Experiment 3: Extraction

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Procedure

As outlined in the lab manual ("Experiment 3: Extraction", Organic Chemistry Laboratory Manual, Department of Chemistry, University of Ottawa, 2013, Exp. 3, pg. 5, 6).

Observations

Part A

- Methylene blue
    - blue solution settled at the bottom
    - colourless, transparent solution settled on top
- Methyl red
    - red solution turned light orange, settled on top
    - colourless, transparent solution settled at the bottom
- Methylene blue + Methyl red
    - red solution turned orange & settled on top
    - blue solution settled at the bottom
- Crystal violet without NaCl
    - opaque, dark violet layer settled at the top
    - translucent violet layer settled at the bottom
- Crystal violet + NaCl
    - all the violet solution concentrated at the top
    - bottom layer was colourless & transparent

Part B

- unknown sample ID #310 w/ mass of 0.67 g obtained
    - granulated, white, crystalline solids
- NaOH
    - transparent, colourless solution
- HCl
    - transparent, colourless solution
    - when added to extracts: white, granulated precipitate formed
- cooled sample in ice bath
    - translucent, white solution
- mass of sample obtained after suction filtration: insufficient data
    - not enough sample obtained to weigh in grams
    - had just enough to perform TLC separation

Figure 1: TLC Plate 1

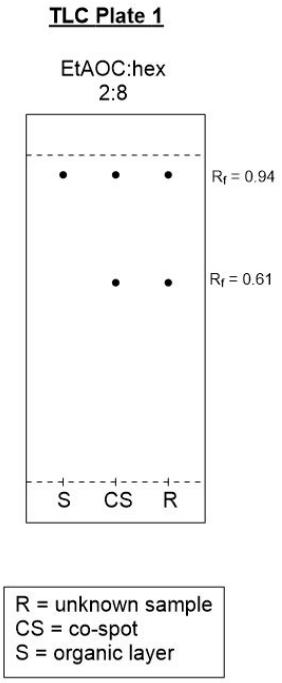


Figure 2: TLC Plate 2

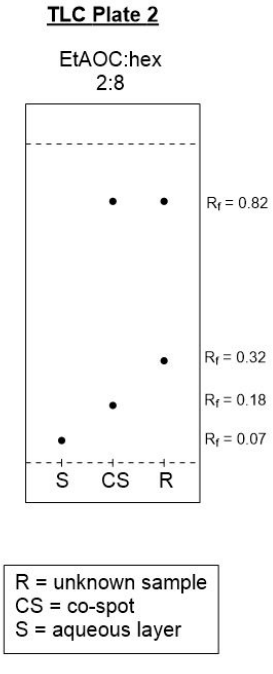
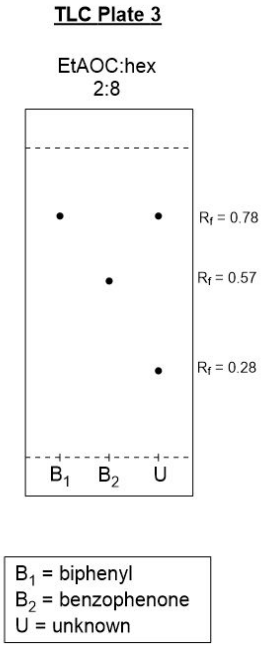


Figure 3: TLC Plate 3

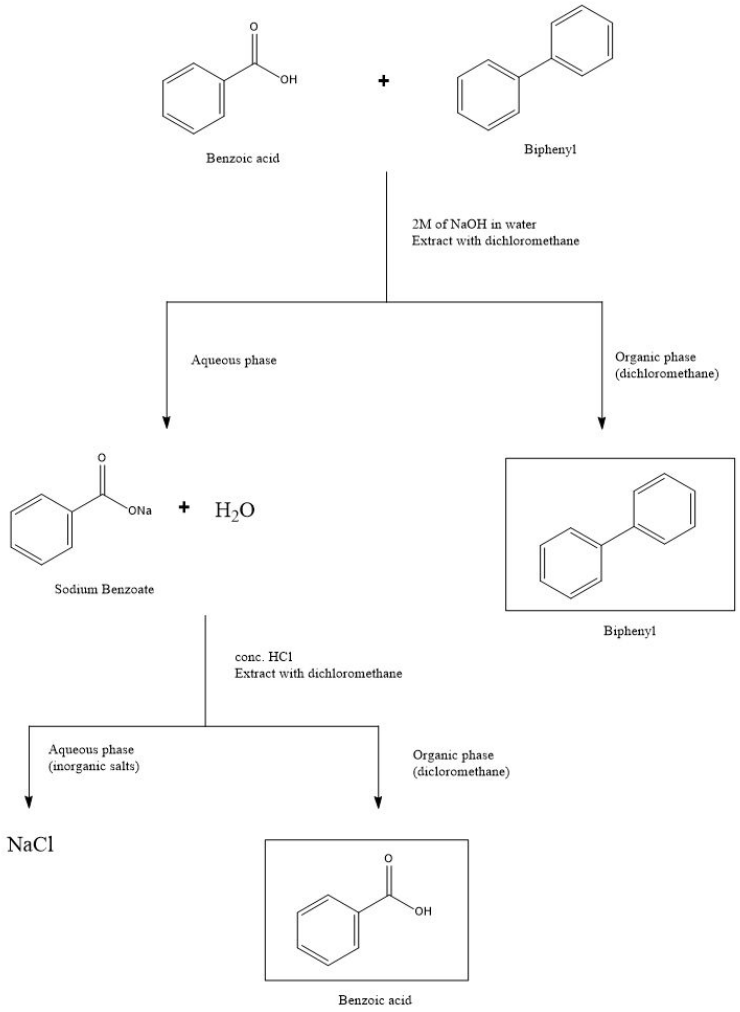


Results

Table 1: mass of unknown compound obtained

Unknown sample ID	Initial mass (g)	Mass obtained (g)	Composition	Percent Yield
310	0.67	N/A	Biphenyl + Benzoic acid	N/A

Figure 4: Flow chart of the reactive extraction



Calculations

Percent Yield

N/A due to insufficient data

R_f values for TLC Plate 1 (Figure 1)

R_f = distance travelled/total distance
= 2.2 cm/3.6 cm
= 0.61

R_f = distance travelled/total distance
= 3.4 cm/3.6 cm
= 0.94

R_f values for TLC Plate 2 (Figure 2)

R_f = distance travelled/total distance
= 0.3 cm/4.4 cm
= 0.07

R_f = distance travelled/total distance
= 0.8 cm/4.4 cm
= 0.18

R_f = distance travelled/total distance
= 1.4 cm/4.4 cm
= 0.32

R_f = distance travelled/total distance
= 3.6 cm/4.4 cm
= 0.82

R_f values for TLC Plate 3 (Figure 3)

R_f = distance travelled/total distance
= 1.3 cm/4.6 cm
= 0.28

R_f = distance travelled/total distance
= 2.7 cm/4.6 cm
= 0.57

R_f = distance travelled/total distance
= 3.6 cm/4.6 cm
= 0.78

Discussion

Part A

- ether and water are immiscible and therefore will separate into two layers
- ether is less dense than water and thus forms the top layer [1]
    - therefore top = organic layer, bottom = aqueous layer
- methylene blue was found in the aqueous layer since it was on the bottom
    - can posit that methylene blue is very polar and/or ionic
- methyl red was found in the organic layer since it was on the top
    - can posit that methyl red is nonpolar
- Crystal violet without NaCl
    - violet dye found in both layers but more concentrated in the top layer
    - therefore crystal violet has a higher affinity for organic layer than for aqueous layer
- Crystal violet + NaCl
    - violet dye found only in organic layer
    - water has a much higher affinity for NaCl since it is ionic, therefore it maximizes its interactions with the ions (becomes saturated), resulting in minimized interaction w/ the organic molecules
    - results in organic layer being sequestered at the top
    - therefore crystal violet was found in the organic layer

Part B

- in the first TLC plate, the organic lane travelled the furthest
- in the second TLC plate, the aqueous lane travelled the shortest distance
    - therefore, nonpolar compounds travel farther than polar compounds, which travel short distances

- when comparing the R_f values between the three lanes in the third TLC plate (Figure 3), it is evident that the R_f of biphenyl (lane B_1) is the same as the R_f of the furthest travelled spot in lane U (unknown sample)
    - the carboxyl group in benzoic acid makes it comparatively more polar than biphenyl
    - based on the previous conclusion, the composition of the spot that traveled the shortest distance in lane U is benzoic acid
    - this leaves the remaining spot in lane U to be biphenyl
    - therefore, the unknown sample contains biphenyl and benzoic acid
- since benzoic acid and biphenyl are miscible, extraction cannot be performed, both are in the organic phase
- however, benzoic acid is an organic acid, thus it can obtain a charge when converted to its conjugate base via deprotonation through an acid-base reaction
- this is achieved by adding NaOH (base) to the solution, which reacts with benzoic acid to produce the ion benzoate, which is water-soluble
- resulting product dissolves in aqueous layer, biphenyl remains in organic layer with dichloromethane, extraction can now be performed
    - by adding NaOH, an extraction between two once-miscible compounds can now be performed
- once the organic phase was removed from the aqueous phase (as much as possible), we needed to obtain solid benzoic acid and determine how much remained
    - by comparing the resulting mass to the initial mass, we can calculate the percent yield, which allows us to see the success of our extraction
    - need to obtain the solid benzoic acid to perform TLC analysis
- can convert benzoate back to its conjugate acid via protonation through an acid-base reaction
- HCl is a strong acid, reacts with benzoate to produce benzoic acid
    - benzoic acid is uncharged, immiscible on water (since it's saturated with NaCl)
    - benzoic acid forms in the organic layer with dichloromethane, allowing us to proceed with extraction

Sources of error

- the source of error that prevented us from obtaining the mass of the benzoic acid after extraction was that we did not use the coffee filter to drain the mixture and instead went straight to the suction filtration
    - this was caused by rushing to complete the experiment before we ran out of time, causing us to misread the procedure/skip steps
    - this resulted in very little solid being obtained
- another source of error could have risen from adding slightly too much HCl
    - this would have caused some NaCl to precipitate out

Questions

1.
    - acetone is miscible in water [2]
    - miscible compounds cannot be extracted as they are not distinctly separated, cannot distinguish between the two solutions, both water and acetone occupy both layers simultaneously
    - extraction can only be performed between immiscible compounds as they form distinct layers/phases
2.
    - amount of dye would decrease from the aqueous layer due to the salting out effect
    - NaCl is an ionic compound, therefore it has a higher affinity to water than all the other compounds in the test tube
    - despite methylene blue also being soluble in water, the majority of its constituent atoms are nonpolar, therefore water has a higher affinity for NaCl and thus will maximize its interactions with the ions, thus reducing interactions with methylene blue
    - this forces a lot of the methylene blue to occupy the organic layer
3.
    - $KD = (W_1 / V_1)/(W_2 / V_2)$
      $KD = (2.0\ g / 100\ mL)/(20.0\ g / 100\ mL)$
      $KD = 0.1$
    - let W represent mass of compound Y in aqueous layer and E represent mass of compound Y in organic layer
    - $1.4\ g = W + E$
      $1.4\ g - W = E$
    - $0.1 = (W / 100\ mL)/(1.4\ g - W / 100\ mL)$
      $W = 0.13\ g$
      $E = 1.27\ g$
    - therefore, the mass of compound Y extracted would be 1.27 g
4.
    - 1st extraction:
        - $0.1 = (W / 100\ mL)/(1.4\ g - W / 50\ mL)$
          $W = 0.23\ g$
          $E = 1.17\ g$
        - therefore, the mass of compound Y after 1 extraction would be 1.17 g
    - 2nd extraction:
        - $0.1 = (W / 100\ mL)/(0.23\ g - W / 50\ mL)$
          $W = 0.038\ g$
          $E = 0.19\ g$
        - therefore, after the 2nd extraction, an additional 0.19 g of compound Y would be extracted
    - the total mass of compound Y extracted would be 1.36 g

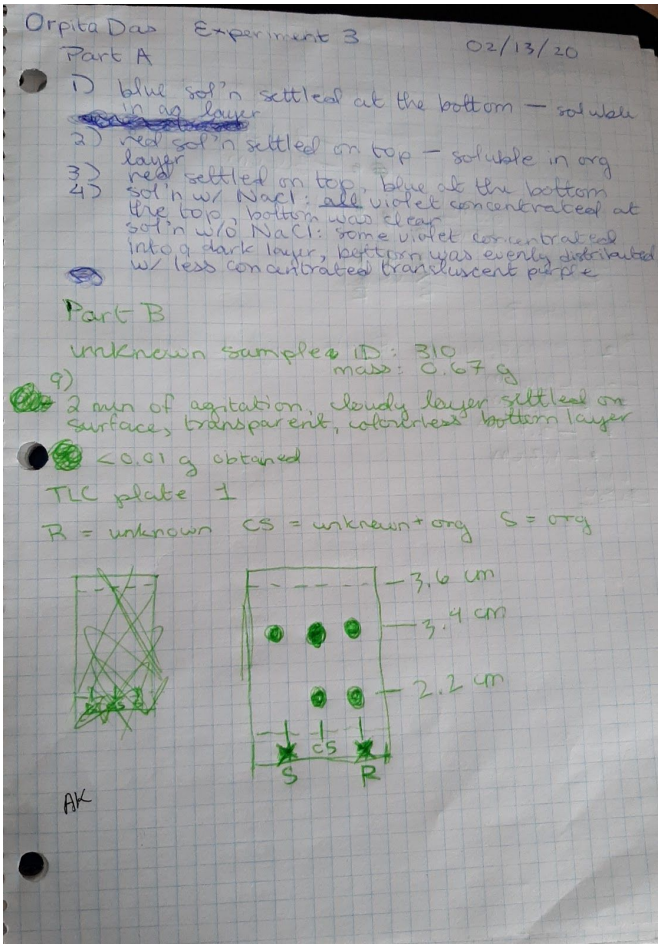
5.
    - the student could add a polar solvent (i.e. water) that has been labelled with a dye to the mixture; wherever the dyed solution settles will be the aqueous layer
    - the student could compare the densities of the organic & aqueous compounds; the compound with the highest density would be on the bottom & the lower density would be on top
    - the student could add an organic solvent that has been labelled with a dye to the mixture as well as an ionic salt; this would cause the salting out effect, in which the aqueous layer would become saturated with the ions, causing all the dyed solution to accumulate in one layer, which would be the organic phase
6.
    - I would add ether to the mixture of benzyl amine and naphthalene
    - then I would add a strong acid (i.e. HCl) to the mixture, which would react with benzyl amine to produce its conjugate acid, which is a protonated ion
    - the resulting product would no longer be miscible with naphthalene and ether (since it's ionic), causing them to separate into an aqueous and organic phase

References

[1]: "Experiment 3: Extraction", Organic Chemistry Laboratory Manual, Department of Chemistry, University of Ottawa, 2013, Exp. 3, pg. 4

[2]: National Center for Biotechnology Information. PubChem Database. Acetone, CID=180, https://pubchem.ncbi.nlm.nih.gov/compound/Acetone (accessed on Feb. 27, 2020)

Raw Data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Part A

1) blue sol'n settled at the bottom — soluble in aq layer
2) red sol'n settled on top — soluble in org layer
3) red settled on top, blue at the bottom
4) sol'n w/ NaCl: all violet concentrated at the top, bottom was clear
sol'n w/o NaCl: some violet concentrated into a dark layer, bottom was evenly distributed w/ less concentrated translucent purple

Part B

unknown sample ID: 310
mass: 0.67 g

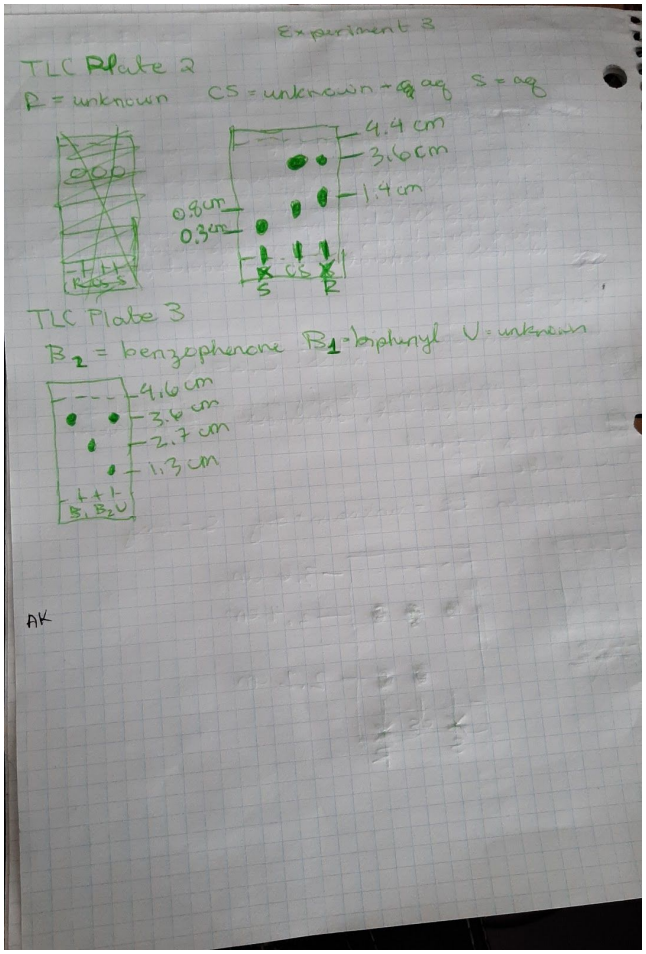
9)
2 min of agitation, cloudy layer settled on surface, transparent, colorless bottom layer
<0.01 g obtained

TLC plate 1

R = unknown CS = unknown + org S = org

3.6 cm
3.4 cm
2.2 cm
CS
S R

AK


Experiment 3

TLC Plate 2

R = unknown CS = unknown + aq S = aq

4.4 cm
3.6 cm
1.4 cm
0.8 cm
0.3 cm
CS
S R

TLC Plate 3

B_2 = benzophenone B_1 = biphenyl U = unknown

4.6 cm
3.6 cm
2.7 cm
1.3 cm
B_1 B_2 U

AK