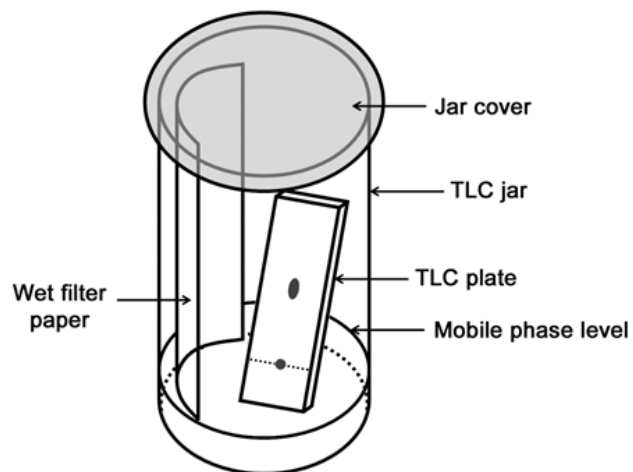


# Elution



## Elution

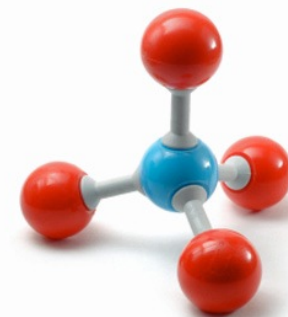
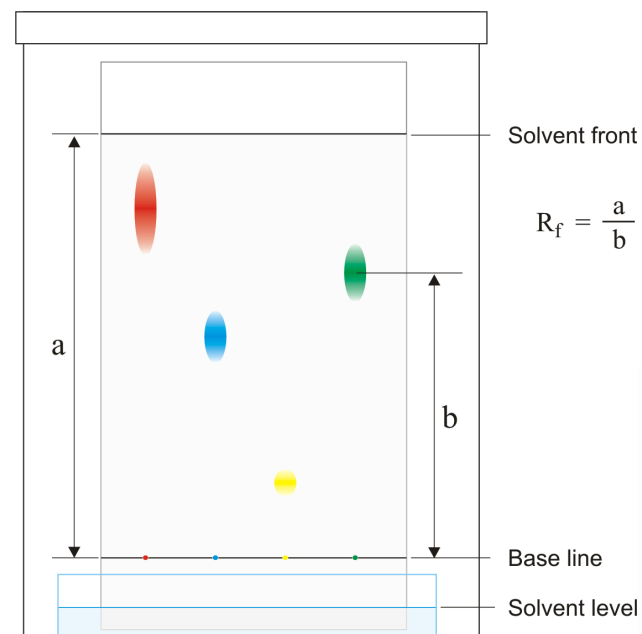
- ▶ Separating Matter
- ▶ Chromatography
  - ▶ Mobile & Stationary
  - ▶ Solvent Effects
- ▶ Applications
  - ▶ Analytic
  - ▶ Preparatory



## ▶ The Experiment

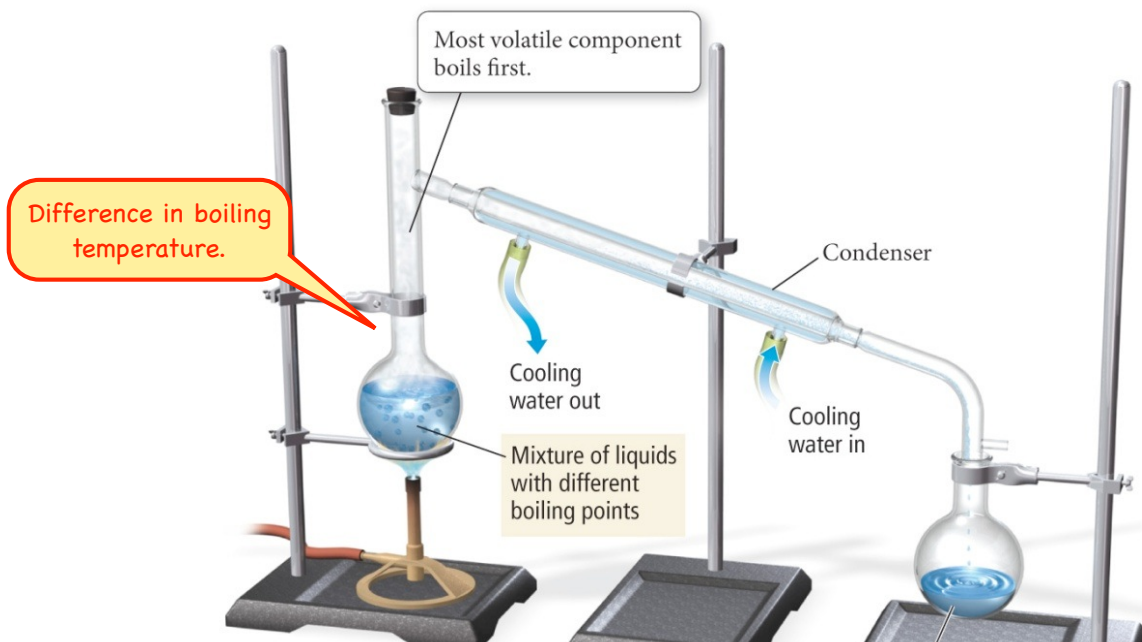
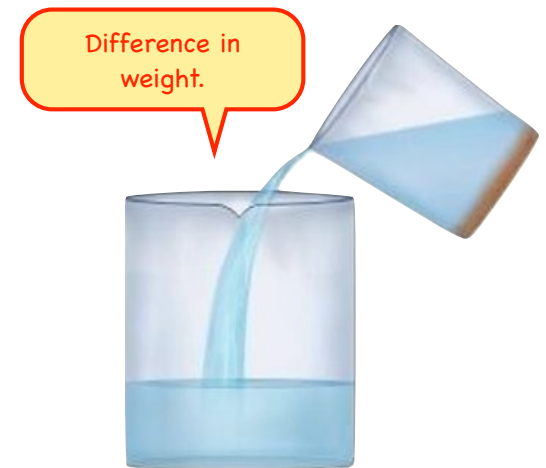
- ▶ Part A
  - ▶ Use TLC to Separate Compounds
- ▶ Part B
  - ▶ Determine Elution Solvents for Separation

## ▶ For Next Week



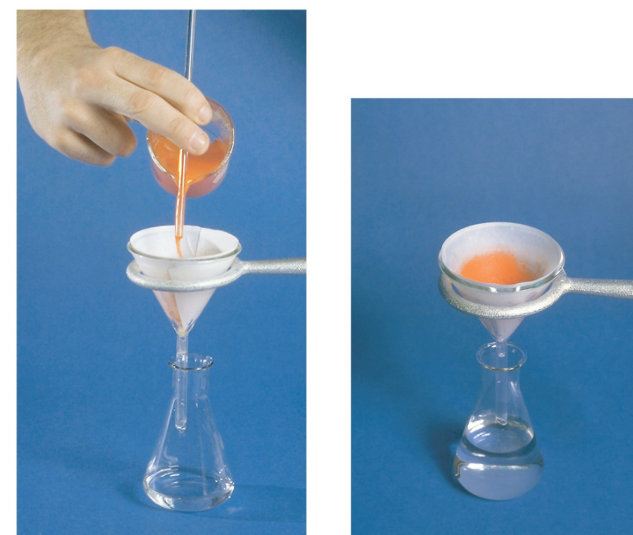
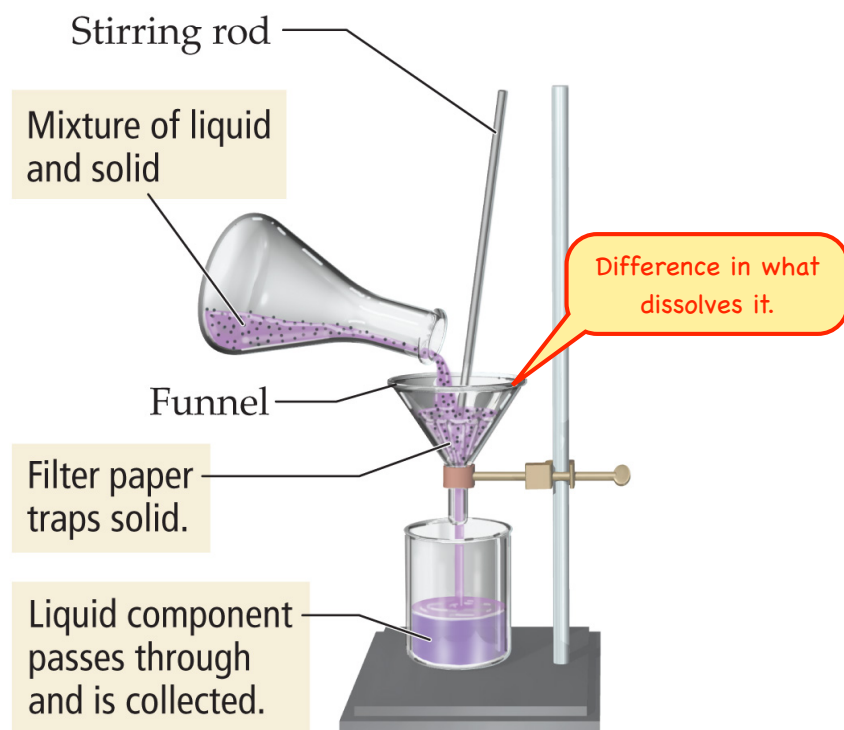
# Separating Mixtures

- ▶ Chemists spend a lot of time, separating mixture and isolating pure substances.
- ▶ We take advantage the different physical properties of the pure substances in a mixture to separate those pure substances.
  - ▶ **Decanting:** A mixture of sand and water can be separated by decanting—carefully pouring off the water into another container.
  - ▶ **Distillation:** A mixture of liquids can usually be separated by distillation, a process in which the mixture is heated to boil off the more volatile (lower boiling) liquid. The volatile liquid is then re-condensed in a condenser and collected in a separate flask.



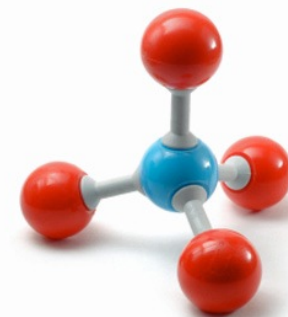
# Separating Mixtures

- ▶ Chemists spend a lot of time, separating mixture and isolating pure substances.
- ▶ We take advantage the different physical properties of the pure substances in a mixture to separate those pure substances.
- ▶ **Filtration:** A mixture of an insoluble solid and a liquid can be separated by filtration—process in which the mixture is poured through filter paper in a funnel. Most coffee machines rely on this process to separate the mixture of coffee beans and coffee beverage.



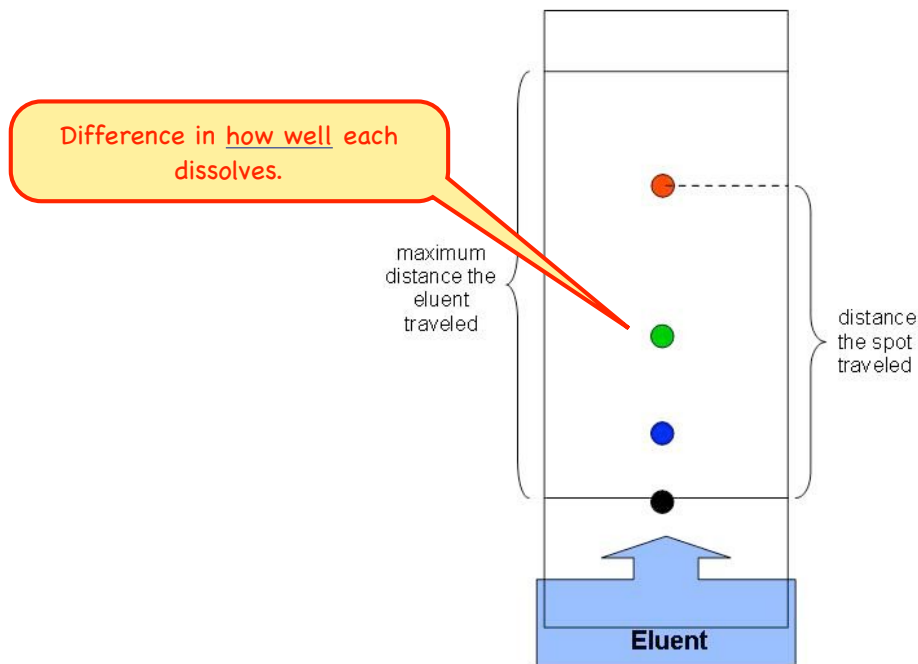
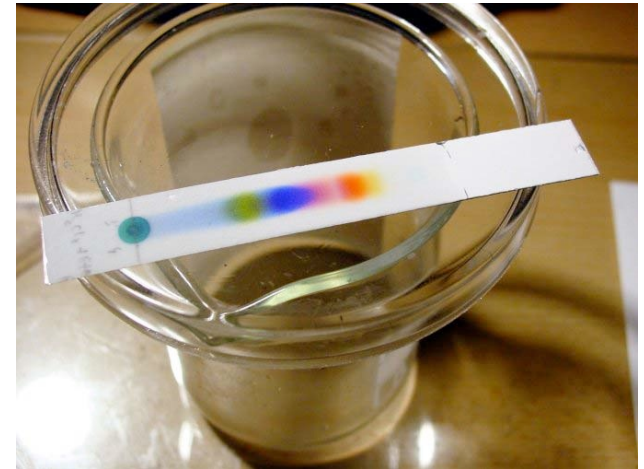
# Chromatography

- ▶ **Chromatography** is a technique for both identifying (analytic chromatography) substances and purifying them (preparatory chromatography).
- ▶ Chromatography involves placing a sample on silica gel and eluting it (running a solvent over it) to push the substance across the gel.
  - ▶ As the solvent runs across the gel, it will dissolve some of the substances and they will move with the solvent.
  - ▶ Substances that are on the edge of solubility, will fall back to the gel, then get absorbed again by more solvent.
  - ▶ So some will move faster and others slower across the gel.
- ▶ Moving substances over a solid phase, by partially carrying them in a mobile liquid phase, is **elution**.
- ▶ **Eluents** are solvents used to elute substances across a solid phase.



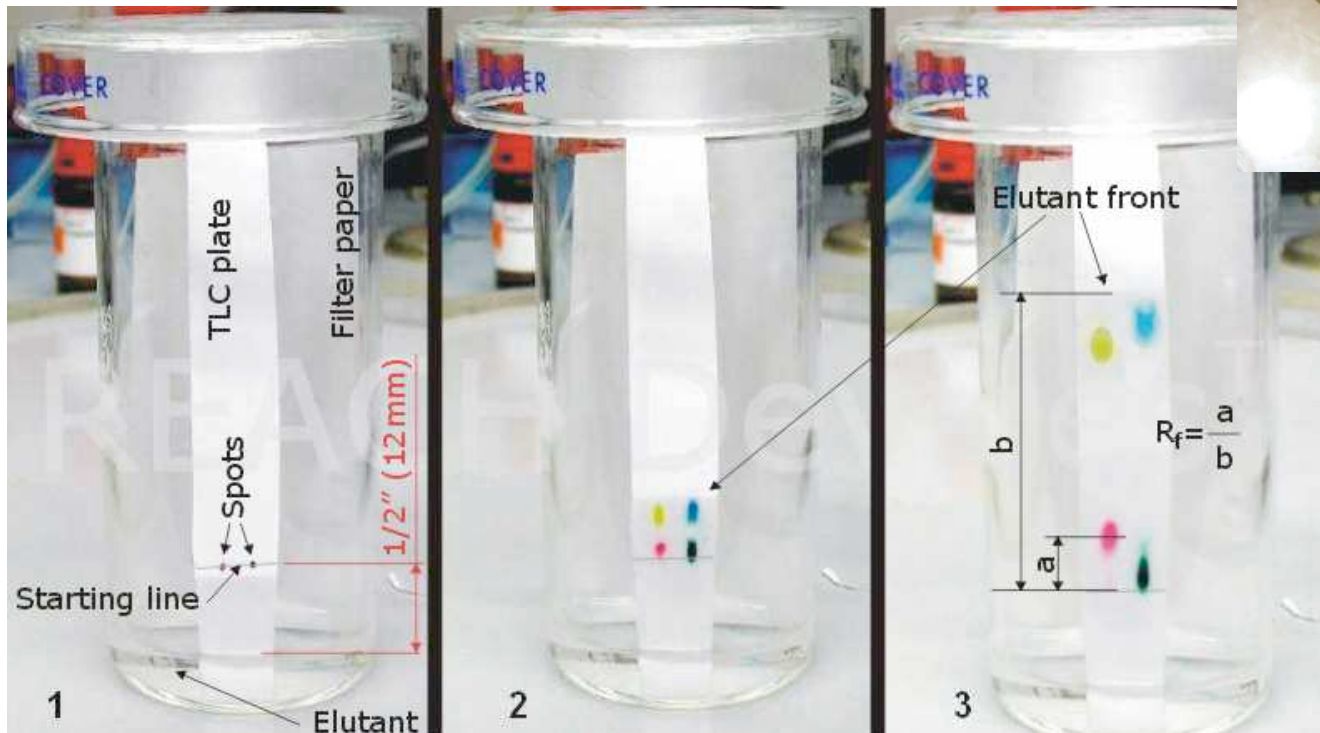
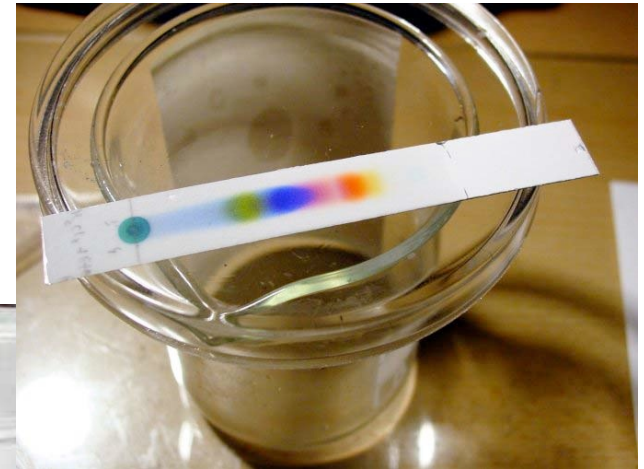
# Chromatography

- ▶ Chromatography separates substances when both are soluble, but one is *more* soluble than the other.
- ▶ **Thin Layer Chromatography:** runs samples up a silica plate coated with silica gel.



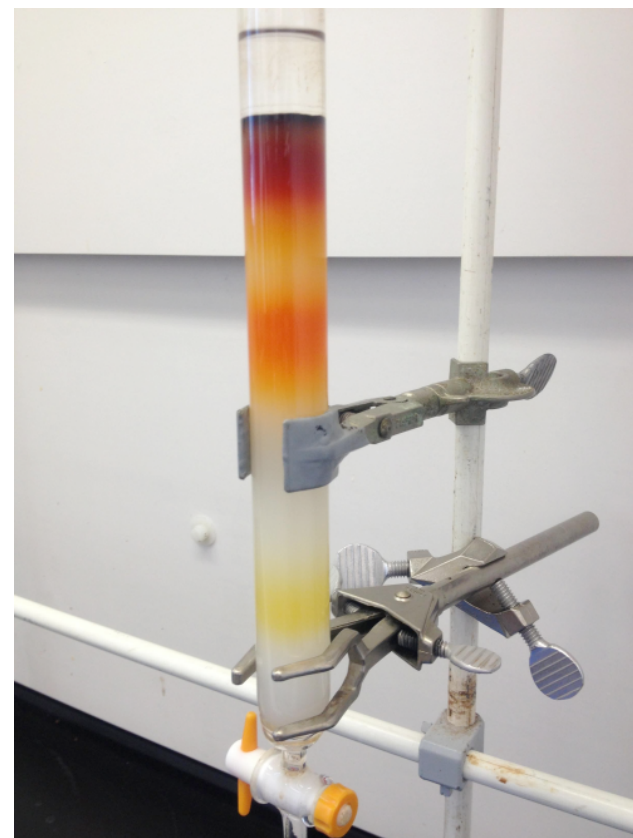
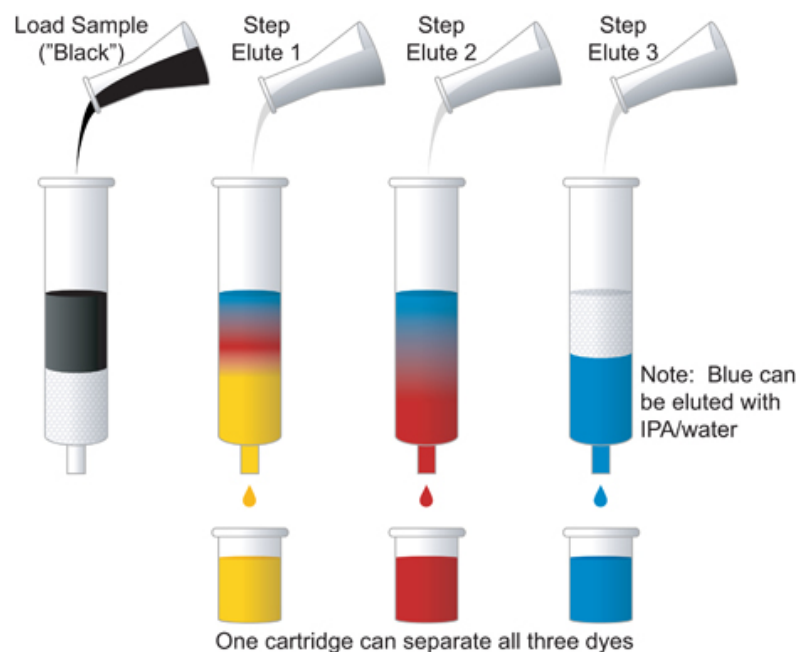
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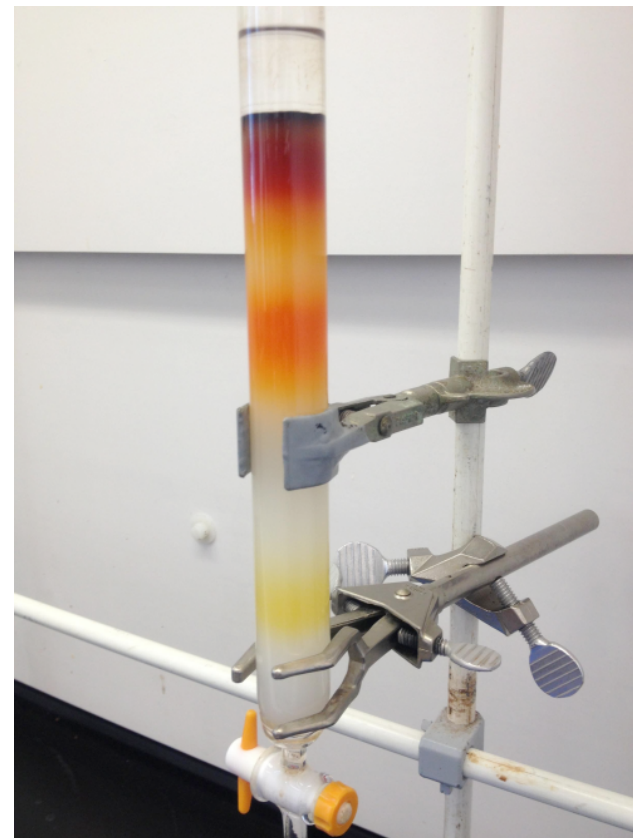
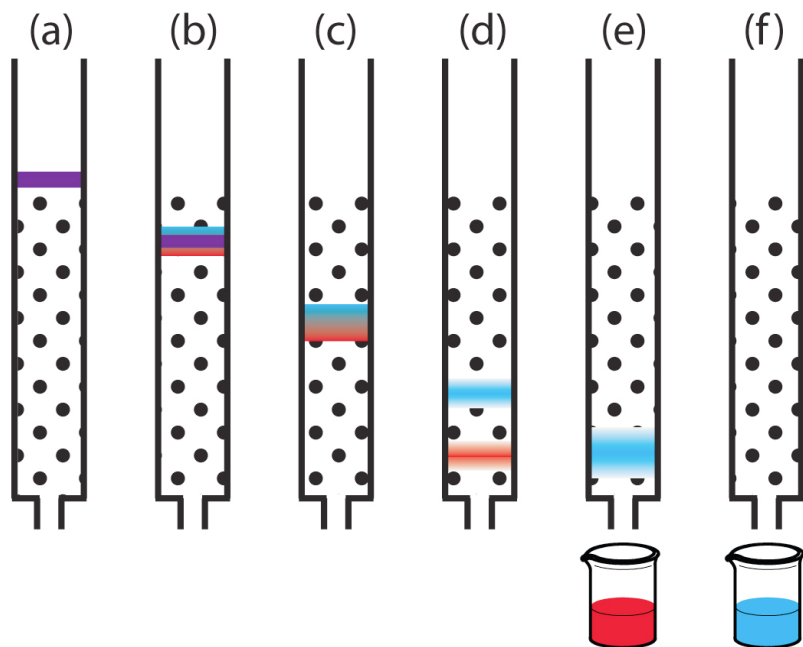
# Chromatography

- ▶ Chromatography separates substances when both are soluble, but one is *more* soluble than the other.
- ▶ **Column Chromatography:** runs samples down a tube filled with silica gel. The more soluble material is more easily carried along by the solvent.



# Chromatography

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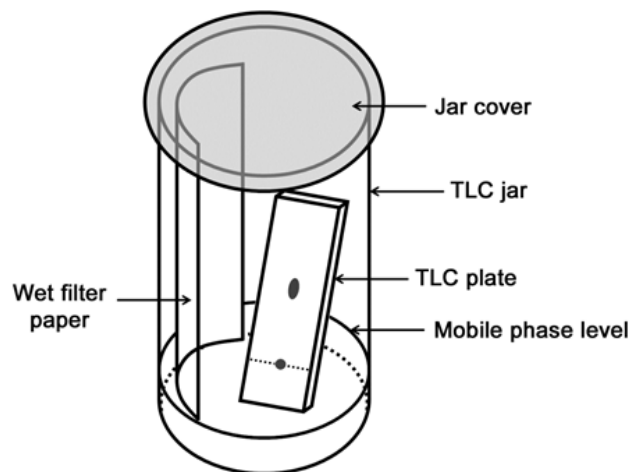




# Elution

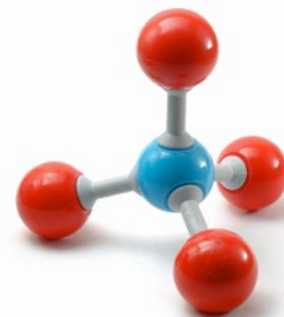
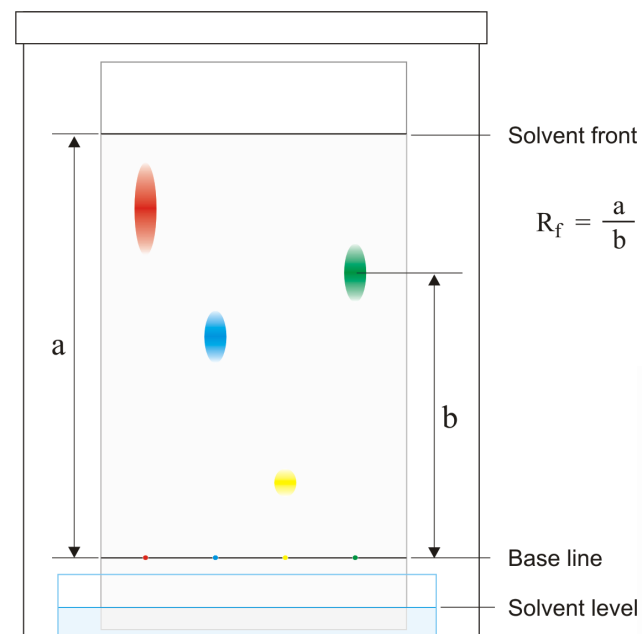
## ▶ Elution

- ▶ Separating Matter
- ▶ Chromatography
  - ▶ Mobile & Stationary
  - ▶ Solvent Effects
- ▶ Applications
  - ▶ Analytic
  - ▶ Preparatory



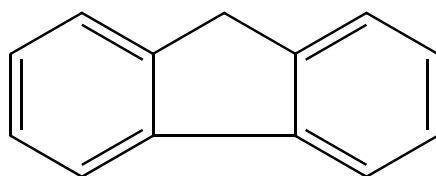
## ➔ The Experiment

- ▶ Part A
    - ▶ Use TLC to Separate Compounds
  - ▶ Part B
    - ▶ Determine Elution Solvents for Separation
- ▶ For Next Week

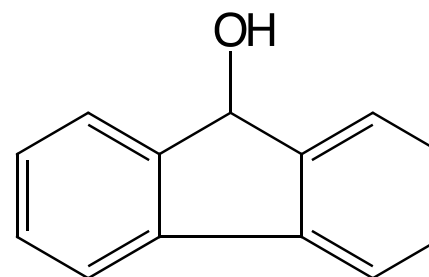


## Part A - Analytic TLC

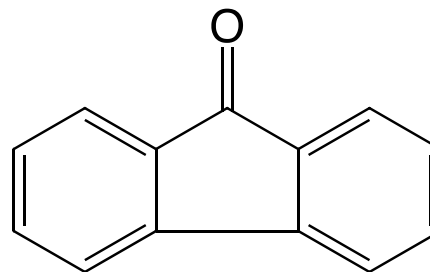
- ▶ Goal: Explore thin layer chromatography as an analytic technique.
- ▶ Objective: Use analytic TLC to determine which compounds are in your unknown.



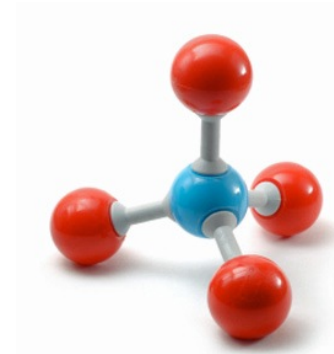
1 (Fluorene)



2 (Fluorenol)



2 (Fluorenone)

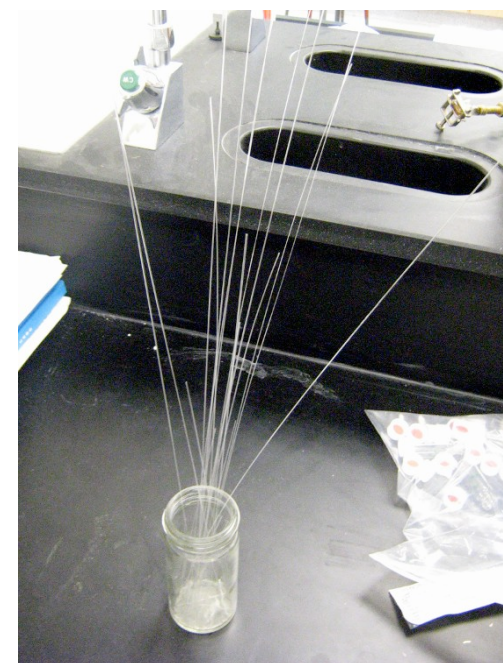


# Part A - Analytic TLC

## ▶ Prepare Capillaries

### ▶ Make 10 micro capillaries.

- ▶ Heat capillary or disposable pipet at tip of burner flame for 30-90 seconds
  - ▶ Time needed will vary.
  - ▶ Look for glass to get jelly like.
- ▶ Remove from flame before pulling
- ▶ Pull as soon as removed from flame
- ▶ Let cool, break into two



1.



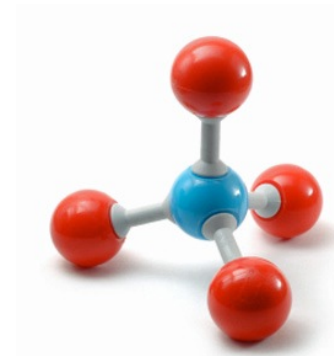
2.



3.



4.

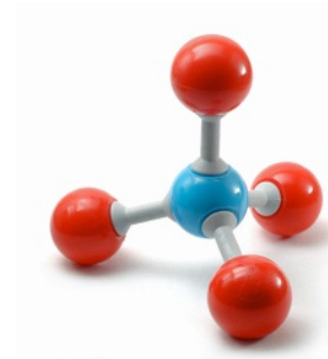
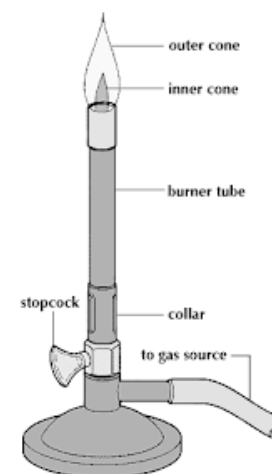
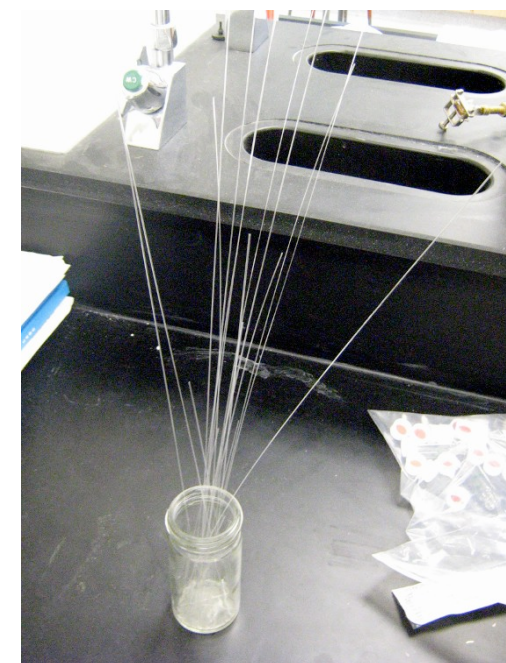
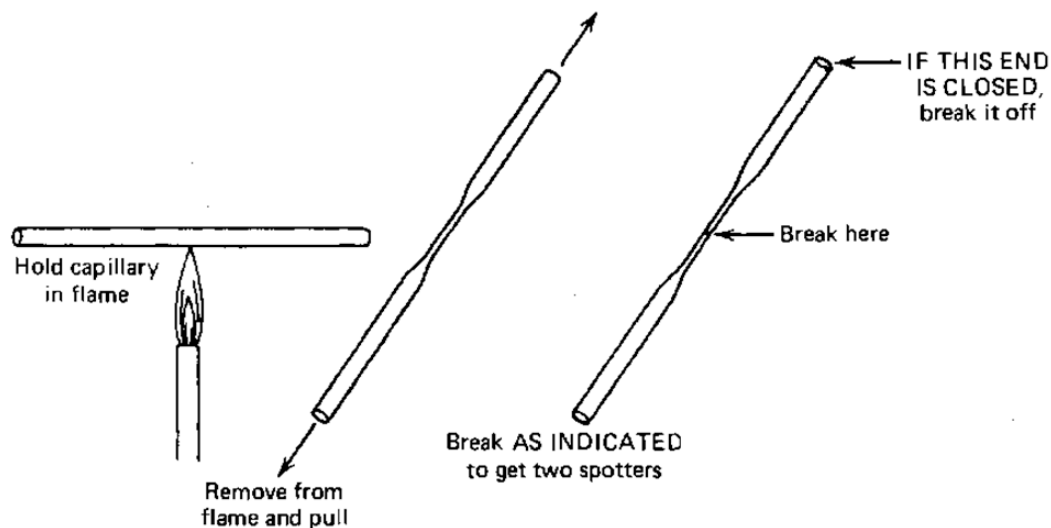


# Part A - Analytic TLC

## ▶ Prepare Capillaries

### ▶ Make 10 micro capillaries.

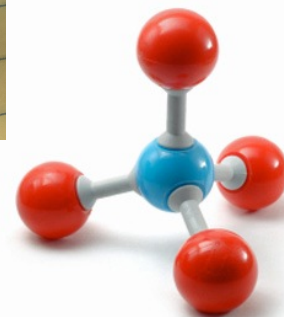
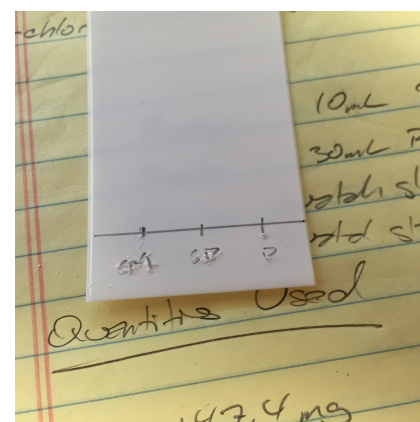
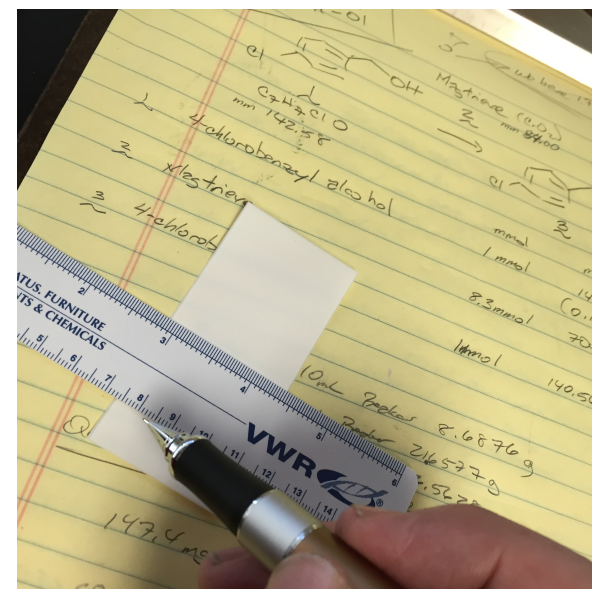
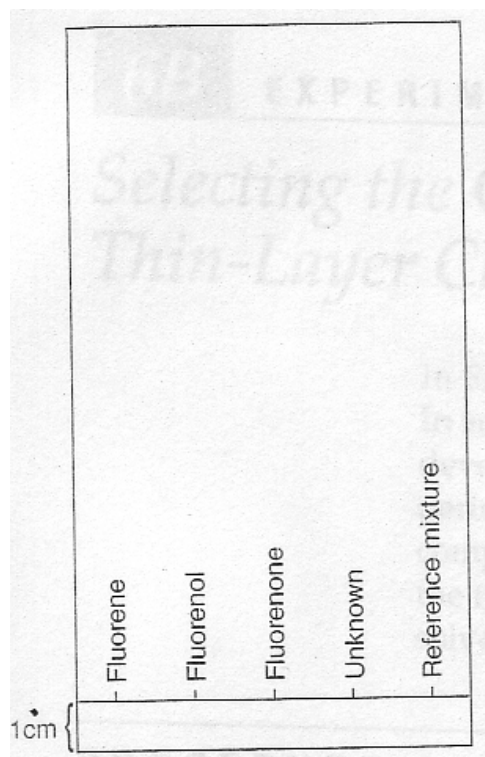
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  - ▶ Time needed will vary.
  - ▶ Look for glass to get jelly like.
- ▶ Remove from flame before pulling
- ▶ Pull as soon as removed from flame
- ▶ Let cool, break into two



# Part A - Analytic TLC

## ▶ Prepare Plate

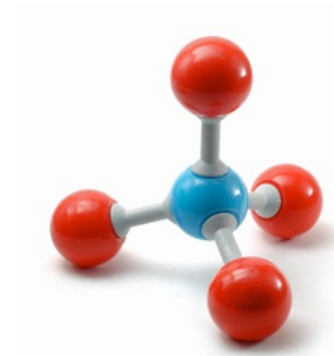
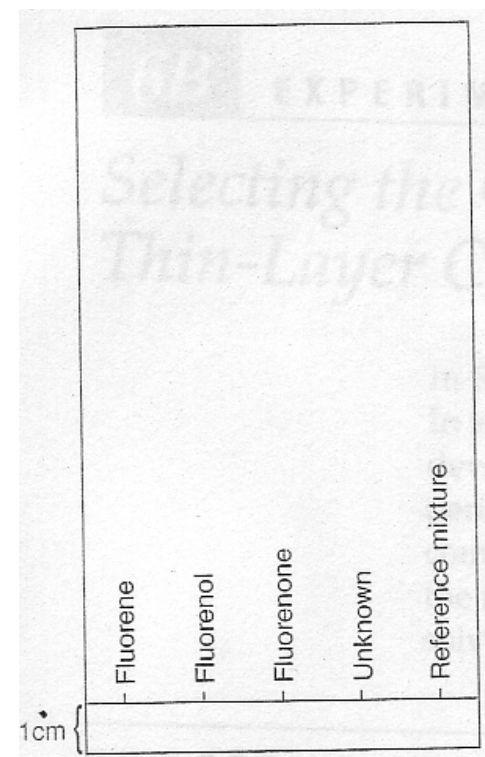
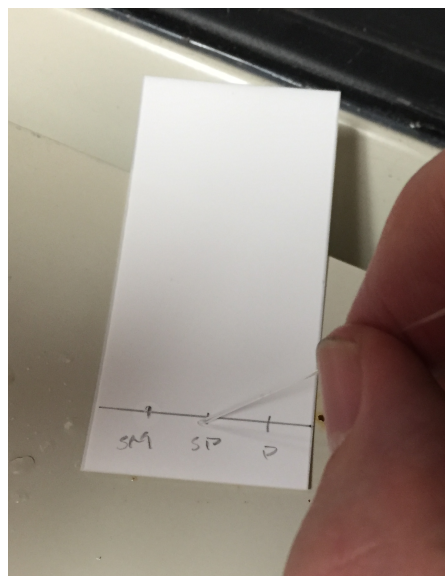
- ▶ Use Pencil – (Pen Ink will Run!)
- ▶ Draw start line about 1 cm from bottom
  - ▶ use ruler to get straight line parallel to bottom of plate
- ▶ Mark 5 channels, starting points



# Part A - Analytic TLC

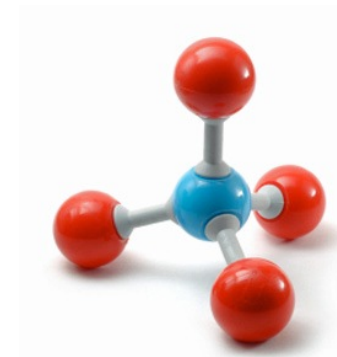
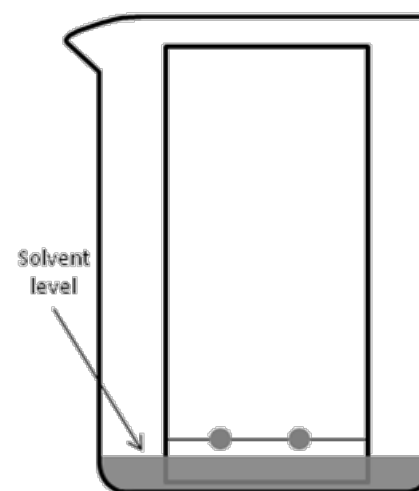
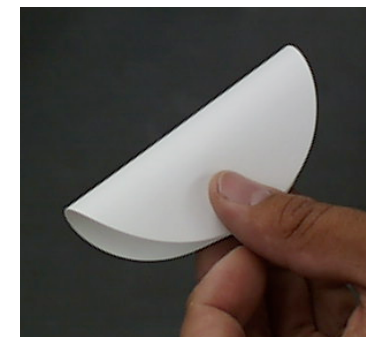
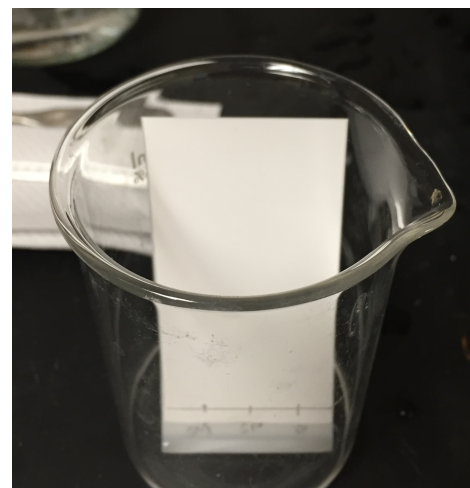
## ▶ Spot Plate

- ▶ Plate a small (tip of spatula) amount of each substance in a section of spotting dish
  - ▶ Including unknown and mixture of three knowns
- ▶ Add 1-2 drops acetone to dissolve
- ▶ Touch capillary to sample to draw in
- ▶ Touch capillary to plate to deposit 2mm dot of sample



# Part A - Analytic TLC

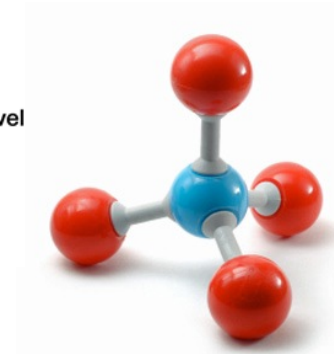
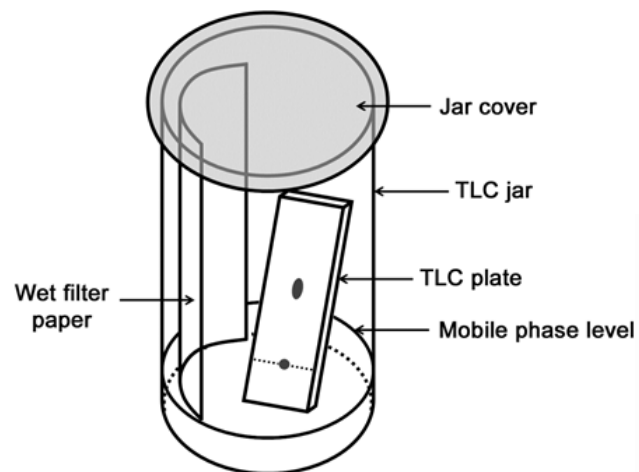
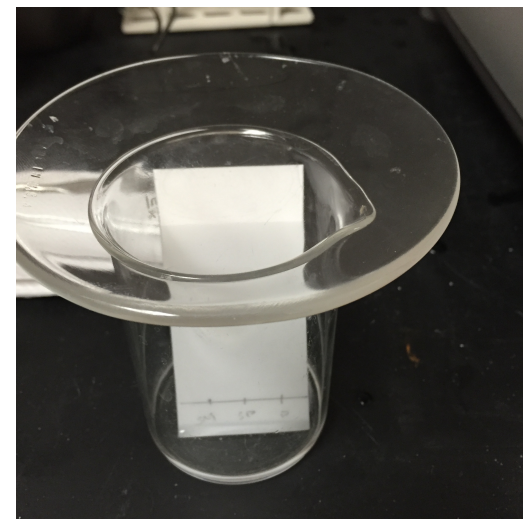
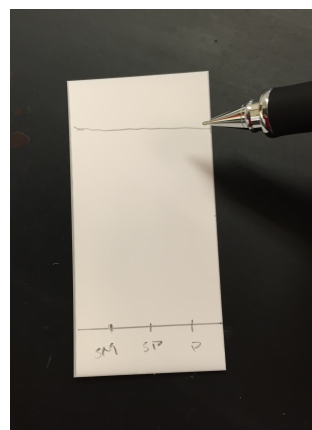
- ▶ Prepare Chamber
  - ▶ Fold filter paper in half
  - ▶ Place in bottom of beaker (~150 mL beaker)
  - ▶ Wet bottom of flask with methylene chloride
- ▶ Elute Plate
  - ▶ Set plate in chamber
    - ▶ Careful not to have it touch filter paper
  - ▶ Start line should not touch elution mixture
  - ▶ Cover chamber with watch glass
  - ▶ Allow solvent to wick up plate



# Part A - Analytic TLC

## ▶ Develop Plate

- ▶ When solvent is 1-2 cm from top of plate, remove plate from chamber.
- ▶ Draw pencil line to show solvent front before it evaporates.
- ▶ Shine UV light on plate.
- ▶ Using pencil circle each spot shown under UV light.

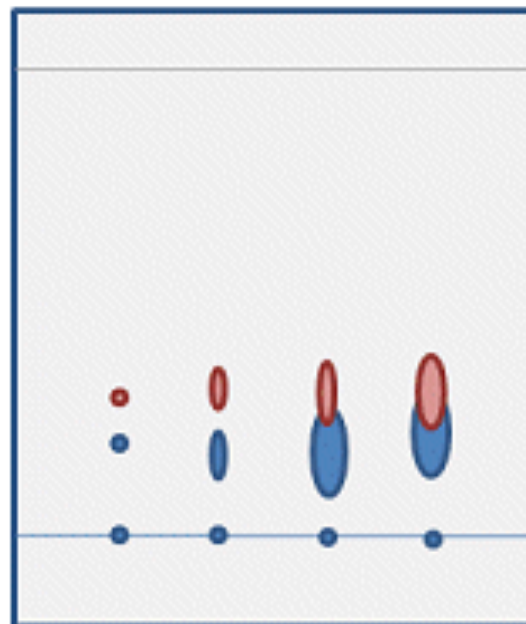




## Part A - Analytic TLC

### ▶ Analysis

- ▶ A good plate will have round spots.
- ▶ If spots are slightly oblong, it's still useable.
- ▶ Identify the center of the of the spot
  - ▶ Center of round shape within spot if it's shaped like an inverted tear.
- ▶ Mark this center as the location of the spot.
- ▶ Determine the distance traveled from the starting line to that center for each spot, in each track.
- ▶ If spots overlap, the plate may have been overloaded.
- ▶ It may be necessary to repeat the process spotting less of the sample on the start line.

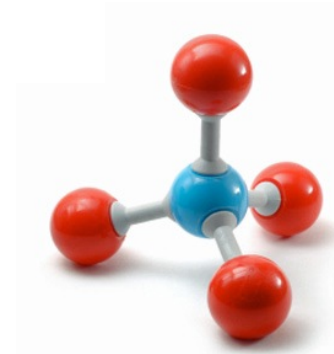


1 2 3 4

Ideal  
shape



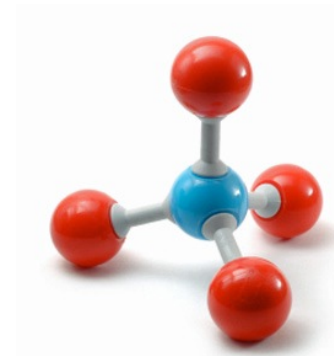
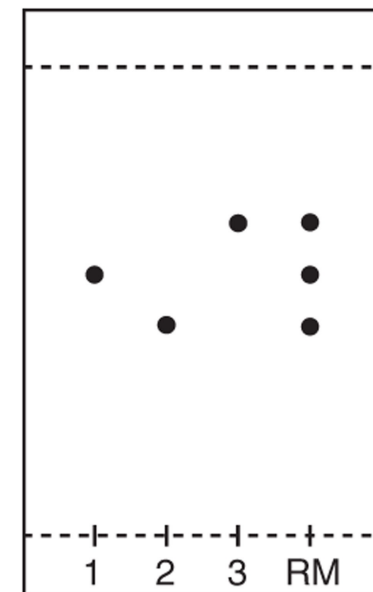
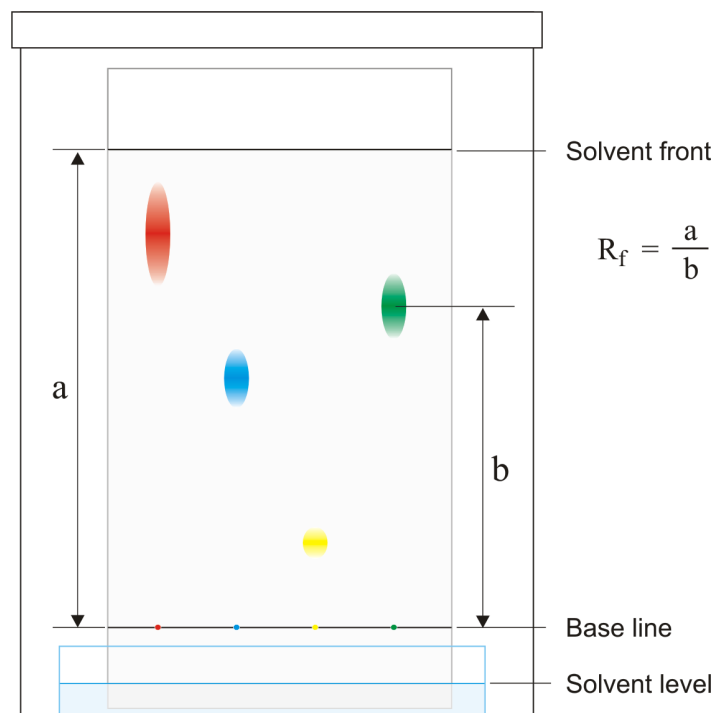
Bad  
tailing



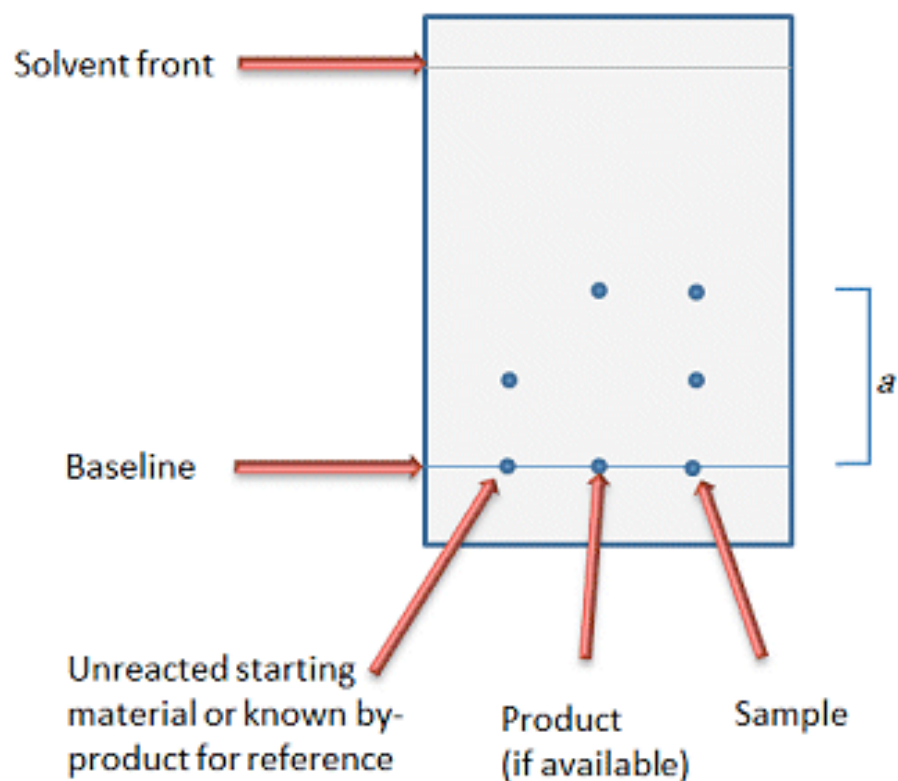
# Part A - Analytic TLC

## ▶ Analysis

- ▶ Determine the retention factor ( $R_f$ ) for each spot.
  - ▶  $R_f$  is the distance traveled by the center of the spot, divided by distance traveled by the solvent front.
- ▶ Record a sketch of your plate with  $R_f$  for each spot labeled.
- ▶ Include a copy of this sketch with your lab report.
- ▶ Use this data to identify the substances in your unknown.
- ▶ Include our conclusion in your lab report.



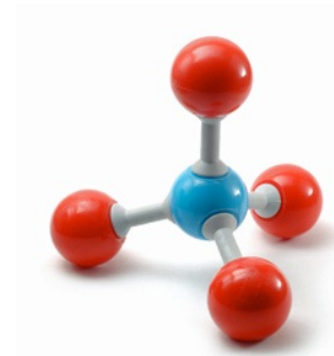
# Part A - Analytic TLC



$$R_f = \frac{\text{Distance from baseline traveled by solute}}{\text{Distance from baseline traveled by solvent (solvent front)}}$$

$$R_f = \frac{a}{b}$$

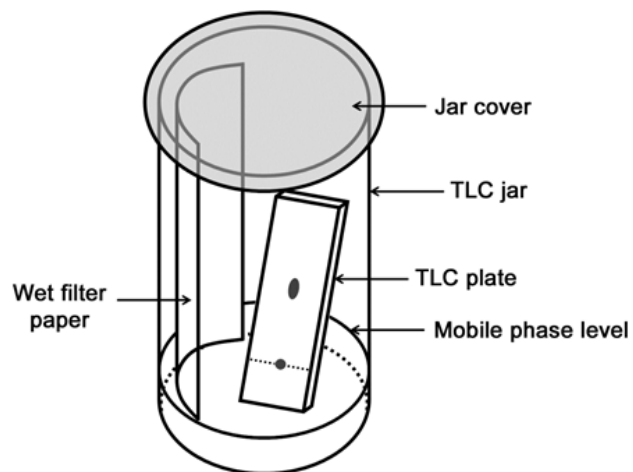
- ▶ Determine the retention factor ( $R_f$ ) for each spot.
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# Elution

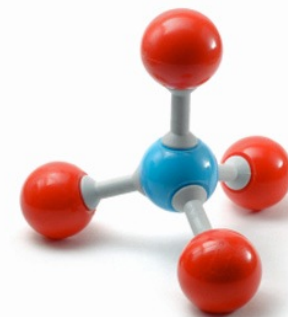
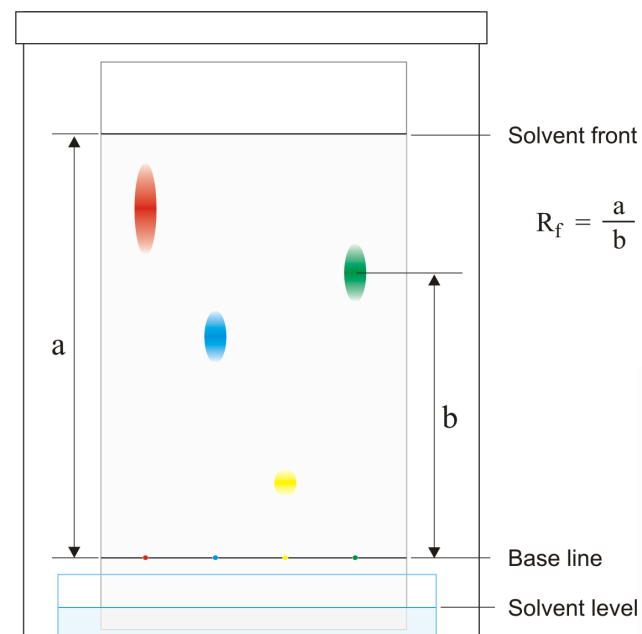
## ▶ Elution

- ▶ Separating Matter
- ▶ Chromatography
  - ▶ Mobile & Stationary
  - ▶ Solvent Effects
- ▶ Applications
  - ▶ Analytic
  - ▶ Preparatory



## ▶ The Experiment

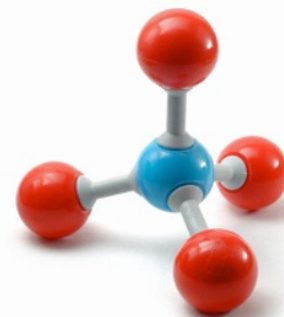
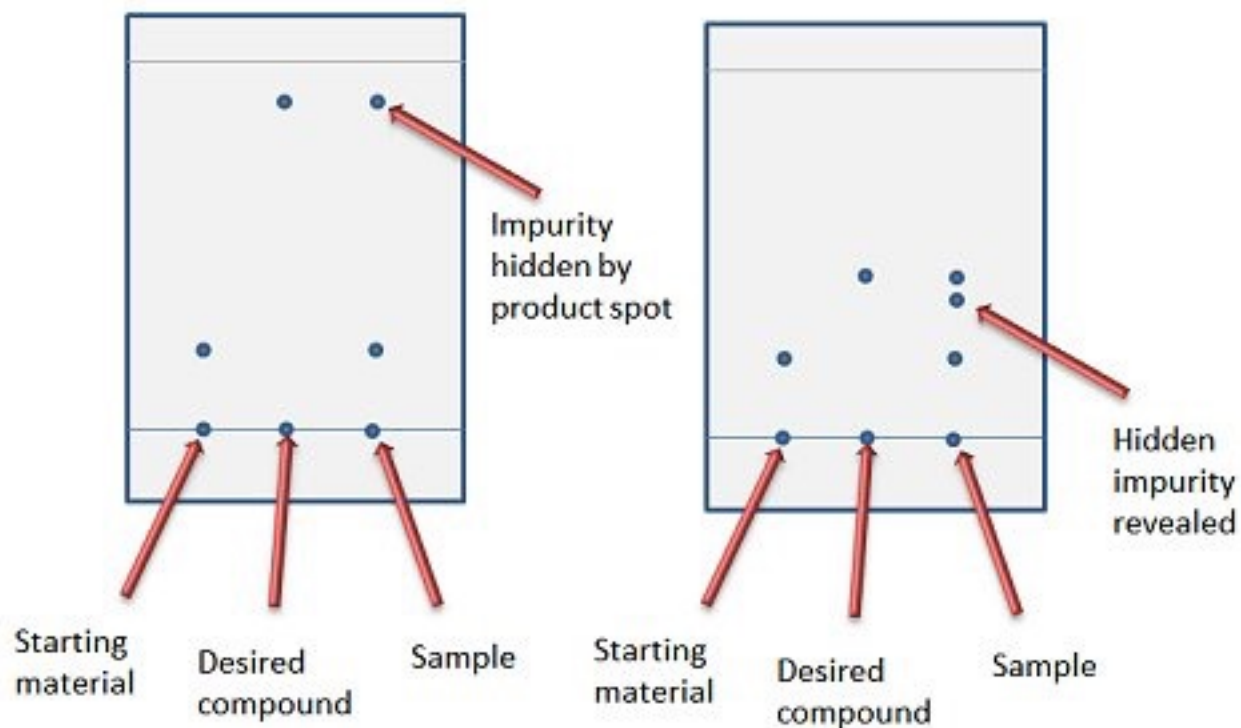
- ▶ Part A
  - ▶ Use TLC to Separate Compounds
- ▶ Part B
  - ▶ Determine Elution Solvents for Separation



## ▶ For Next Week

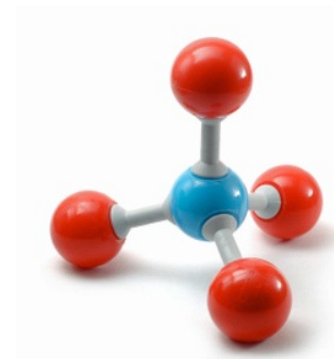
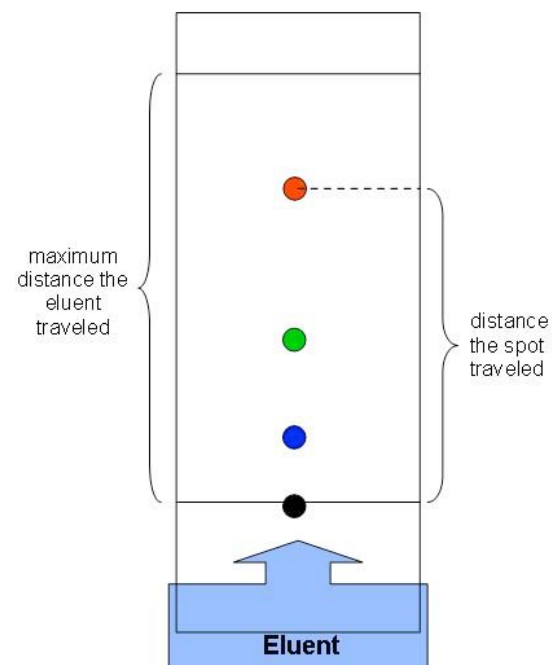
## Part B - Solvent Selection

- ▶ Goal: Explore how different elution mixtures produce different separation in TLC.
- ▶ Objective: Determine which elution solvent produces the clearest separation of a mixture.



## Part B - Solvent Selection

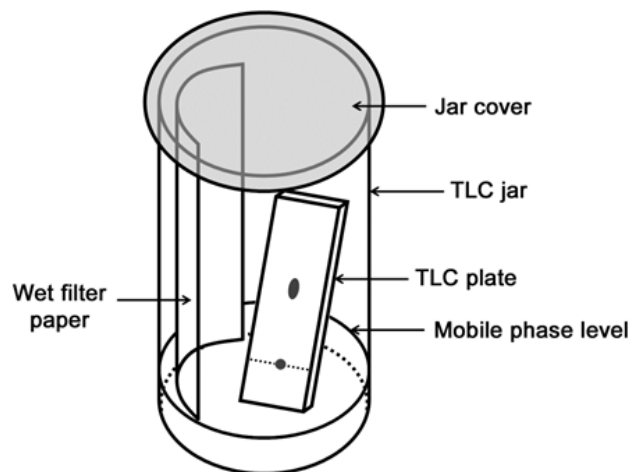
- ▶ Comparing Solvents
  - ▶ You will each be assigned one pair of unknowns and a set of three possible solvents.
  - ▶ Prepare and elute a TLC plate with three tracks.
    - ▶ Each individual compound
    - ▶ In the third track spot both
  - ▶ Run the plate, determine  $R_f$  and comment on the separation.
    - ▶ With each solvent.
  - ▶ In your report, identify the solvent that provides the best separation.
  - ▶ Offer an explanation as to why this solvent separation was better.
  - ▶ Comment on functional groups and polarity of the unknown and solvents.



# Elution

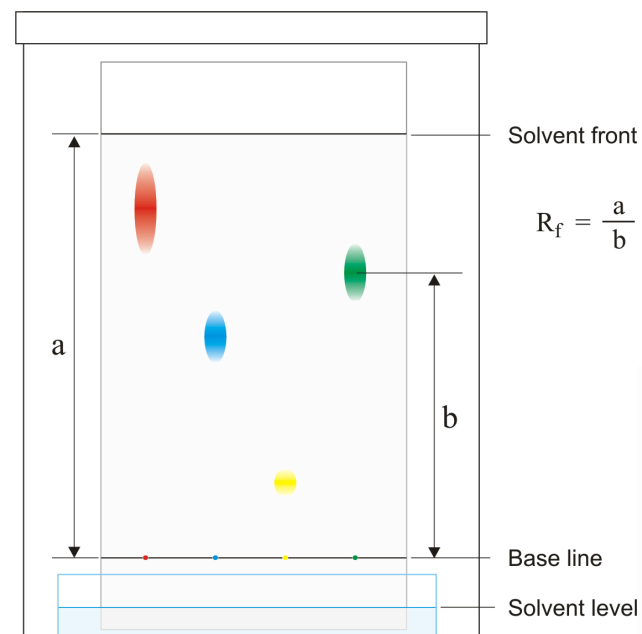
## ▶ Elution

- ▶ Separating Matter
- ▶ Chromatography
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  - ▶ Solvent Effects
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  - ▶ Analytic
  - ▶ Preparatory

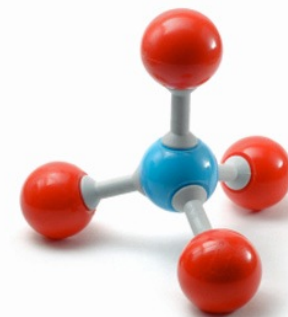


## ▶ The Experiment

- ▶ Part A
  - ▶ Use TLC to Separate Compounds
- ▶ Part B
  - ▶ Determine Elution Solvents for Separation



➔ For Next Week

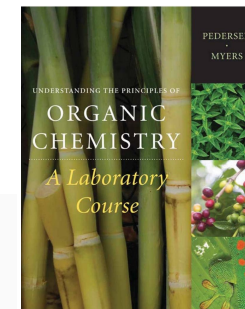
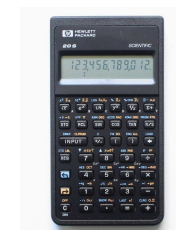
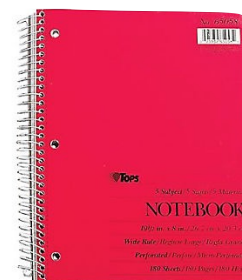


# Next Meeting

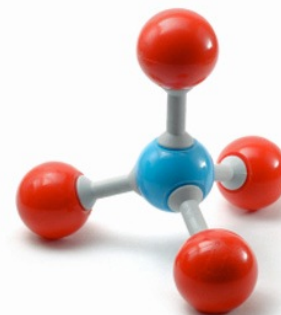
► For next Meeting:

Read: Experiment 12 (page 91)  
Technique 19 - Column Chromatography  
Technique 20 - Thin Layer Chromatography

Do: Identify Objectives  
List Materials w/ Properties  
Organize Procedures



We will start  
with a quiz about  
the experiment and  
reading.





# Questions?

