Procedure:

- Please refer to lab manual, 1321 Organic Chemistry lab pg. 17-18.

Observations:

Part 1 TLC

- 2:8 ethyl acetate solvent was used (10mL) and it was a clear colourless liquid.
- Sample number 79 was used. It was a white small granulated solid powder.
- After mixing the sample number 79 and 10mL of dichloromethane solvent, the solution became clear and colourless.

Part 2 TLC

- Ethyl acetate solvent was a clear colourless liquid
- Under the UV light the spots were seen as black circles of various diameter.
- Hexane solvent was a clear colourless liquid.
- It was seen that the spots moved at a much slower rate in a hexane solvent than in the ethyl acetate solvent. It was also seen that some spots did not move from their starting positions.

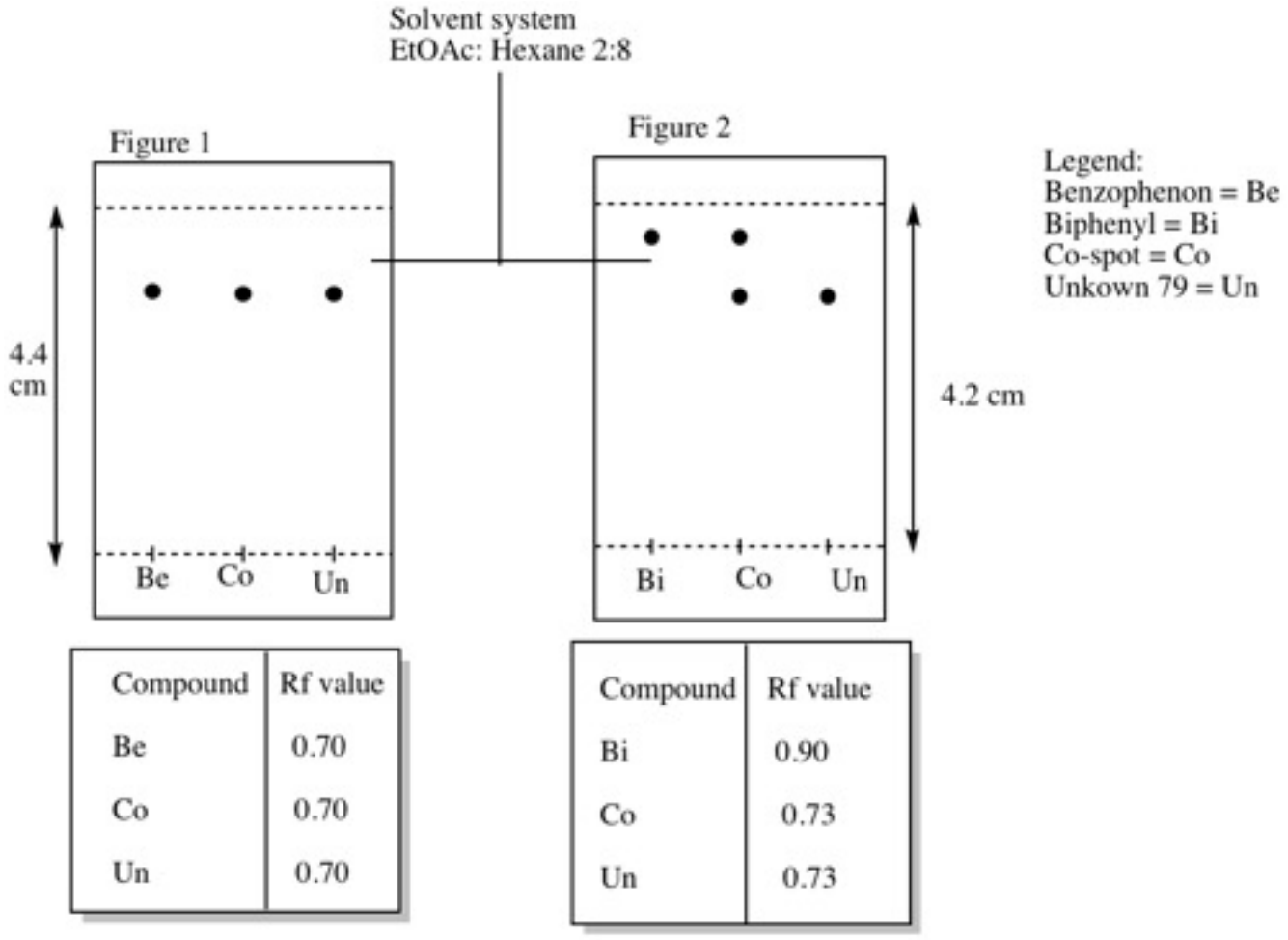
Part 3 TLC

- The unknown mixture of XX was used and it was a colourless liquid. We used approximately 2mL of this liquid.

Discussion

Part 1

- The main reason for the first part of the experiment was to compare an unknown compound number 79 to two other known compounds in order to determine the identity of compound number 79. Moreover, to see in action how difference in polarity can help us identify and compare different compounds.
- By doing the TLC, it was observed that the biphenyl spot was higher, at 0.90 (fig. 2) than the spot made by the unknown compound. This observation tells us that the unknown compound is more polar than biphenyl.
- However, it was seen that the benzophenone spot corresponded to the spot made by the unknown compound. This tells us that the unknown compound and benzophenone have similar polarity compared to each other.
- To further investigate if the unknown compound was indeed benzophenone, the R_f values of the unknown and benzophenone were compared and they were both 0.75 (fig.1) indicating that the unknown compound and benzophenone were both same compounds.



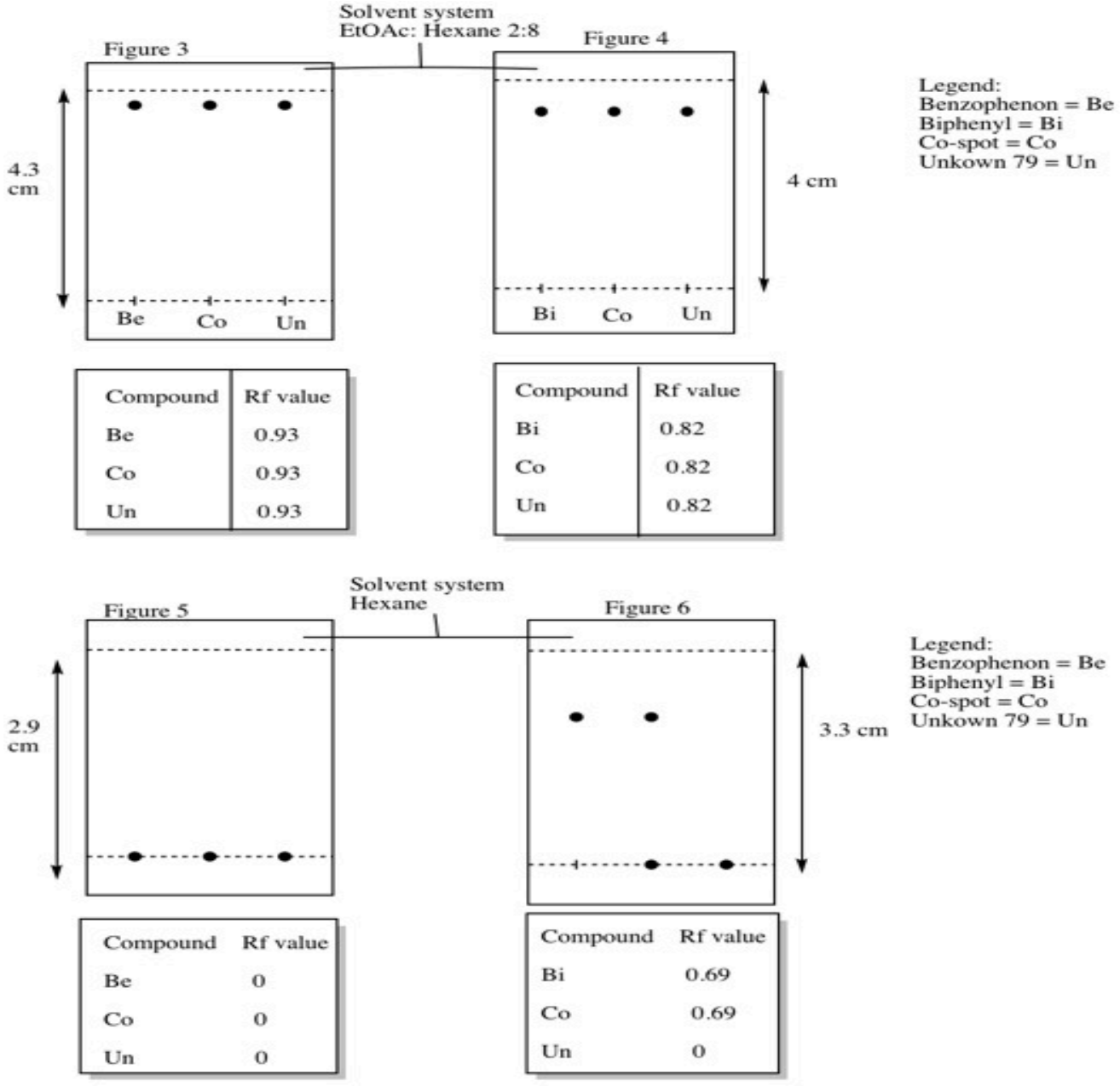
Compound	Rf value
Be	0.70
Co	0.70
Un	0.70

Compound	Rf value
Bi	0.90
Co	0.73
Un	0.73

Part 2

- In part two of the experiment there were two different solvent used. One was ethyl acetate and another was hexane in order to observe how changing the polarity of the solvent can affect how much displacement the different compounds showed in the TLC.
- With the ethyl acetate as the solvent it was observed that all the spots moved quickly up the silica gel strip. This indicates that either all the compounds used in this part of the experiment were same or the polarity of the solvent was too strong for the compounds to give any meaningful results. In the case of this experiment, the later was true because the more polar compound (benzophenone), which had a strong attraction between the stationary phase (silica gel) had been overpowered by the polar solvent (ethyl acetate) to the point that benzophenone and biphenyl both ended up getting the same R_f value.
- In the second part of the experiment hexane was used as a solvent, which is non-polar in nature
- The non-polar hexane had almost no effect on the attraction between the stationary phase (silica gel) and the benzophenone because the benzophenone spot showed almost no displacement on the stationary phase.
- On the other hand, biphenyl showed displacement which meant that biphenyl was more attracted towards the hexane solvent than the silica gel as seen by the R_f value of 0.69.

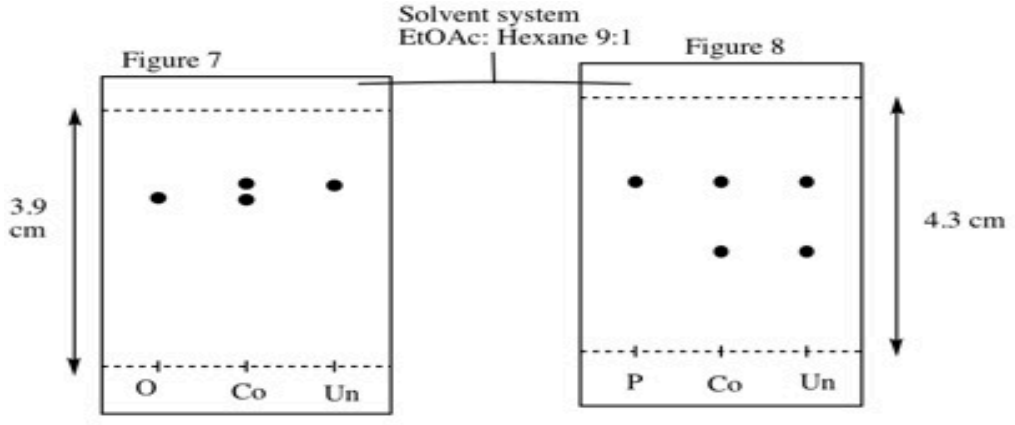
- This part of the experiment showed us that non-polar solvents will always have a lower R_f value than polar solvents because non-polar solvents cannot overcome the binding forces (hydrogen bonding) between the adsorbent and the polar compound.



Part 3

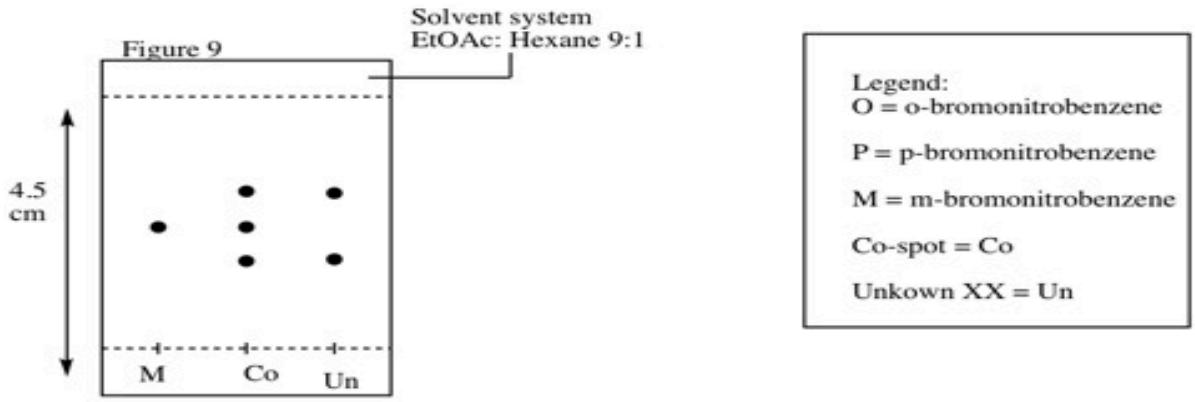
- In this part of the experiment the composition or identity of an unknown solution had to be deduced using three reference compounds on the TLC. The three reference compounds' shortened names were ortho-, para- and meta-.
- Using the three reference points it can be observed that the mixture of 'YY' is a combination of ortho- and para- because for both ortho- and para- TLC, there was a spot that had the same

displacement as the ones showed in the sample; whereas, there was no spot that matched between the meta and the 'XX' sample.



Compound	Rf value
O	0.66
Co	0.66
Un	0.71

Compound	Rf value
P	0.67
Co	0.67
Un	0.39



Compound	Rf value
M	0.35
Co	0.48
Un	0.62

Results

Through the image J software, the percentage composition of ortho- in the XX compound was 44.1% while the para percentage composition was 55.9%. Using the calibration curve yield, it can be determined that the ratio of ortho to para in the compound is 2:3.

Questions

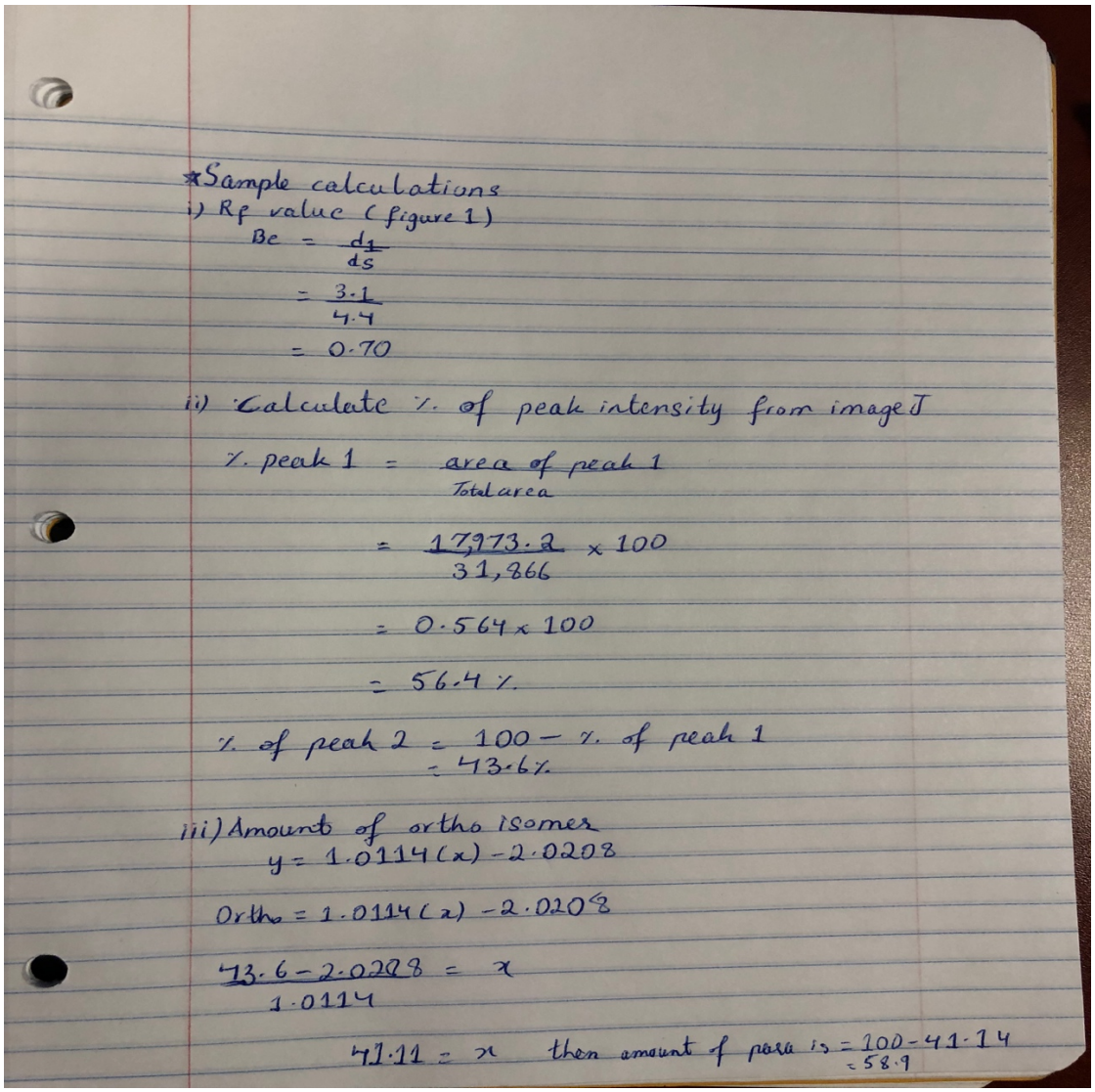
1. Increasing the polarity of the solvent system means that the spotted compounds will be more attracted to the solvent than the stationary phase, which will cause the compounds to move higher up the plate. Moreover, this increase in displacement would also mean that the R_f values of the compounds are also higher. The reason for this increased displacement is because the compounds are making more hydrogen bonds with the solvent system than the stationary system which also the compound to move up higher on the plate.

2. **a)** Benzyl alcohol, which is the most polar compound out of the three mentioned, will have the smaller R_f value on the silica gel as it has an alcohol as its functional group which is the more polar than its aldehyde and ester counterparts.

   b) Aniline will be the compound with the smallest R_f value as it is more polar due to the free lone pair electrons

   c) The compound with the smallest R_f will be benzoic acid as its functional group which is an carboxylic acids makes its more polar than the benzophenone (ketone) or biphenyl (hydrocarbon)

Sample Calculations:



*Sample calculations

i) Rf value (figure 1)

$Be = \frac{d_1}{d_S}$

$= \frac{3.1}{4.4}$

$= 0.70$

ii) Calculate % of peak intensity from imageJ

% peak 1 = $\frac{\text{area of peak 1}}{\text{Total area}}$

$= \frac{17,973.2}{31,866} \times 100$

$= 0.564 \times 100$

$= 56.4\ \%$

% of peak 2 = 100 − % of peak 1

$= 43.6\ \%$

iii) Amount of ortho isomer

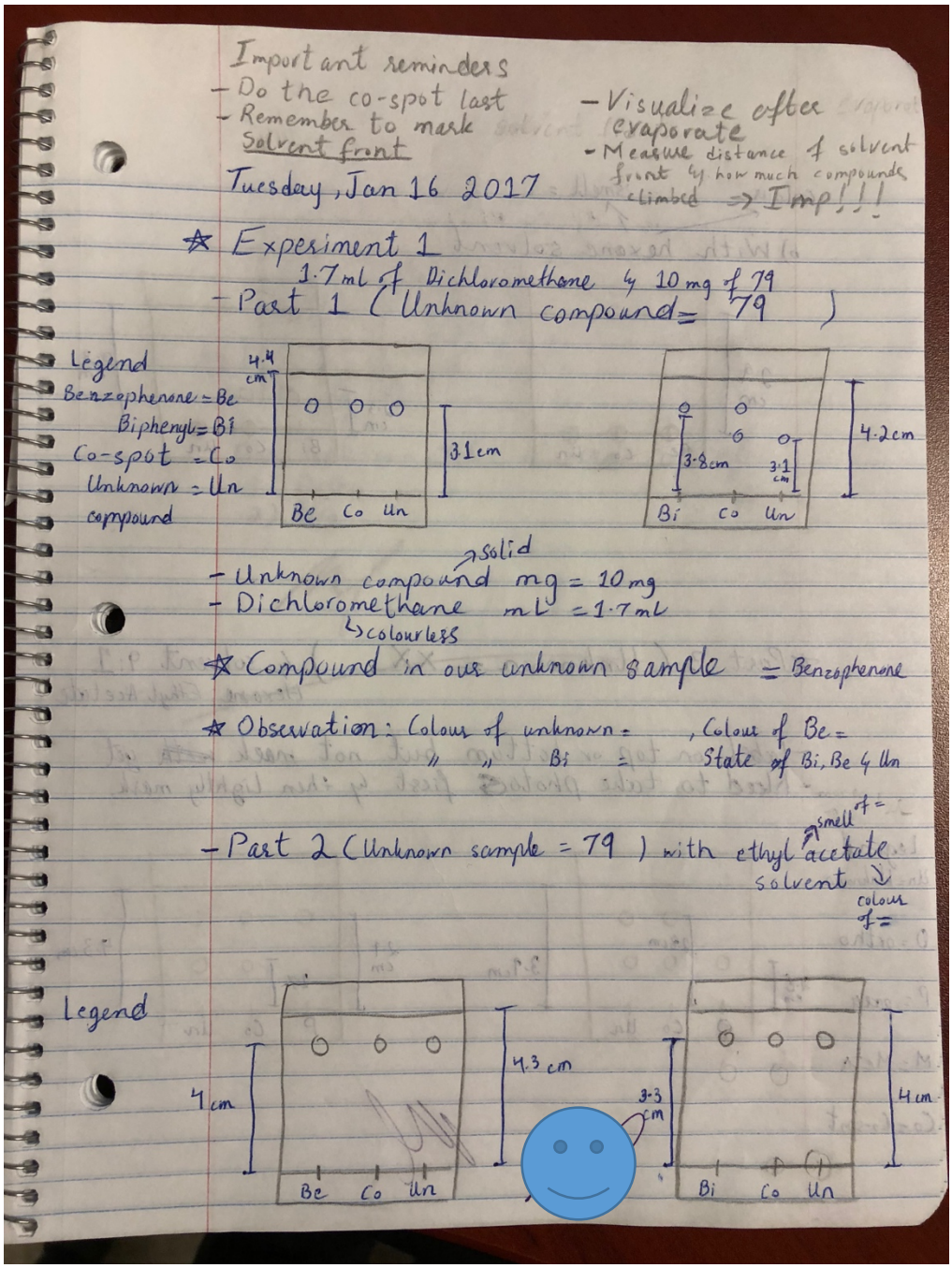
$y = 1.0114(x) - 2.0208$

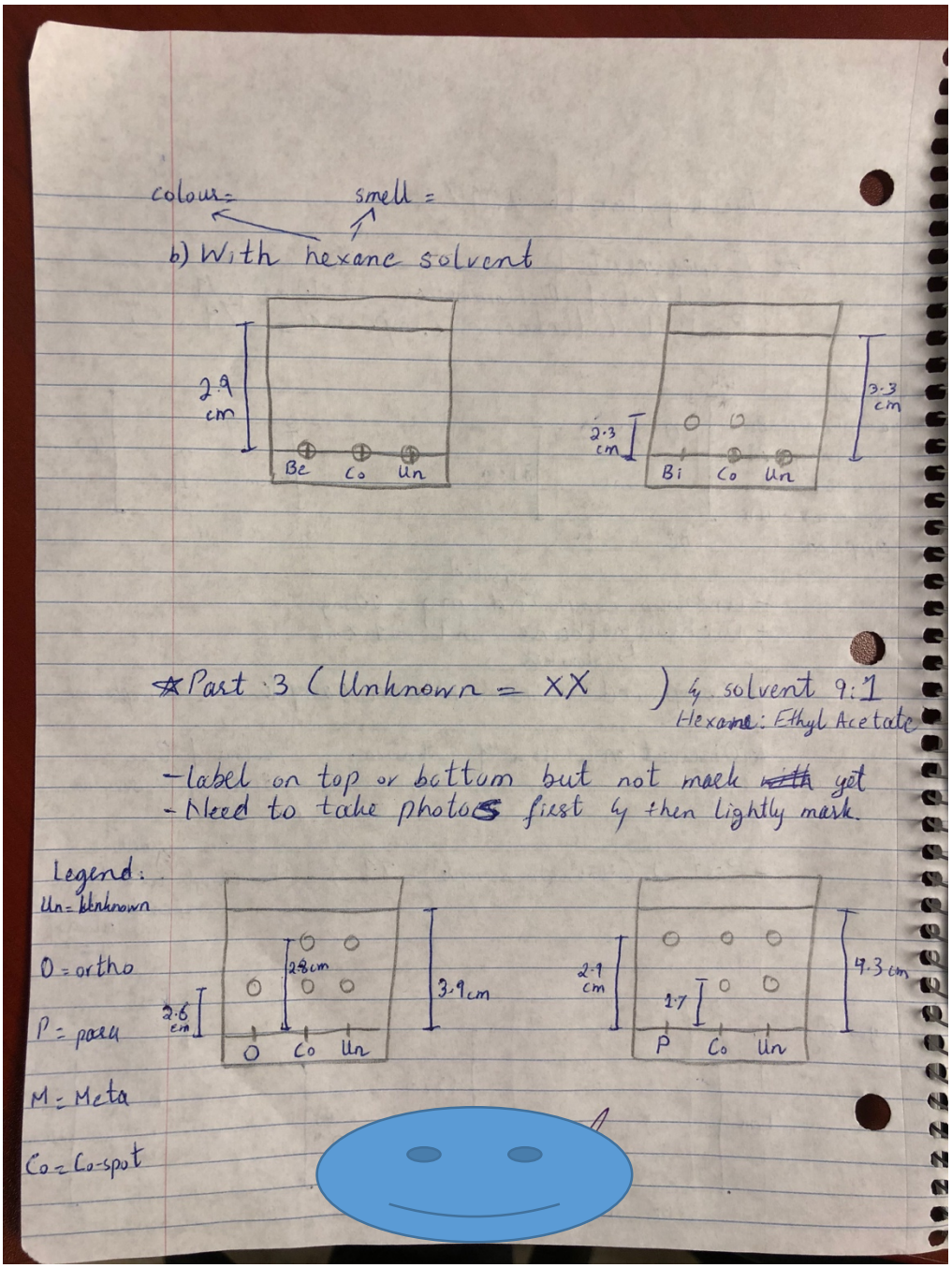
$Ortho = 1.0114(x) - 2.0208$

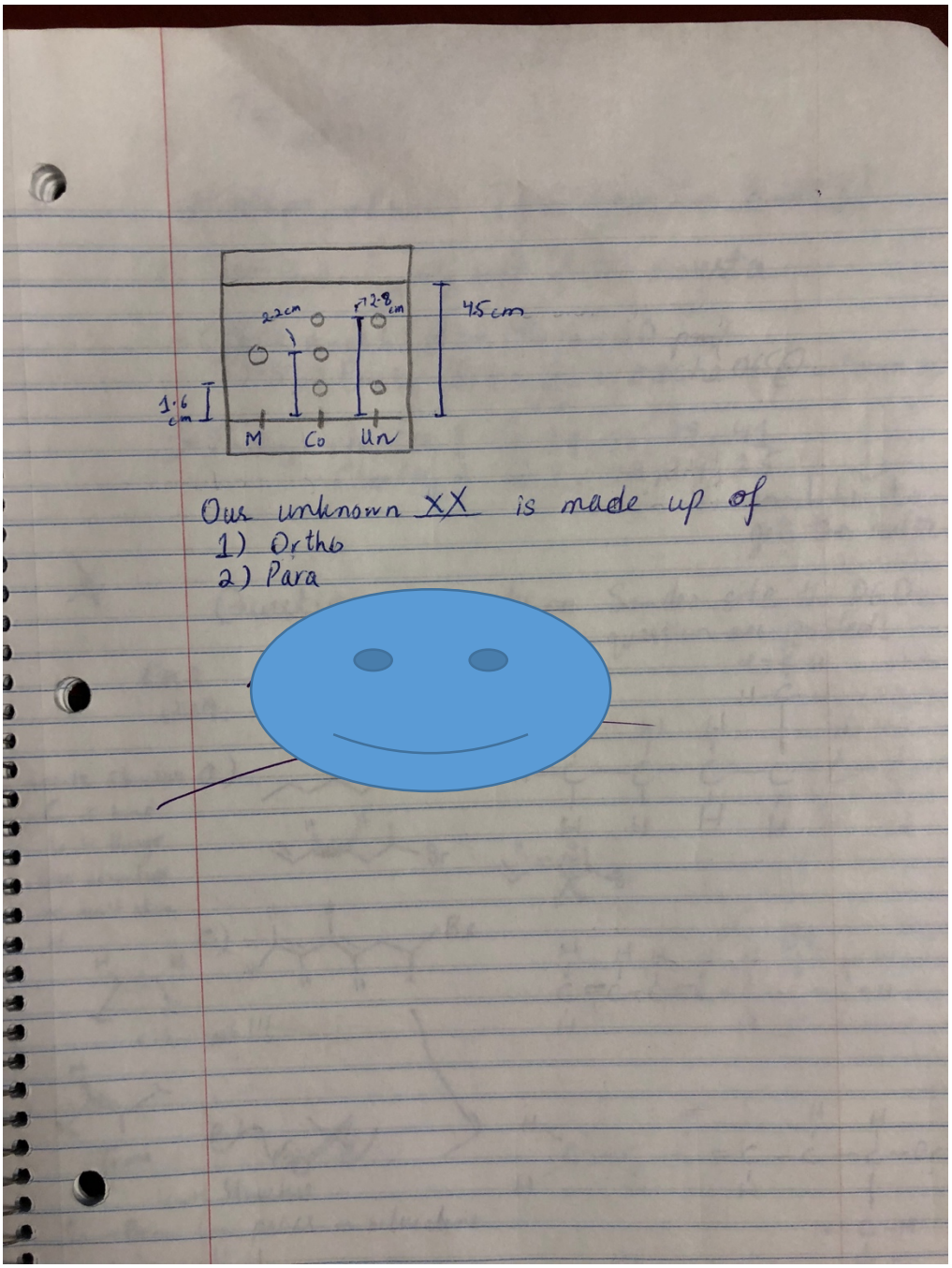
$\frac{43.6 - 2.0208}{1.0114} = x$

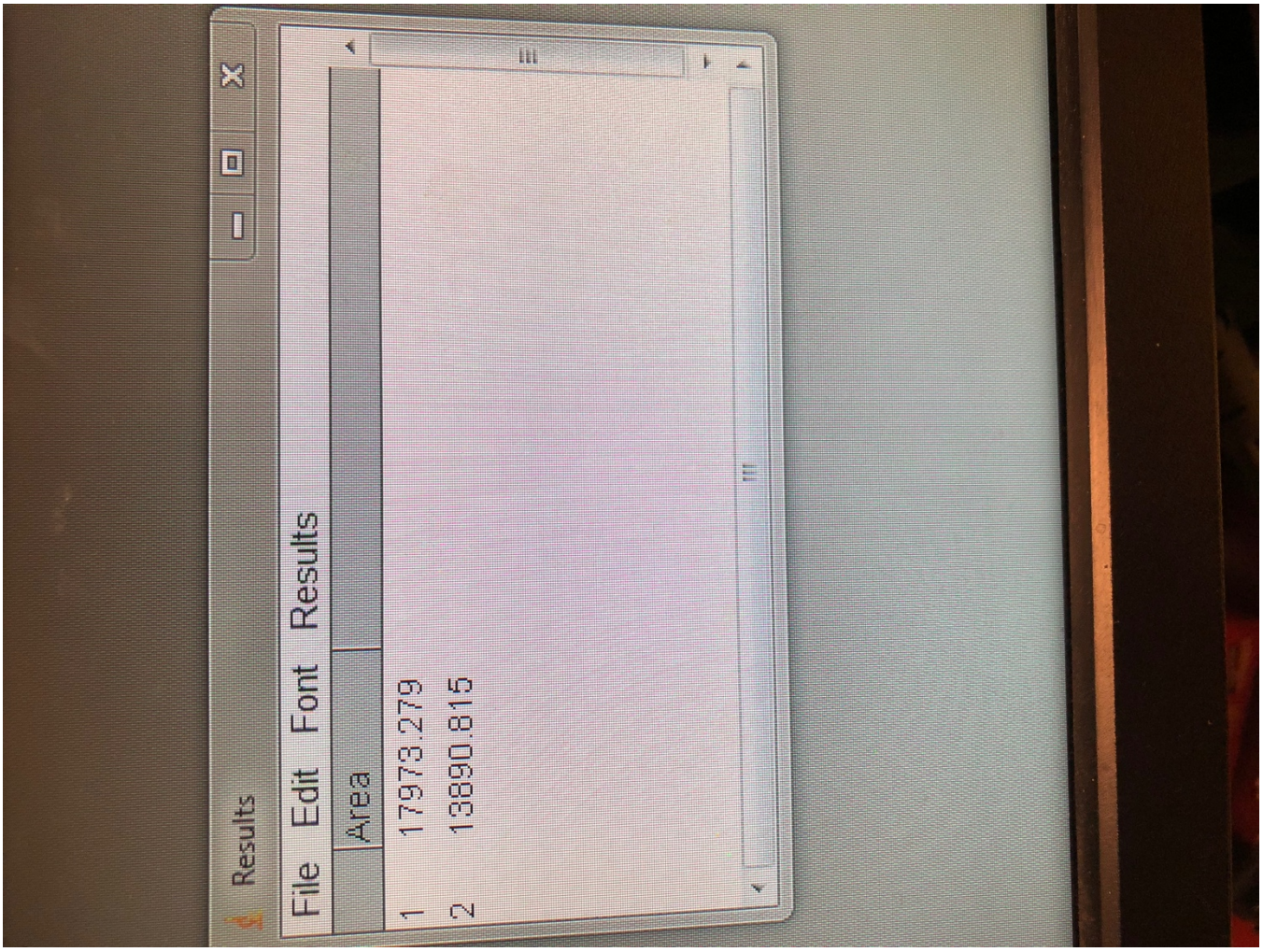
$41.11 = x$ then amount of para is $= 100 - 41.14 = 58.9$

Raw Data


Important reminders
- Do the co-spot last
- Remember to mark Solvent front
- Visualize after evaporate
- Measure distance of solvent front & how much compounds climbed => Imp!!!
Tuesday, Jan 16 2017
☆ Experiment 1
1.7mL of Dichloromethane & 10 mg of 79
- Part 1 (Unknown compound = 79)
Legend
Benzophenone = Be
Biphenyl = Bi
Co-spot = Co
Unknown = Un
compound
4.4 cm
3.1cm
Be Co Un
3.8cm
3.1 cm
4.2cm
Bi Co Un
- Unknown compound (solid) mg = 10mg
- Dichloromethane (colourless) mL = 1.7mL
☆ Compound in our unknown sample = Benzophenone
☆ Observation: Colour of unknown = , Colour of Be =
" " Bi = State of Bi, Be & Un
- Part 2 (Unknown sample = 79) with ethyl acetate (smell of =) solvent (colour of =)
Legend
4 cm
4.3 cm
3.3 cm
4 cm
Be Co Un
Bi Co Un


colour =
smell =
b) With hexane solvent
2.9 cm
Be
Co
Un
2.3 cm
3.3 cm
Bi
Co
Un
Part 3 (Unknown = XX) 4 solvent 9:1
Hexane: Ethyl Acetate
- label on top or bottom but not mark yet
- Need to take photos first then lightly mark.
Legend:
Un = Unknown
O = ortho
P = para
M = Meta
Co = Co-spot
2.6 cm
2.8 cm
3.9 cm
O
Co
Un
2.9 cm
1.7
4.3 cm
P
Co
Un


2.2cm
7.2cm
4.5cm
1.6 cm
M
Co
Un
Our unknown XX is made up of
1) Ortho
2) Para


Results
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Area
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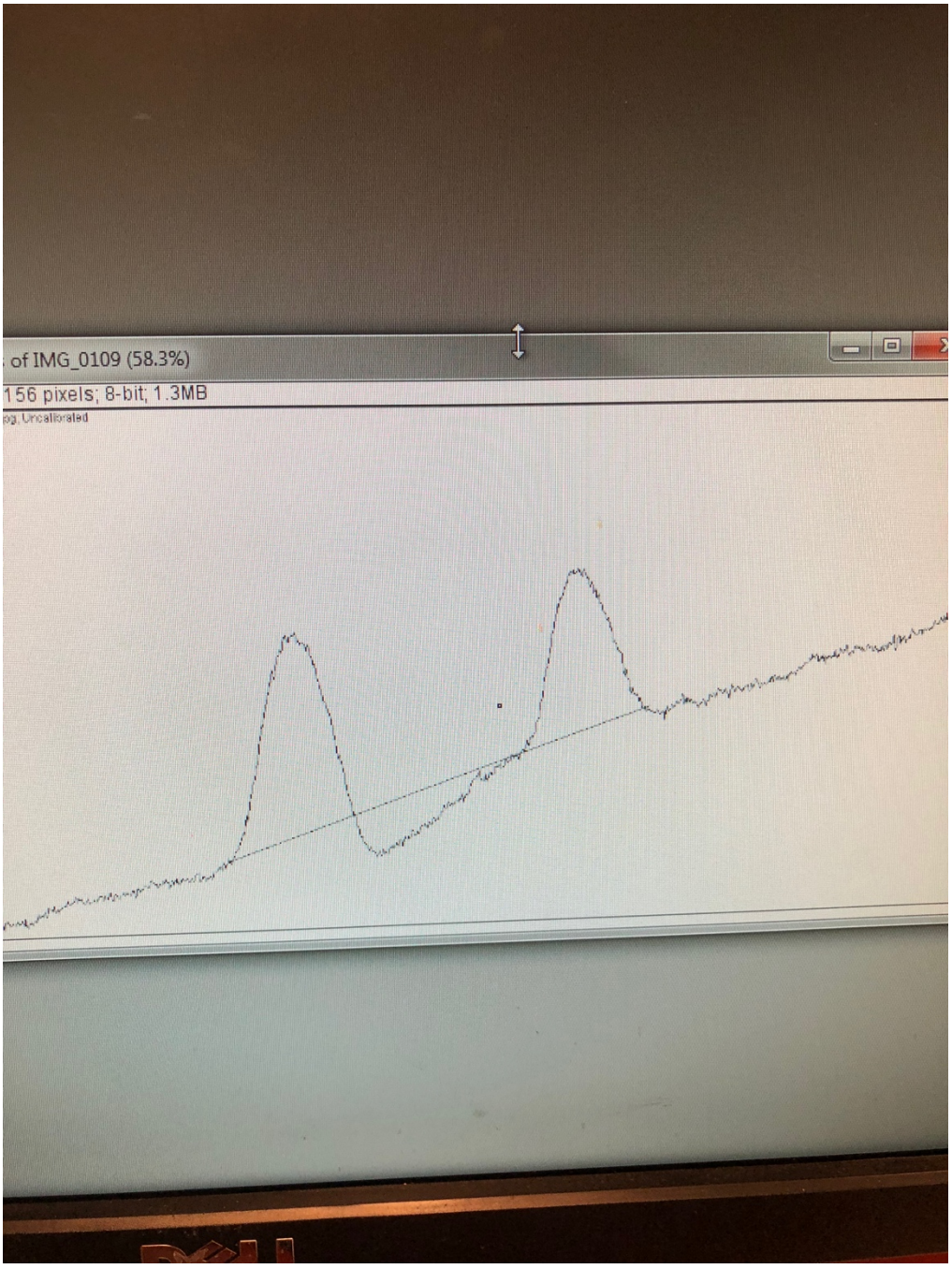
2 13890.815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