

**COLLEGE OF THE BAHAMAS  
SCHOOL OF CHEMISTRY, ENVIRONMENTAL & LIFE  
SCIENCE**

**ORGANIC CHEMISTRY  
CHEMISTRY 230**

**PRACTICAL HANDBOOK**

SPRING 2015

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Written by  
K. S. Massie

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**ORGANIC CHEMISTRY — CHEMISTRY 230**  
**PRACTICAL HANDBOOK**  
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# INTRODUCTION

**Welcome to the laboratory component of Organic Chemistry CHEM230.** The purpose of the laboratory component is to provide you, the student, with the opportunity to apply the concepts that you meet in the theory component as well as to build your practical skills. This manual contains details of the practical work you will be doing during this course.

In order to obtain maximum benefit from laboratory activities, you are expected to do pre-lab activities. These include reading the instructions preparing a plan of action and constructing a data table for the chemicals to be used. The laboratory session should be used to conduct the experiment. You should already know what steps to follow.

## KEYS TO SUCCESSFUL ORGANIC LABORATORY EXPERIENCE:

1. Read through the details for each lab at least 2 days in advance.
2. Make a plan of action, along with your lab partner.
3. Prepare your pre-lab work **BEFORE** your assigned lab day.
4. Conduct your lab in compliance with laboratory safety rules and supervisors instructions.
5. Record your observations and data during the lab session.
6. If you are uncertain about any step be sure to seek guidance from the instructor and/or technician.

## REQUIRED MATERIALS FOR CHEM230 :

- laboratory manual
- a hardcover notebook
- Laboratory coat
- Safety goggles or face shield
- Closed shoes

## NOTES:

1. Attendance at practicals is compulsory. If you do not attend, your mark will automatically be zero, unless you present a medical certificate. (Medical certificates may be obtained from the campus nurse at no charge.)
2. Make optimum use of your laboratory time. Some of the questions/follow-up work can be done later.
3. Check the materials list for each lab. Use this to make sure that you have the required apparatus and chemicals before you start your experiment. Let your instructor know if anything is missing.
4. Write data directly into your hard cover laboratory notebook. Do not record data on loose paper. Your lab notebook should include a diagram showing how apparatus was set up, details on any changes made to the method, your results and observations.

## **SAFETY**

A chemistry laboratory can and should be a safe place in which to work, provided that the correct precautions and safety procedures are followed.

The experiments you will be doing have been chosen in part because they involve little risk, if the instructions are followed carefully in the correct order. **READ THE INSTRUCTIONS!**

If an accident does occur, **DO NOT PANIC**. A minor accident may easily be turned into a serious one by over-reacting. If an accident happens to another student, tell them what to do, help them if necessary, and inform the lecturer. Clean up any spills and/or breakages thoroughly. Ask the supervisor if in doubt as to what to do.

### **Safety Equipment.**

Make careful note of the positions of the safety shower/eye-bath, fire extinguishers and fume cupboards, and know how to use them.

### **Eye protection.**

Students must wear safety glasses at all times when in the laboratory (over your eyes, not up on your forehead/hair!). Each student must come to class with his or her own pair – available from Chapter One. It is not advisable to wear contact lenses in the laboratory.

### **Clothing.**

Come to the laboratory in sensible clothing. Long flowing clothing is dangerous. Shorts and open shoes/sandals offer less protection than pants and regular shoes with non-slippery soles.. High heels are dangerous. Keep long hair tied back.

## **Writing Laboratory Reports**

You will be working in groups of two for most of your experiments. This allows you to share equipment and the workload. However each student is expected to submit his/her own work. In cases where lab reports are to be submitted, you must work separately using the group results, but giving your own discussion and conclusions. In cases where you obtain data from print or online sources, you must give appropriate credit.

### ***Components of a Short Form Laboratory Report***

1. Title and date – Name of the experiment, date it was done , name of your lab partner
2. Purpose/Goal/Objective- What scientific principle(s) is/are being examined/demonstrated?
3. Method/Procedure- What steps were involved ? ( You may simply refer to the relevant portion of the lab manual, noting any changes made to the steps.)
4. Observations – What did you SEE, hear, smell etc .?
5. Results – What data did you obtain ? /numerical information
6. Calculations – Show the steps involved. Show all units. Discuss errors.
7. Conclusion – Did you fulfil the purpose? What did you achieve?

Your instructor will provide you with additional guidelines regarding the laboratory report.

### **ORGANIC CHEMISTRY LABORATORY SAFETY RULES**

The Organic Chemistry Laboratory can be a place of discovery and of danger. These rules are intended to reduce the chances of injury and other hazards whilst allowing you to gain valuable practical skills.

- 1) Always wait for a supervisor before conducting any experiments.
- 2) Protect your eyes by wearing safety glasses whenever you are in the lab and ANY laboratory work is being done.
- 3) Wear a lab coat, safety eye wear and closed shoes to protect yourself and reduce the chances of exposing others to harmful chemicals.
- 4) Avoid loose or dangling clothing, hair or jewellery, these may knock over apparatus or become contaminated by chemicals, or catch fire.
- 5) Always follow your supervisor's instructions carefully.
- 6) Treat chemicals as dangerous. Handle all with care.
- 7) Never smell chemicals unless specifically instructed to do so by your supervisor ( using the wafting technique)
- 8) Protect your skin from chemical burns. Wash areas that are exposed to chemicals.
- 9) Unless otherwise instructed, most of your work should be done in the fume cupboard.
- 10) Avoid ingesting harmful substances .Never eat or drink in the laboratory.
- 11) There are several safety items in the lab. These include an emergency shower, eyewash stations, first aid kit and fire extinguishers. Locate each of these and familiarise yourself with instructions. Make sure that you know when and how to use them.
- 12) Stand when you are conducting experiments. You may need to exit quickly.
- 13) Keep your workbench and the floor free of clutter to reduce the risk of accidents.
- 14) Never heat substances in sealed test tubes or other containers. They may explode.
- 15) Clean up spills and broken glass-ware immediately. They can be dangerous.

**Chemistry Etiquette:** Please assist the department by also observing the following:

- 1) Do not throw solid matter (e.g. filter paper) into the sinks. They easily get blocked up. Put waste materials in designated areas.
- 2) Report all breakages to the supervisor.
- 3) Avoid contamination of reagent containers:
  - i. take out just what you need to your workbench
  - ii. do not return unused chemicals to reagent containers. This may cause contamination.
- 4) Leave your bench clean, dry and free of clutter.
- 5) Return all apparatus (after cleaning) to your assigned equipment storage space.

# PRACTICAL NUMBER 1

## EXTRACTION: SEPARATION AND ISOLATION TECHNIQUES

### INTRODUCTION

In this experiment you will separate the components of a mixture consisting of an acid, a base and a neutral substance by extraction. Extraction is a common technique used to separate a desired organic product from a reaction mixture or to isolate an organic substance from its natural source.

Extraction involves the use of two immiscible solvents which form two layers upon standing. Usually the solvents are water and an organic solvent. The desired substance (or a derivative of it) should be far more soluble in one solvent than in the other. When a mixture of substances is shaken with the two solvents, most of the desired substance (or its derivative) should dissolve in one of the solvents.

Although organic compounds are generally more soluble in an organic solvent than in water, many, especially those which can form hydrogen bonds, are partially soluble in water. They distribute themselves between the aqueous solvent and the organic solvent in proportion to their relative solubilities (*S*) in the two solvents.

The ratio of the concentrations (expressed in mass per unit volume of solvent e.g. g cm<sup>-3</sup>) of a substance in the two solvents *at equilibrium* is an equilibrium constant, called its **distribution coefficient,  $K_D$** :

$$K_D = \frac{S_o}{S_w} = \frac{C_o}{C_w}$$

where the subscript *o* stands for *oil* and *w* for *water*.

For example, suppose the solubility of compound A in ether and water is 0.30 g/100 cm<sup>3</sup>

and 0.060 g/100 cm<sup>3</sup> respectively, then  $K_D = \frac{0.30}{0.060} = 5.0$

In general, performing several extractions using smaller volumes of solvent is more efficient than performing a single extraction with a larger volume of solvent (see post lab question 1).

The extraction solvent

- must be immiscible or sparingly soluble in the solvent from which the desired substance is to be extracted.
- must readily dissolve the substance to be extracted and should extract only the desired substance or as small an amount as possible of any other substance present.
- should not react chemically with the solute.
- should have a low boiling point so it can be easily evaporated or distilled off from the desired solute after extraction. Common organic solvents used in extraction include ethoxyethane (diethyl ether or ether), petroleum ether<sup>1</sup> (a mixture of low-boiling alkanes), dichloromethane, trichloromethane (chloroform), and tetrachloromethane (carbon tetrachloride), ethyl ethanoate (ethyl acetate).

### CAUTION :

Chlorinated hydrocarbons are not often used in this lab since their vapours are toxic and some are carcinogenic. These compounds may be absorbed through the skin, causing harm. Ethoxyethane (diethyl ether or ether) is highly flammable and, upon standing in air, its solutions may develop dangerous concentrations of explosive peroxides. **No flames must be in the lab when working with ether.** Furthermore, ether is slightly water-soluble (about 7g/100cm<sup>3</sup>). However, because most organic

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<sup>1</sup> Often known as "pet ether".



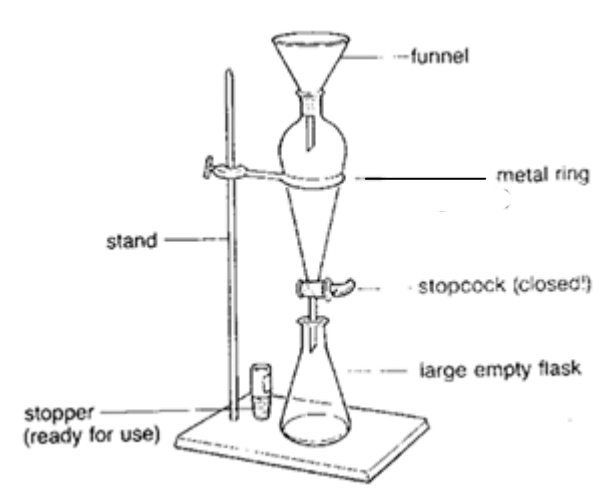
compounds are highly soluble in it and because of its low boiling point (35°C), ether is frequently used.

#### SKILLS:

1. Use of digital balance
2. Use of separatory funnel
3. Calculation of distribution coefficient
4. Extraction of components of a mixture
5. Melting point determination
6. Vacuum filtration

#### *Use of the separatory funnel*

Extractions are usually performed with a separatory funnel. **Your instructor will demonstrate the proper use of a separatory funnel.**



#### **Method**

You will be provided with a mixture containing equal masses of m-nitroaniline (a base), benzoic acid (an acid), and naphthalene (a neutral substance) will be separated into its components by extraction. Weigh out 1.5-2.0 g of the mixture and record the mass. Dissolve the mixture in 15 cm<sup>3</sup> of solvent (\_\_\_\_\_) or diethyl ether. Pour the solution into a 125cm<sup>3</sup> separatory funnel. Extract three times with 15 cm<sup>3</sup> portions of 3 M HCl. Finally extract with 5 cm<sup>3</sup> water to remove excess HCl that may dissolve in the ether layer. Combine the three acid extracts with the water extract and set them aside.

Extract the remaining \_\_\_\_\_ or ether solution three times with 15cm<sup>3</sup> portions of 10% aqueous NaOH and once with 5cm<sup>3</sup> of water. Combine the alkaline and water extracts and set them aside.

Pour out the remaining \_\_\_\_\_ or ether solution through the top of the separatory funnel into a small Erlenmeyer flask. Add enough anhydrous calcium chloride to cover the bottom of the small Erlenmeyer flask, and swirl the mixture occasionally for 15min. Decant the \_\_\_\_\_/ether into a small beaker of known weight. Rinse the flask contents with a small amount (3cm<sup>3</sup>) of ether, and add the rinsing to the beaker. Place the beaker in the hood to allow the \_\_\_\_\_/ether to evaporate. Weigh the residue(X) and determine its melting point. Transfer the compound to a test tube labelled with the letter X and with your name and date. Cork it and submit it to your instructor.

Neutralize the combined acid extracts by adding 10% aqueous sodium hydroxide until the solution is alkaline to litmus paper. This is done by dipping a clean glass rod into the solution and then touching it to the litmus paper. Extract the alkaline solution twice with 15 cm<sup>3</sup> portions of ether. Combine both ether extracts. Add anhydrous calcium chloride, and swirl as described above. Decant the ether solution into a small beaker of known weight, rinse as described above, and place the beaker in the hood to allow the ether to evaporate. Weigh the residue (Y) and determine its melting point. Transfer the compound to a test tube labelled Y and with your name and date. Cork it and submit it to your instructor.

Neutralize the combined alkaline extracts by adding concentrated hydrochloric acid drop by drop until the solution is acid to litmus paper. Again, dip a clean glass rod in the solution and touch it to the litmus paper. The solution may be kept cool in an ice bath during neutralization. Recover the solid (Z) by vacuum filtration using a Buchner or Hirsch funnel, or by extraction with ether as described above. Allow the product to air dry, weigh it, and determine its melting point. Transfer the compound to a test tube labelled with the letter Z and with your name and date. Cork it and submit it to your instructor. (Note that it is likely that some of the ether evaporated off during the extractions and may require replenishment.)

Calculate the percent recovery of each component.

### **Waste Disposal**

Neutralize the basic and acidic aqueous filtrates with dilute hydrochloric acid and 10% aqueous sodium hydroxide, respectively, and pour into the aqueous waste container provided by your instructor.

### **Pre lab questions**

- 1) Draw the structures of the three components of the mixture.
- 2) a) Write two equations (using structural formulae) for the reactions used in this experiment to separate and recover benzoic acid.  
b) Write two equations (using structural formulae) for the reactions used in this experiment to separate and recover m-nitroaniline.
- 3) Complete the table for the stated solvents.

<i>Solvent</i>	<i>Density/ gcm<sup>-3</sup></i>
Diethyl ether	
Dichloromethane (methylene chloride)	
Tetrachloromethane	
Ethyl ethanoate ( acetate)	

- 4) Complete the table.

<i>Substance</i>	<i>Melting Point/°C</i>
benzoic acid	
m-nitroaniline	
naphthalene	

- 5) Construct a flow chart to represent the separation of the three components of the mixture.

### **Post lab Questions**

- 6) a) The distribution coefficient  $K_D$  for a compound, A, between diethyl ether and water is 5 i.e.

$\frac{C_o}{C_w} = 5$ , calculate the mass of A that is removed from a solution containing 50°mg of A in 50°cm<sup>3</sup> of

water by extracting with 10°cm<sup>3</sup> of ether. (Hint. Let x°mg be the mass of A extracted into the ether layer).

- 7) b) Show that extraction twice with 50 cm<sup>3</sup> of diethyl ether instead of once with 100 cm<sup>3</sup> of diethyl ether would remove a larger mass of A from the water.
- 8) List three criteria that should be considered when selecting a solvent for extraction.
- 9) State two *advantages* and two *disadvantages* of using diethyl ether as an extraction solvent.
- 10) What is the practical advantage of using an organic solvent which is denser than water in an extraction?
- 11) Why must the stopper of the separatory funnel be removed before the liquid can be withdrawn through the stopcock?
- 12) a) Write two equations for the reactions used in this experiment to separate and recover benzoic acid.  
b) Write two equations for the reactions used in this experiment to separate and recover *m*-nitroaniline.
- 13) Which of the components in the mixture was
  - a) X?
  - b) Y?
  - c) Z?
- 14) Use the Henderson Hasselbalch equation to find
  - a) the pH at which an aqueous solution would contain 10 times more benzoic acid molecules than benzoate ions.
  - b) the pH at which an aqueous solution would contain 100 times more benzoic acid molecules than benzoate ions.
- 15) Phenol forms salts when treated with inorganic bases. However, phenol is a weaker acid than ethanoic acid. Aqueous sodium bicarbonate is usually alkaline enough to convert a carboxylic acid to its salt, but not alkaline enough to convert phenol to its sodium salt. Use this information to suggest an extraction procedure to separate phenol and ethanoic acid.

## Sources

Experiments for Introduction to Organic Chemistry, Bettelheim and Landesberg, Saunders College Publishing, 1997

Laboratory Manual, Organic Chemistry .. A Short Course Hart, Craine, Hart, Vinod 12<sup>th</sup> Ed, Houghton Mifflin, 2007

## PRACTICAL NUMBER 2

# RECRYSTALLISATION: A PURIFICATION TECHNIQUE FOR CRYSTALLINE ORGANIC COMPOUNDS

In this experiment you will purify an impure sample of acetanilide by recrystallisation.

### Introduction

Recrystallisation involves dissolving an impure solid in a solvent and allowing the solid to precipitate from the solution. The term **recrystallisation** is used to describe the technique because the solid recrystallises from its solution.

Recrystallisation involves a number of steps:

- 1) selecting a suitable solvent.
- 2) dissolving the impure solid in a **minimum volume** of solvent at or near its boiling point.
- 3) filtering the hot solution to remove insoluble impurities.
- 4) allowing the solution to cool slowly to allow crystallization of the solid.
- 5) separating the crystals from the **mother liquor** by filtration.
- 6) washing the crystals with a little cold solvent to remove the mother liquor.
- 7) drying the crystals.

### Choosing a solvent

The choice of a suitable solvent is most important to accomplish good results. Certain criteria must be fulfilled:

- 1) The product and the impurities should show a large difference in solubility.
- 2) The desired compound should be much more soluble in the hot solvent than in the cold solvent.
- 3) The solvent should readily dissolve impurities at a low temperature or not dissolve them at all.
- 4) The solvent must not react chemically with the desired product.
- 5) The solvent should have a low enough boiling point so that it can be readily removed from the crystallized product in the drying step.
- 6) If a suitable single solvent cannot be found, a mixture of two solvents may be used. For example, the compound *p*-dibromobenzene is very soluble in hot and cold ethanol but insoluble in hot and cold water. In this case, neither ethanol alone nor water alone is suitable. However a mixture of water and ethanol can be used (*see experiment 2 below*).

It is advisable to use the **smallest possible** volume of hot solvent to minimize the amount of material retained in the mother liquors. About 3-5% more solvent than the absolute minimum required is used so that the hot solution will be not quite saturated. This helps to prevent premature crystal formation.

Traces of coloured impurities may be removed by adding a **small** quantity of finely divided charcoal to the mixture before heating, or to the **warm** solution before filtering it. Do **not** add decolourising charcoal to a **hot** solution as it will cause rapid boil-over. Excess charcoal may also absorb significant amounts of the desired substance.

If crystals do not form spontaneously when the hot solution is cooled, crystallisation may be induced by scratching the walls of the vessel beneath the surface of the solution with a stirring rod or by *seeding* the cold solution with one or two crystals of the substance being purified.

If the dissolution process is slow, a mixture of the solid and solvent can be *refluxed* in order to obtain a solution of the material to be purified. Your instructor will explain how refluxing achieves heating a substance in a solvent for a long time without boiling away the solvent.

## METHOD

### *Experiment 1: Recrystallisation of acetanilide using a single solvent*

- 1) Determine the melting point of the impure acetanilide provided.
- 2) Weigh out 1.5-2.0 g of impure acetanilide. Record the mass.
- 3) Place the sample in a 100-mL round-bottomed flask, connect this flask to a reflux condenser, and start a slow stream through the jacket of the condenser. Add 35 cm<sup>3</sup> of water to the flask through the top of the condenser, and bring the water to a boil by heating with a mantle. Adjust the mantle temperature so that the water refluxes steadily. Continue refluxing until no more solid appears to dissolve. Remove the heat source, and allow the flask to cool a few moments after reflux stops to avoid boil-over. Remove the condenser momentarily, and add a small amount (about 0.2 g) of decolorizing charcoal to the contents of the flask. Replace the condenser and reflux for a further five minutes.
- 4) Filter the hot mixture through fast-flow fluted filter paper. (Pour 15-20 cm<sup>3</sup> of boiling water through the funnel to warm it and to wet the filter paper before pouring the solution. Discard the water).
- 5) As the filtrate cools, crystals will begin to form immediately. Chill the vessel containing the filtrate in an ice bath to complete the crystallization.
- 6) Set up the vacuum-filtration apparatus and pour 15-20 cm<sup>3</sup> of cold water through the funnel to wet the filter paper. Discard this water. Collect the crystals by vacuum filtration. Rinse the crystals (with the vacuum on) with a few cm<sup>3</sup> of ice-cold water. Use a clean spatula to press the crystals as dry as possible on the funnel. Transfer the crystals to a piece of clean, white paper, spread them in a thin layer, and cover the crystals with a watch glass. Store them for drying until the next laboratory period.
- 7) Weigh the dried product and determine its melting point. Calculate the percentage recovery.
- 8) Submit the purified acetanilide, labelled with your name and date, to your instructor.

### SKILLS:

1. Dissolution of an organic solid
2. Hot filtration
3. Crystal washing
4. Mixed solvent recrystallization

### *Waste Disposal*

Place organic solids recovered on filter paper in a solid-waste container provided by your instructor. Pour the aqueous mother liquor solutions into an aqueous-waste bottle provided by your instructor.

### *Experiment 2: Recrystallisation of p-dibromobenzene using a mixed solvent*

Since *p*-dibromobenzene is soluble in cold and hot ethanol and insoluble in hot and cold water, neither of these solvents, by itself, can be used for its recrystallisation. However, a mixture of the two solvents, which are miscible, can be used since the compound is soluble in the hot mixture but relatively insoluble in the cold mixture.

- 1) Determine the melting point of the impure *p*-dibromobenzene provided.
- 2) Weigh out 1-1.5 g of impure *p*-dibromobenzene and record its mass.
- 3) Transfer the solid to a 25 cm<sup>3</sup> Erlenmeyer flask, add 5 cm<sup>3</sup> of ethanol, and heat the mixture on a **steam bath**. Swirl the solution until the solid dissolves. If any undissolved solid remains after several minutes of heating, filter the hot solution using fluted filter paper, a small stemless funnel, and a small

Erlenmeyer flask. If, however, the solid dissolves completely, add water in 0.5 cm<sup>3</sup> portions to the hot solution until it becomes cloudy. Then add a little ethanol (0.5 cm<sup>3</sup> or less) until the cloudiness just disappears. If filtration is necessary, treat the hot filtrate with water and ethanol as just described.

- 4) Chill the flask in an ice bath for several minutes, and collect the crystals by vacuum filtration.
- 5) Rinse the crystals (with the vacuum off) with a few cm<sup>3</sup> of an ice-cold mixture containing equal volumes of ethanol and water. Carefully stir the contents on the funnel for just a few seconds then turn the vacuum on. Repeat this operation until a white product is obtained or until most of the orange impurity is removed. Allow the pure crystals to air-dry, with the vacuum on, for a few minutes and then transfer them to a clean sheet of paper for final drying.
- 6) Weigh the dry product and determine its melting point. Find the percent recovery.
- 7) Submit your product, labelled with your name and date, to your instructor.

**Note that *p*-dibromobenzene sublimes if stored at temperatures above 20 C.**

***Sources:***

Experiments for Introduction to Organic Chemistry by Bettelheim and Landesberg, Saunders College Publishing, 1997

Laboratory Manual, Organic Chemistry, A Short Course by Hart.H , Craine.L, Hart,D and Vinod.T, 12<sup>th</sup> Ed, 2007, Houghton Mifflin Company.



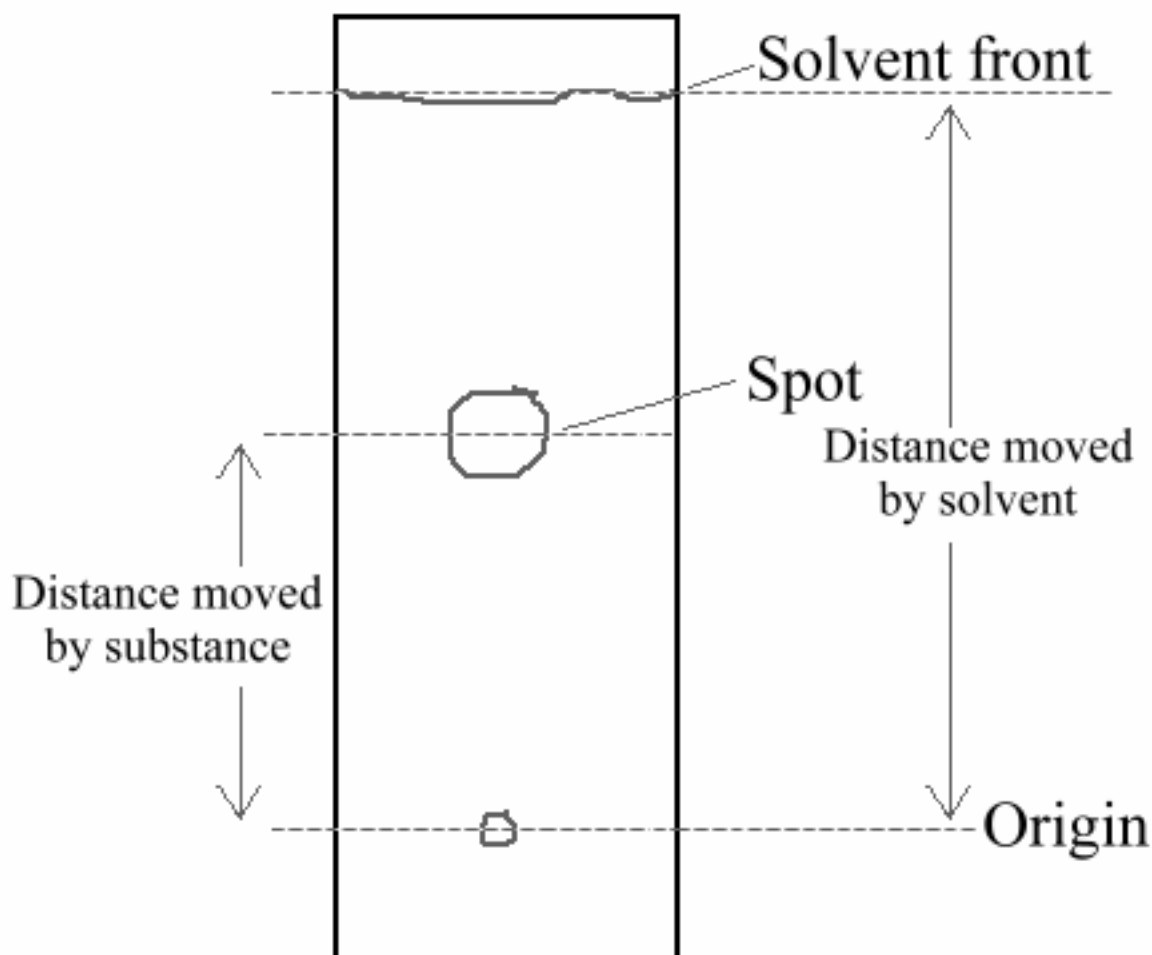
## PRACTICAL NUMBER 3: THIN LAYER CHROMATOGRAPHY

Thin Layer Chromatography (TLC) is a technique used to separate the components of organic mixtures and to identify organic compounds. It involves applying a minute amount of a solution of the sample to be analyzed to a thin layer of a solid adsorbent (usually silica gel or aluminium oxide) as a spot. The solid adsorbent is spread as a thin layer on a glass plate or sheet of aluminium foil or plastic. This layer of adsorbent constitutes the **stationary phase**. The plate is then placed in a container containing a solvent, called the **mobile phase**, which travels up the adsorbent by capillary action and pulls the components of the organic sample along with it. The solvent is called an “eluant” or “eluting solvent”. Different components in the sample move at different rates based on differences in sample-adsorbent and sample-solvent attraction. The component which has the greatest attraction for the solvent will move the furthest up the plate whereas the component with the greatest attraction for the adsorbent will move the least up the plate. The separation of the components as they move up the plate called “development” of the chromatogram.

A chromatogram is recorded by its  $R_f$  (retention factor) value which is given by:

$$R_f = \frac{\text{distance moved by spot}}{\text{distance moved by solvent}}$$

as shown in the diagram below:





## Pre-Lab Assignment

- 1) a) Draw the structures of benzoic acid, benzanilide and acetanilide. These are possible components of a mixture which you will separate and give the dipole moment of each substance..
  - b) Which of the three substances is
    - i) the most polar?
    - ii) the least polar
- 2) Complete the table for the eluting solvents given:

<i>Solvent</i>	<i>Dipole moment/D</i>
Methanol	
Acetone	
Hexane	
Acetonitrile	
Ethyl acetate	
Methylcyclohexane	
Ethanol	
Pyridine	
Dichloromethane	

## Method

You will be provided with a small glass jar with a cover to be used as the developing jar.

You will also be provided with commercially prepared chromatography plates. Cut a rectangular piece about ½ inch wide and almost the height of the jar. Using a pencil, make a straight line approximately 0.5 cm from the bottom of the narrow end of the plate- this line is called the **origin**. Make two vertical lines approximately 0.5 cm from each of the longer edges of the plate.

Dissolve a tiny quantity of the sample in approximately 0.25 cm<sup>3</sup> ethyl acetate. Spot a tiny volume of the sample to the origin line on the plate, using a fine capillary tube. Allow to dry, then add another spot over it and allow to dry. Repeat a few times until sufficient sample is applied to the plate. A small spot obtained from multiple applications is better than a larger spot obtained from fewer applications.

Add 2-3 cm<sup>3</sup> of the mobile phase to the chromatography jar, close it and allow the chamber to become saturated with the solvent vapour. This will prevent the solvent from evaporating as it rises up the plate. Using forceps, carefully insert the spotted plate into the jar. The origin must be **above** the level of the mobile phase. Allow the solvent to travel until the solvent front is approximately 0.5 cm from the top edge of the plate. Do not disturb the jar until the elution is complete. When the solvent front reaches the desired level, carefully remove the plate and make a horizontal mark to indicate the solvent front. Allow the plate to dry in a fume hood.

Visualize the chromatogram by viewing it under an ultraviolet lamp. The adsorbent contains a fluorescent indicator which reveals the spots under ultra violet light. A pure substance will give one spot. A mixture will give more than one spot as each component will give a spot. Calculate the R<sub>f</sub> for each spot.

### SKILLS

1. Thin layer chromatography