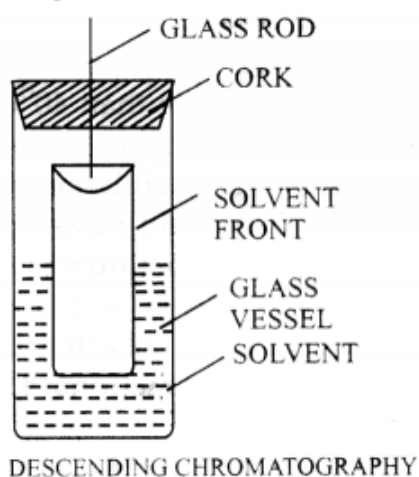
**Chromatography**

**Classification:** Chromatography can be classified into following types:

- (1) Paper Chromatography
- (2) Column Chromatography
- (3) Gas Chromatography
- (4) Ion-Exchange Chromatography

**1. Paper Chromatography: (Liquid-liquid chromatography):** The principle of paper chromatography is based on the fact that solutes have the capacity to migrate through filter paper at different rates as a solution is drawn into strip of paper by capillary action.

In paper chromatography the dissolved substance is applied as a small spot about 2-3 cm. from the edge of a strip or square of filter paper and is allowed to dry. This strip is then suspended in a large close container where atmosphere is saturated with the solvent system whereas the end containing the sample is dipped into the mobile phase which has already been saturated with stationary phase. When the solvent front has reached the other end of the paper, the strip is removed and the zones are located by analytical methods.



Paper chromatography

However in descending order chromatography as shown in diagram given above the top end of the paper is dipped into the solvent and hung over an antisiphon rod while in ascending order method the lower end of paper is dipped directly into the solvent in the bottom in the container.

### **Precautions in Paper Chromatography**

1. Heavy metals and complexing agents should not be used as they convert the solute into a complex with different properties resulting in multiple spots.
2. Neutral salts should not be used as they disturb the cellulose water complex which will result in separation of liquid water on paper.

## R<sub>F</sub> Value

$R_F$  value is defined as the ratio of the velocity of movement of zone with that of developer front i.e.,

## 2. Column Chromatography (Liquid partition chromatography):

The liquid partition chromatography consists of a glass tube at the bottom to retain the packing and permit the liquid to pass through. Inert silica or celite is mixed with 5—30% of the stationary phase and packed into column as a slurry in mobile phase. Now the solvent is added until all the sample has passed from the column. The different fractions obtained are analysed by different suitable methods such as spectrophotometry, by changes in refractive index etc.

## Thin Layer Chromatography

This is a special type of column chromatography and now-a-days it is generally used for the quantitative determination of the radioactive substances, separation of amino acids, fatty acids, mineral oils, glycerides and phenols. It is also used for the separation of essential oils and fats.

## Experimental technique

In the experimental procedure, a glass plate is taken on which a solution consisting of the homogeneous mixture of the adsorbing agent with water is applied in a thin and uniform layer. Such pasted plates are then dried in open at 383 K for about 30-40 minutes. The sample to be analysed is spotted on the plate and the plate is placed vertically in the developing trough containing suitable solvent. After about 30-40 minutes the plate is taken out from the trough and dried in air. The substance becomes visible in ultra-violet light or turned to be visible by a colour reaction and hence the mixtures present in the same are detected.

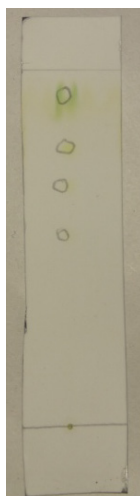
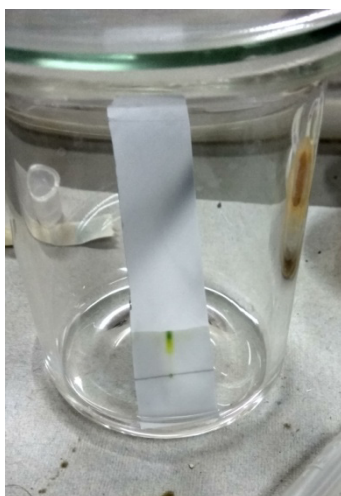
## 3. Gas Chromatography

Gas chromatography is now-a-days used as an important analytical tool for the separation of gases and volatile substances. Gas chromatography is defined as the method of separation in which gaseous or the vaporized components to be separated are distributed between two phases, a fixed stationary phase with large surface area and a moving gas phase. Gas-solid chromatography is defined as a method of separation in which fixed phase is a solid adsorbent.

Gas-liquid chromatography is defined as a method of separation which has a fixed phase, a liquid distributed in an inert support. This is also known as vapour-chromatography.

**4. Ion-Exchange Chromatography:** If a mixture of two or more different cations X, Y etc. is added into an ion exchange column and suppose X is held more firmly by the exchange resin than cation, Y all the Y present will flow out of the bottom of the column before any of X is liberated, provided that the column is long enough and other experimental factors are favourable for the particular separation. This separation technique is called **ion-exchange chromatography**.

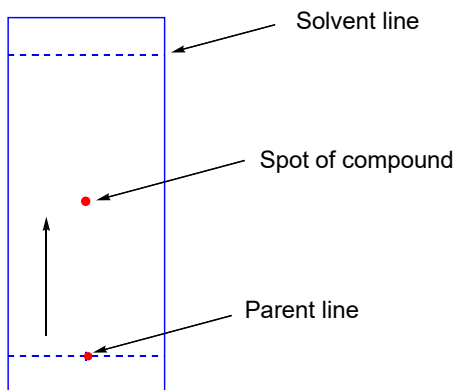
### Thin Layer Chromatography



#### Experimental Procedure:

1. Cut the 2X5 inch size TLC strip, carefully.
2. Draw straight line with lead pencil around 1 cm away from one end.
3. Crush the green leaves in a mortar.
4. Take fine semi-solid pest of leaves and make a small dot on prepared TLC plate on line.
5. Take 2 mL ethylacetate as mobile phase TLC flask/beaker.
6. Put the TLC plate in flask and ensure that end of plate should dip in to solvent (mobile phase).
7. Cover the TLC flask with glass cover.
8. Carefully notice the movement of solvent, when it reaches to around 1 cm below to top end of TLC plate, take out the plate.
9. Mark the solvent extreme line with pencil.
10. Notice the different spots of chlorophyll a, b and pigments under UV light.
11. Calculate Rf value for spots.

E-source: <https://www.youtube.com/watch?v=GtV4K8V9bks&t=4s>



$$R_f = \frac{2}{4} = 0.5$$

Post Lab:

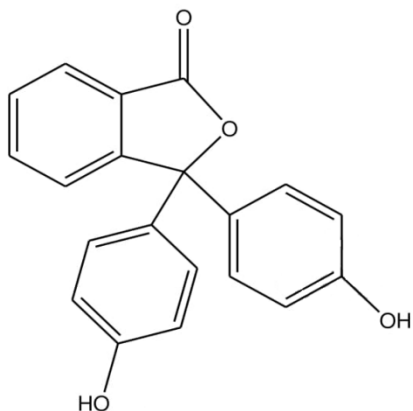
1. Name the substance of stationary phase of TLC.
2. How can we identify solvent system for TLC?
3. Why should we cover TLC flask during experiment?
4. What is capillary action?
5. What is role of UV light cabinet?

## Experiment 09

### 4. To find out saponification number of oil

**Chemical required:** Standard alc.  $\frac{N}{2}$  KOH solution, standard alc.  $\frac{N}{2}$  HCl solution, ethyl methyl ketone as solvent.

**Indicator:** Phenolphthalein



Phenolphthalein

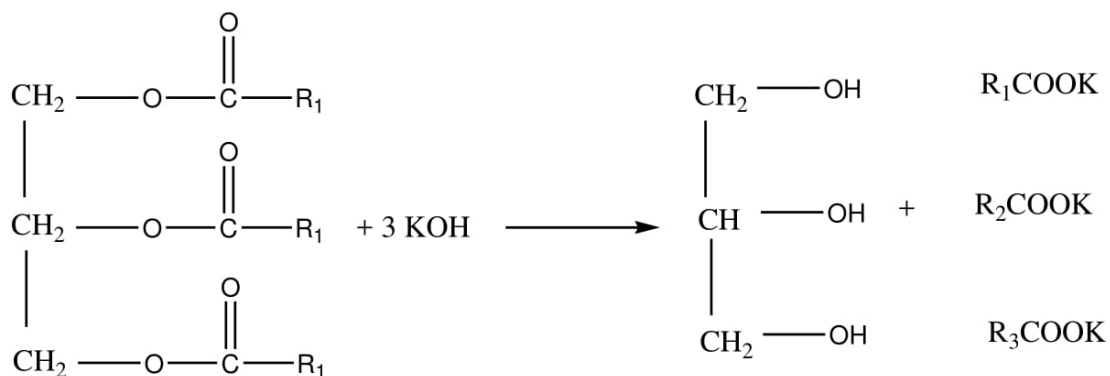
**End point:** Disappearance of pink colour

### Theory

**Saponification** is process of alkaline hydrolysis of oils (vegetable or animal) and fats giving soap.

**Saponification number** is defined as number of milligrams of KOH required to saponify 1 mg of a fatty oil.

For determination of saponification number of an oil, a known weight of oil is refluxed with a known excess of standard alc. KOH in a suitable solvent. During refluxing saponification of oil takes place.



(R1, R2, R3 may be same or different)

Amount of unreacted KOH left behind is determined by titration of mixture against standard HCl using phenolphthalein indicator. Disappearance of pink colour of solution marks the end point.



A blank experiment without oil is also performed. From the volume of HCl used in two titrations, saponification of oil can be obtained.

### Procedure

1. Take two 250 mL conical flasks and label them as I and II. Weigh accurately about 1-2 gm of given oil in a weighing bottle and transfer it to flask I. Weigh the empty bottle also.
2. Add 25 mL of ethyl methyl ketone and 25 mL of alc.  $\frac{N}{2}$  KOH solution to both flasks.
3. Put an air condenser in mouth of each bottle of each flask and keep them for refluxing on a water bath for 45 minutes.
4. Remove the flask from water bath. Wash the inner walls of each condenser with some distilled water into the respective flasks. Remove with condenser and cool both the flasks at room temperature.
5. Add 7-8 drops of phenolphthalein indicator to each flask. Titrate the solution of each flask against  $\frac{N}{2}$  HCl taken in a burette till pink colour just disappears. Note burette reading in both titrations.

### Observations

Weight of weighing bottle + oil sample =  $W_1$  g      51.5 g  
 Weight of empty weighing bottle =  $W_2$  g      50  
 Weight of oil taken for experiment =  $W_1 - W_2$  g      1.5 g  
 Volume of ethyl methyl ketone added to each flask = 25 mL  
 Volume of  $\frac{N}{2}$  alc. KOH added to each flask = 25 mL

**Table: Titration of unreacted KOH vs  $\frac{N}{2}$  HCl**

	Initial burette reading (mL)	Final Burette reading (mL)	Volume of HCl used (mL)
<b>For Flask I</b>	0	23	23
<b>For Flask II</b>	0	25	25

Volume of  $\frac{N}{2}$  HCl used by unreacted KOH in flask I = A mL      23 mL

Volume of  $\frac{N}{2}$  HCl used by unreacted KOH in flask II = B mL      25 mL

Vol. of  $\frac{N}{2}$  HCl is equivalent to volume of  $\frac{N}{2}$  KOH used for saponification of  $W_1 - W_2$  g oil = (B-A)mL      2 mL

Saponification number of oil =  $\frac{\text{Volume of KOH used} \times \text{normality of KOH} \times \text{Eq.weight of KOH}}{\text{Weight of oil sample}}$

$$= \frac{(B-A) \times \frac{1}{2} \times 56}{W_1 - W_2} = \frac{(B-A) \times 28}{W_1 - W_2} = X \quad \frac{2 \times 28}{1.5}$$

**Result:** Saponification number of given oil is X.

## Experiment 10

**Objective-Measure the turbidity given water samples.**

### **Theory of Water Turbidity Test:**

Water is said to be turbid when it is seen containing materials of suspension inside it. While turbidity may be defined as the measure of visible material in suspension in water, turbidity may be caused by algae or other organisms. It is generally caused by silt or clay. The amount and character of turbidity depends upon two things:

1. Type of soil over which flows
2. The velocity of flowing water

When water becomes stationary, the heavier and larger suspended particles settle down quickly and the lighter and finely divided particles settle very slowly and even takes months.

Ground water is less turbid because of low velocity of water. Turbidity may be helpful for controlling growth of organisms by not allowing proper sunlight to water which is necessary for their growth on the other hand it is harmful as the organisms are handling to the suspended particles. When water becomes stationary, the heavier and larger suspended particles settle down quickly and the lighter and finely divided particles settle very slowly and even takes months. Ground water is less turbid because of low velocity of water. Turbidity may be helpful for controlling growth of organisms by not allowing proper sunlight to water which is necessary for their growth on the other hand it is harmful as the organisms are handling to the suspended particles.

There are various units for the measurement of turbidity which are:

1. Standard turbidity unit [mg/lit or ppm]
2. Jackson turbidity unit [J.T.U]
3. Nephelometric turbidity unit [N.T.U]

A device called nephelometric turbidity measures the turbidity of water in N.T.U the intensity of light after passing through the water gives a measure of turbidity of water.

WHO guideline value:

WHO suggested a guideline value for turbidity as [N.T.U] for disinfection the turbidity of water should be less than 1 N.T.U.

### **Apparatus:**

W.H.O Nephelometric turbidity meter formazine solution of the sample by multiplying the scale reading by 0.9 N.T.U, 9 N.T.U, 99 N.T.U, test tubes and water samples.

### **Procedure of Turbidity Test:**



1. Switch on the power supply and check the the turbidimeter,
2. Put the pre-filled tube-1 and scale zero by using calibration switch.
3. A Standard formazine solution of N.T.U is placed on tubidimeter in the path of rays and scale is brought 9 n.t.u
4. The water samples is taken in a test and is placed in turbidimeter.
5. Use A Cell rise if the turbidity is more than 100 N.T.U and get the turbidity dilution factor.

<b>Observations and results</b>			
Sample	Reading	Factor	NTU
1	5	1000	5000
2	5.5	1000	5500
3	4.3	1000	4300
4	4.5	1000	4500

Q. From your results, are there any observable relationships between turbidity and apparent colour and between turbidity and true colour?

Q. What are Turbidity, Total Suspended Solids and Water Clarity?