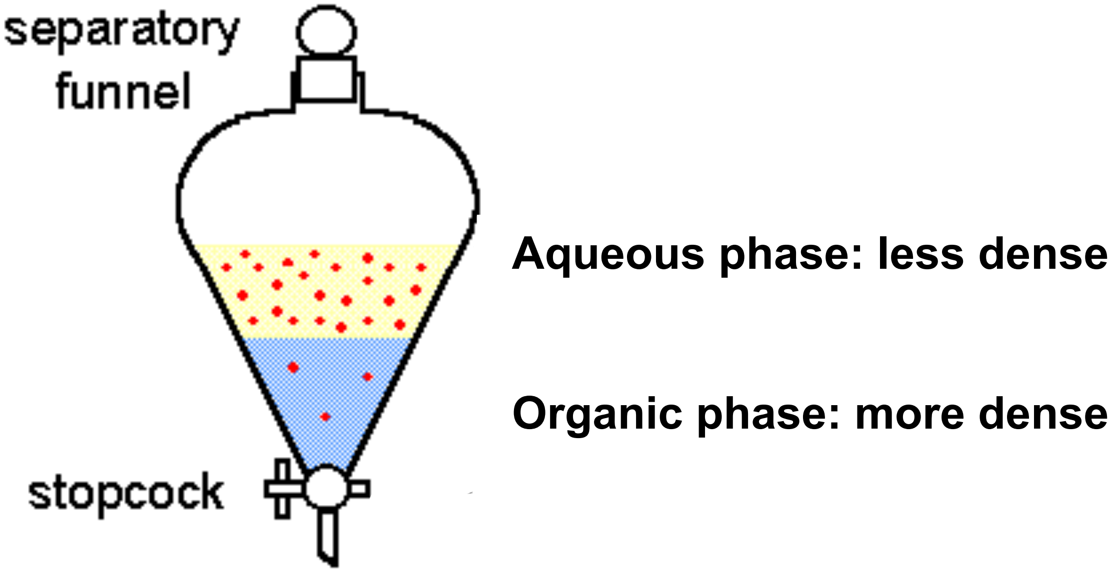
Extraction

Separating Compounds from a Mixture



Procedure:

As described in the Organic Chemistry Laboratory Manual on pages 32 to 33.

Modifications: Part B

In step 11, the lower and upper phases were both drained into separate beakers instead of Erlenmeyer flasks. Additionally, in step 15, we had to subtract the weight of the filter paper soaked in aqueous solution and the watch glass from the weight of the product, filter paper, and watch glass, to calculate a percent yield.

Data and Observations:

Part A: Extraction of water soluble dyes

Ether: transparent, colourless liquid with a faint odour that resembled nail polish remover

Methylene blue: deep blue, odourless liquid

Methyl red: rich red, odourless liquid

- When 1 mL of ether, 1 mL of distilled water and 1 drop of a 0.006 M methylene blue solution was vigorously mixed in a test tube some pressure built up and the blue dye was not spread throughout all of the solution. The organic phase (top layer) remained transparent and colourless meanwhile the aqueous phase was a combination of the methylene blue and water.
- When 1 mL of ether, 2 mL of distilled water and 1 drop of a 0.006 M methyl red solution was vigorously mixed in a test tube some pressure built up but the red colour completely disappeared; the entire solution became transparent and colourless. However, if you looked closely you could see a separation between the 2 phases, being the ether (plus methyl red) and the water.
- When mixing the contents of the 2 test tubes together, a lot of pressure built up inside the test tube. It appeared as though there were 3 levels in the test tube. The top layer was completely transparent, and colourless. The middle layer was cloudy/opaque and blue, and the bottom layer was blue and transparent. Therefore, the blue dye made up the majority of the aqueous phase and the bottom layers whereas the top layer/the organic phase was colourless and transparent.

The Salting out Effect

Aqueous crystal violet: dark purple, almost black, odourless liquid
1-butanol: transparent, colourless liquid with a very faint odour
NaCl: salt; white, crystalline solid

- The test tube without salt was dark purple/indigo, transparent and the colour was distributed evenly throughout the solution.
- The test tube with the salt displayed a colour gradient. The solution was opaque so the purple colour was not as dominant, and it looked like there was a stronger concentration of violet dye towards the top of the test tube in the organic phase. Some precipitate was also noted.

Part B: Effect of solvent on TLC

Unknown Sample (2): White, odourless, crystalline solid

Dichloromethane: Transparent, colourless liquid with a strong chlorine-like odour

NaOH: Transparent, colourless liquid with a strong odour

Concentrated HCl: Corrosive, transparent, colourless liquid with an unpleasant odour

2:8 ethyl acetate and hexane: Transparent, colourless liquid with a mild odour

Biphenyl: Transparent, colourless liquid with a mildly pleasant odour

Benzophenone: Transparent, colourless liquid with a faint odour

- Once the mixture had settled after being shaken, the aqueous layer was opaque and white, on the other hand the organic phase was cloudy, but more translucent than the aqueous phase.
- When the HCl was added to the aqueous phase at first it dissolved but eventually white, grainy precipitate formed.
- The suction filtration process resulted in a clear solution (solvent) and a little bit of solute.
- The unknown mixture on the TLC always had two spots, the circular one closer to the solvent front and an ovular smudge-shaped spot below.

TLC:

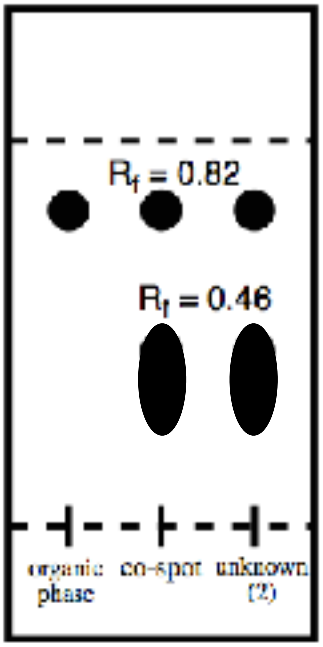


Figure 1:
TLC with unknown in the organic phase
Co-spot: organic phase + unknown 2
Solvent System: 2:8 ethyl acetate and hexanes

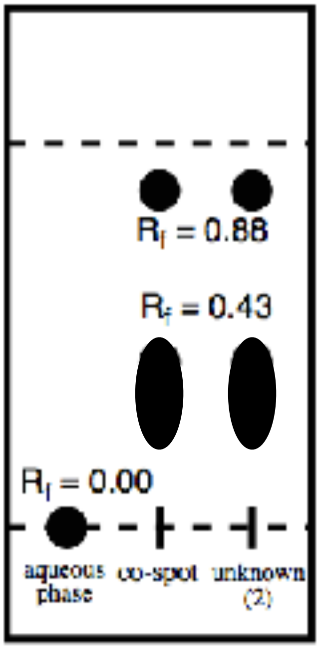


Figure 2:
TLC with unknown in the aqueous phase
Co-spot: aqueous phase + unknown 2
Solvent System: 2:8 ethyl acetate and hexanes

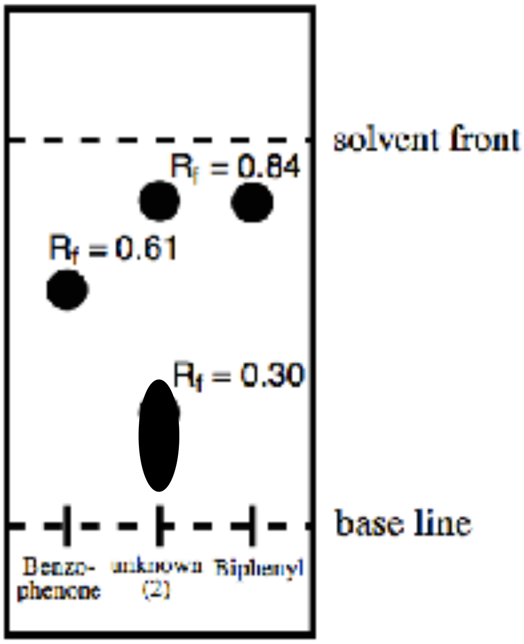
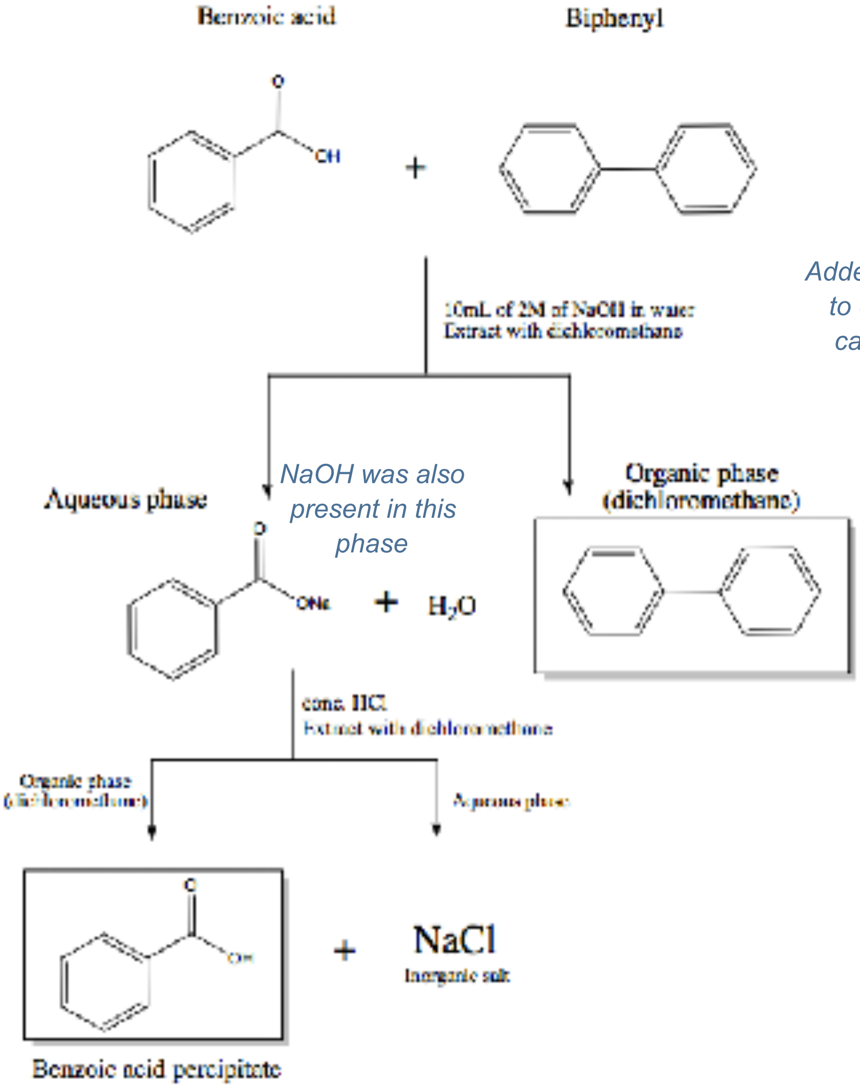


Figure 3:
TLC comparing unknown 2 to biphenyl and benzophenone
Solvent System: 2:8 ethyl acetate and hexanes

Flow Chart



Added a base (NaOH) to deprotonate the carboxylic acid (3 times)

NaOH was also present in this phase

Added an acid (HCl) to reprotonate the carboxylic acid so that it is no longer soluble

Table of Results

Table 1: Results on the unknown sample

Unknown number	2
Initial Mass	0.81 g
Mass Obtained	0.19 g
Composition	benzoic acid & biphenyl
Composition %	23.5%

Table 2: R_f values for Part B's TLC plates

Spot Number (left to right)	Figure 1	Figure 2	Figure 3
1	0.82	0.00	0.81
2	0.82	0.88	0.84
3	0.48	0.45	0.30
4	0.82	0.88	0.84
5	0.46	0.43	-

Calculations:

Part A:

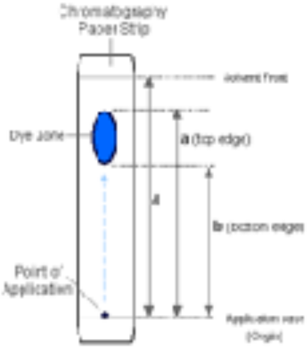
$R_f = [(a + b) / 2] \div d$

$R_f = [(3.3 + 2.6) / 2] \div 3.5$

$R_f = [(5.9) / 2] \div 3.5$

$R_f = [2.95] \div 3.5$

$\mathbf{R_f = 0.843}$



Part B:

<u>Calculating the percent yield</u>

Mass of original unknown sample (2) = 0.81g

Mass of benzoic acid/precipitate obtained = 0.19g

Percent Yield = [(mass of product/precipitate obtained) ÷ (mass of unknown sample 2)] x 100%

= [(0.19g) ÷ (0.81g)] x 100%

= 0.235 x 100%

= 23.5%

Therefore the percent yield/amount of benzoic acid obtained from the original unknown sample is 23.5%

Discussion:

Part A—

Extraction involves a mixture being separated based on its compounds' different solubilities in a medium with 2 immiscible solvents. The immiscible solvents are known as organic and aqueous phases. Using dyes in Part A made the different phases very clear. Specifically in this section, the organic phase was on top and the aqueous phase was below. This is due to the fact that water, being the aqueous phase, is more dense than ether. If the organic phase was composed of a more dense molecule, like dichloromethane in part B, then it would be below the aqueous phase. Methylene blue must be a charged and/or polar molecule because it readily dissolves in water (which is polar). The aqueous layer was therefore blue and transparent. Methyl red is consequently an organic substance which is soluble in ethanol (aqueous). When looking at the test tube, the red colour disappeared however it is assumed that the methyl red was distributed between the organic phase and the aqueous phase as well. Therefore, methyl red would not be the best dye indicator to use when determining phases, but it did allow us to identify a separation between the phases. When the final products of the previous extractions were combined, it appeared as though the volume of the aqueous phase had increased because there was a lot of blue dye. The organic phase was thinner and colourless therefore it contained a combination of methyl red and ether. An extraction between ether and water would be a satisfactory way to separate a mixture of methylene blue and methyl red. Due to their polarities, the methylene blue will be seen but the methyl red will not, therefore it is not completely effective. Thus a good separation would involve two dyes that are immiscible with each other, one being aqueous and the other being organic. In the section titled “The Salting Out Effect”, the aqueous crystal violet dye was evenly distributed unlike the previous section where the dye aided in displaying a separation between the organic phase and the aqueous phase. In other words, aqueous crystal violet is soluble in both the organic (1-butanol) and aqueous (distilled water) phases. When NaCl was added to the test tube, it’s ions dissociated and most of them dissolved in the aqueous phase (water), this is what made the solution more opaque. In the aqueous solvent, the salt ions were favoured over the aqueous crystal violet dye resulting in the prominent violet colour appearing in the organic phase. The addition of a salt increases the ionic strength and makes the aqueous solution more polar. Furthermore, the aqueous phase becomes saturated by the salt so it pushes the crystal violet out towards the organic phase. This can be seen by the small amount of precipitate at the bottom of the test tube; it means that there was too much solute to completely dissolve in the water.

Part B—

To determine the unknown, the compound had to be separated 3 times via extraction. One of the definite components is benzoic acid which contains a non polar benzene ring with a polar carboxylic group. Using the base NaOH in water, the alcohol group of the carboxylic acid will be broken up into it’s ions. The ions are soluble in the aqueous phase meanwhile the other part of the unknown will be found in the organic phase. These compounds are then extracted into two different beakers. To isolate the benzoic acid, we had to do the opposite and add a strong acid so that it is no longer soluble in the aqueous phase. The addition of HCl caused a precipitate to form which would be pure benzoic acid that was obtained by suction filtration. In our case, the precipitate formation was minimal, perhaps more HCl should have been added. The percent yield of the unknown compound was 23.5%. After analyzing the TLC plates, it was determined that the unknown compound contained 2 components, one that was polar, and the other that was non polar. We definitely knew that the unknown would consist of benzoic acid. Figure 3 is what told us the second component, biphenyl. We were able to come to this conclusion based on the fact that one of the unknown spots had the same Rf as biphenyl, and no spots matched benzophenone. Biphenyl is less polar than benzoic acid therefore it is higher up on the TLC plate and consequently has a larger R_f value. The benzoic acid is primarily polar due to the fact that it’s carboxylic acid allows it to slightly dissolve in water. This information corresponds to that of the TLC; the carboxylic acid was the ovular shaped spot which would be bonding to the silica gel and did not travel very far. In both Figures 1 and 2, the co-spot was identical the unknown, which was expected. The TLC for the organic phase shows that biphenyl is present in the organic solvent because we have established that the R_f values are the same across the row and that benzoic acid would not be soluble in the organic phase. As a result, the aqueous column in Figure 2 should have had a spot matching with the ovular spot of the benzoic acid and should have also had an R_f of 0.43. However, an error occurred during the extraction of the aqueous phase resulting in incorrect data. According to the spot with an R_f of 0.0, the substance was extremely polar and did not experience any displacement whatsoever. Therefore, aside from Figure 2, the flowchart explains how the results of the TLC plates came to be.

Sources of Error—

In this experiment, your results would be skewed if you simply did not remove the stopper after shaking any of the mixtures in the test tubes or separatory funnel. Another chance for an error to occur was during the addition concentrated hydrochloric acid. If you did not add enough, you would not obtain a significant amount of benzoic acid precipitate. If you added too much, the NaCl would precipitate out. It was essential to keep and label all the extracts for the TLC plates and to determine the composition of the unknown. Any discrepancies here would cause your final results to be incorrect. When transferring the solutions to the TLC using the capillaries, mistakes could happen such as contamination or incorrectly labeled columns. If this was the case, the TLC plate would have to be repeated. Thus any errors that affect the Rf values and/or the percent yield would negatively impact the results.

Questions:

1. It would be extremely difficult to perform an extraction between ethanol and water because they are both polar and thus would mix/dissolve in each other. For an extraction to be successful, the compounds must have different polarities, and must be immiscible. In the end, water and ethanol would both be present in the aqueous phase, it would be hard to see any separation, and the desired compound would not be able to travel between phases.
2. Adding NaCl to a test tube containing water, ester, and methylene blue would decrease the amount of dye in the aqueous layer. This is due to the fact that NaCl dissolves in water better than methylene because the salt can easily disassociate into its ions, Na^+ and Cl^-. Methylene blue is a larger compound, but is also charged (contains a S^+ and Cl^-). Despite this, the 'salting effect' would occur and the addition of salt would decrease the amount of dye in the aqueous phase.
3. KD = (W1 / V1) ÷ (W2 / V2)

   = (2.0g / 100mL) ÷ (20.0g / 100mL)

   = 0.1

   To find the mass, **o** will represent the mass of Y in ether, the organic phase and **a** will represent the mass of Y in water, the aqueous phase

   1.8g = a + o

   1.8g - a = o

   Combine the 2 equations

   0.1 = (1.8g - a / 100mL) ÷ (a / 100mL)

   a = 0.18g

   o = 1.8g - a

   = 1.8g - 0.18g

   = 1.62g

   Therefore, the mass of compound Y that would be removed is 1.62g after a single extraction.
4. 0.1 = (1.80g - a / 100mL) ÷ (a / 50mL)

   o = 1.50g after the second extraction, which leaves 0.30g *[same process as question 3]*

   0.1 = (0.30g - a / 100mL) ÷ (a / 50mL)

   o = 0.25g after the third extraction, which leaves 0.05g *[same process as question 3]*

Total = 1.50g + 0.25g

= 1.75g

The mass of compound Y that would be removed from the original water solution in question 3 by two extractions using 50 mL of ether each time would be 1.75g.

5. If a student were to lose track of which phase is which they could add water to the solution. Water is polar and will therefore travel to the aqueous phase. The student must take note of where the water ends up, it might be easier to do so with dye. Also, if you know which molecules are the most dense, you can determine if they will lie above or below the water. If you have some TLC plates you can run them comparing your unknown to a compound that you know is polar and would be found in the aqueous phase for example. Finally, the student could use the salting effect if they have dye involved; this would push the dye to the organic phase and they should notice which phase the salt was dissolving.
6. To separate a mixture of the organic base benzyl amine and naphthalene which is also an organic compound, it would be useful to add a strong acid to the mixture. A strong acid such as HCl would interact with the organic base, but not with naphthalene. HCl donates it's proton to benzyl amine because it has an amine functional group which is capable of accepting another proton. The newly formed ions will be soluble in water (aqueous phase) meanwhile naphthalene would remain in the organic phase (ether). Since ether is not very dense, the organic phase would be on top and the aqueous phase would be on the bottom. Once again opposite to what was done in the experiment, the aqueous phase would require the addition of a base so that the ammonium group can deprotonate and benzyl amine can form precipitate. The salt would be removed via suction filtration. Therefore, benzyl amine could be extracted in a separatory funnel with dichloromethane and distilled water now because it would move to the organic phase (dichloromethane).

Raw Data