Experiment 3:
Extraction

CHM 1321

Name: Alexey Shokun
Student number: 300061608
Lab Partner: Lasindu Perera
Student number: 300072523
CHM 1321 - Z10
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Procedure

Refer to lab manual *Experiment 3: Extraction,* pg.05-06

Observations:

Part A:

1. Dye in both layers.
    a. Ether had a strong odour.
    b. Blue methylene has a blue colour.
2. Red layers in the beginning, dye in both layers.
    a. Yellow colour at the beginning.
    b. Ether had a strong odour.
3. When mixed, light blue, yellow and dark blue layers form.
    a. After shading, every layer is blue.
4. Methyl blue did not mix at the beginning with ether, similar to crystal violet and 1-biphenol.
5. The crystal violet moved completely to the upper layer, so there is a separation between two layers.

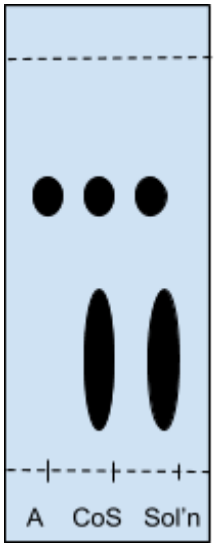
Part B:

1. A little amount of gas escapes each time the top is opened.
2. Shaken for 5-10 seconds, the mixture separates into two layers.
3. Repetitions through the addition of 50 ml of the compound leaded to the same observations.

Weight with filter paper and salt equals 15.38g.
Weight with filter paper equals 14.76g.
Percent composition equals 4%.

TLC's:

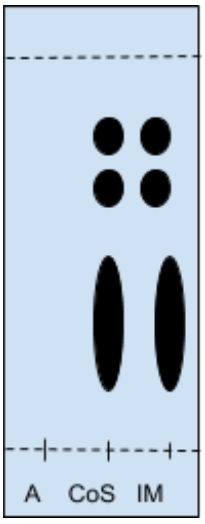
Strip 1: TLC of unknown and organic phase using EtOAc: Hexane (2:8) solvent system.



A - Organic phase
Cos - Co-spot
Sol'n = unknown

Rf of unknown: 0.39, 0.63
Rf of organic phase: 0.63

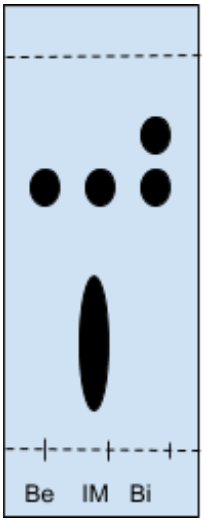
Strip 2: TLC of unknown and aqueous phase using EtOAc: Hexane (2:8) solvent system.



A - Aqueous phase
Cos - Co-spot
Sol'n = unknown

Rf of unknown: 0.38, 0.71, 0.83
Rf of co-spot: 0.38, 0.71, 0.83

Strip 3: TLC of biphenyl, benzophenone and unknown using EtOAc: Hexane (2:8) solvent system.



Bi - Biphenyl
IM - Unknown
Be - Benzophenone

Rf of unknown: 0.71, 83
Rf of biphenyl: 0.71
Rf of benzophenone: 0.71

Calculations

$$\%\ composition = (mass\ of\ benzoic\ acid\ obtained \div initial\ mass\ of\ unknown) \times 100\%$$
$$= 0.76g/15.38g \times 100\%$$
$$= 4\%$$

Discussion

Justification of procedure

- Extraction is a technique commonly used in chemistry to separate chemical compounds in a mixture based on their differing solubilities in two immiscible solvents, which are referred to as phases. One solvent is called the “aqueous phase” and the other the “organic phase.”
- Extractions involve the movement of the desired compound in one phase, while leaving leaving impurities in the other phase. The aqueous phase dissolves polar compounds (inorganic reagents or byproducts), and the organic phase dissolves neutral compounds (the organic products). A separatory funnel is used often in this technique
- A number of extractions were carried out in this experiment, firstly with test tubes and then with the separatory funnel
- An extraction was the correct technique to use to separate the mixtures in both parts *A* and *B*, because they varied in solubilities (one solution was polar the other nonpolar)

Analysis

- In *Part A,* the ether was the organic phase and the water was the aqueous phase (upper layer is the ether because it is less than dense than water).
    - Methylene blue moved into the aqueous phase as seen by the dark blue bottom layer and colourless top layer. This indicates that the solution is polar
    - Methyl red solution moved into the organic phase, which was evident by the yellow tinged upper layer and colourless bottom layer. This indicates that the solution is nonpolar
- Initially placing the contents of the first two test tubes from steps 1 and 2 caused layers to be formed and after shaking vigorously it was evident that once again the methyl red moved into the top layer (yellow tinge in top layer) and the methylene blue moved into the bottom layer (dark blue colour in the bottom layer). This further displayed the differing solubilities of the two solutions. Therefore, an extraction between an ether and water is a good way to separate a mixture of methylene blue and methyl red
- The addition of NaCl to create a “salting effect” was successful which was seen by the violet crystals moving into the organic layer (top layer). This showed that the NaCl effectively saturated the aqueous layer, which pushed the crystals up to the organic phase
- In *Part B,* the 3 TLC plate results indicated a successful separation. Dichloromethane is denser than water, so in this separation, the organic layer was the bottom layer and the aqueous the top layer. In a successful separation, the

biphenyl/benzophenone would remain dissolved in the organic phase while the charged benzoic acid would have dissolved in the aqueous phase. The reaction of NaOH with benzoic acid made sodium benzoate

Source of Error and Possible Improvement

- Sources of error include, inaccurately measuring out volumes of solutions in both parts *A* and *B,* the solutions of dichloromethane, methylene blue, red methyl, etc being slightly impure (will not react properly or as well as it is supposed to), not properly shaking and giving the test tubes enough time to settle before observing the results in *Part A,* broken/torn tubing for the water
- A number of ways to improve the results of this procedure are to have pre-measured solutions to work with, taking extra care in measuring, and weighing all substances, wait 30 seconds before removing the stoppers on the test tubes in *Part A,* check how old each substance and solution is, and examining all equipment and glassware before beginning the procedure

Questions:

1. Why would it be difficult to perform an extraction using acetone and water?
    a. It would be difficult to perform an extraction using acetone and water because these compounds are miscible due to them being polar solvents. For an effective extraction, immiscible compounds should be applied.
2. Would adding NaCl to a test tube containing water, ether and methylene blue increase or decrease the amount of dye in the aqueous layer?
    a. Adding NaCl to a test tube containing water, ether and methylene blue would decrease the amount of dye in the aqueous layer. This result can be observed due to the fact that less dye can be dissolved in the aqueous layer as it becomes saturated by the NaCl. Then, some of the dye will be relocated to the organic layer. This is known as a salting out effect.
3. Compound Y has a solubility of 2.0 g/100 mL in water and 20.0 g/100 mL in ether. What mass of compound Y would be removed from a solution of 1.4 g of Y in 100 mL of water by a single extraction with 100 mL of ether?

    K_D = [A]organic / [A]aqueous
    [A]organic = 0.2g/mL
    [A]aqueous = 0.02g/mL
    K_D= 0.2/0.02 = 10
    10= (xg/100 mL) / ((1.4-x) g/100 mL)
    10 = x/(1.4-x)
    14-10x = x
    14 =11x
    x =14/11
    x=~1.3g

That is, mass of compound Y that will move to organic phase is approximately 1.3g.

4. What mass of compound Y would be removed from the original water solution in question 3 by two extractions using 50 mL of ether each time?

K_D= 10

If x is the mass of the compound Y that will transfer from the aqueous to the organic after the first extraction then,

10 = (xg/50 mL) / ((1.4-x) g/100 mL)

10 = 2x/(1.4-x)

14 = 12x

x= 14/12

x=1.2g

According to the calculations above, mass of compound Y that would be removed from the original water solution after first extraction is 1.5 g. Aqueous layer would contain 1.4g - 1.2g = 0.2g.

If x is the mass of the compound Y that will move from the aqueous phase to the organic phase after the second extraction then,

10 = (xg/50 mL) / ((0.2g-b) g/100 mL)

10 = 2x/0.2-x

2 = 12x

x = 2/12

x= 0.17g

According to the calculations above, mass of compound Y that would be removed after the second extraction is 0.17 g.

5. During an extraction a student loses track of which layer is the organic layer. How could she determine which layer is the aqueous phase?
    a. The student can add a little amount of water to the mixture. The aqueous phase will mix with water, while the organic phase will be immiscible with the water added.
6. Describe how you would separate a mixture of benzyl amine (an organic base) and naphthalene. Both compounds are insoluble in water and soluble in ether.
    a. To separate a mixture of benzyl amine and naphthalene, one can add HCl to react with the benzyl amine, as it is an organic base. This would form a product of a neutralization reaction, salt, that would dissolve in the aqueous phase. The aqueous layer can now be removed. Then, base NaOH, added to the mixture, would cause a reaction with the salt to produce benzyl amine. Finalizing, filtration of this mixture through a suction filter can be performed to obtain the isolated compound.

Raw Data:

Experiment 8.

Observations:

Part A.

Methyl blue did not mix at the beginning with ether, same like crystal violet and 4-biphenol.

Ether and methyl red were mixed at the beginning. (before mixing) physical. (shaking)

Color separated to the top after shaking for violet, the crystal

The crystal violet moved completely to the upper layer, so now, they are completely separated and the lower part has less of a violent color. compared to before added the NaCl due to the increase of an ionic character of water.

Part B.

0.76g of an unknown compound with a number, 101 was obtained. Initial weight of the filter is 15.76g. The final weight is 15.38g. The percent composition is 49%.

Lasindu Perera (300072523)
lpere029@uottawa.ca
9059667334

Feb. 14, 2019.

Experiment #3

Part A

red ① layers ~~mixed~~ in the beginning, dye is in both layers
→ yellow colour at the beginning (red methylene)
→ ether had a strong odour

① ~~in~~ in layers in the beginning, dye is in both layers
→ blue methylene was ~~violet colour~~ blue colour
→ ether strong odour

② when put together, they are in layers, light blue, yellow, dark blue (layers gradually get thinner)
→ after shaking, every layer is blue, top one has a tinge of yellow

Part B

trial #2
- little bit of gas escapes each time the top is opened (evident because of the vapour at the top near the stopcock
- shaken for 5-10 seconds each time (repeated about 3 times)
- split into layers ~~(organic is layer is)~~ (approx. equal thickness)

trial #3
- in layers when poured together, both transparent, ~~(clear)~~ not viscous,
- shaken and stopcock opened 3 times
- split into distinct layers, organic thicker than the aqueous layer

- each drop of HCl caused a cloudy precipitate to be formed
- eventually solid precipitate formed after many drops, this means that the benzoic acid was seperated from the aqueous sol'n

-

- weight w/ filter paper = 14.76g
- weigh w/ filter paper & salt = 15.38g

percent composition = 4%

- water create pressure during the suction filtration process

TLC Plate #1

- o o
  o o o

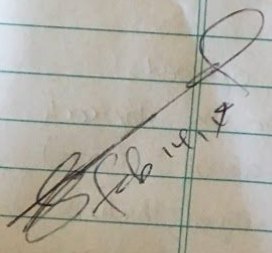
TLC Plate #2

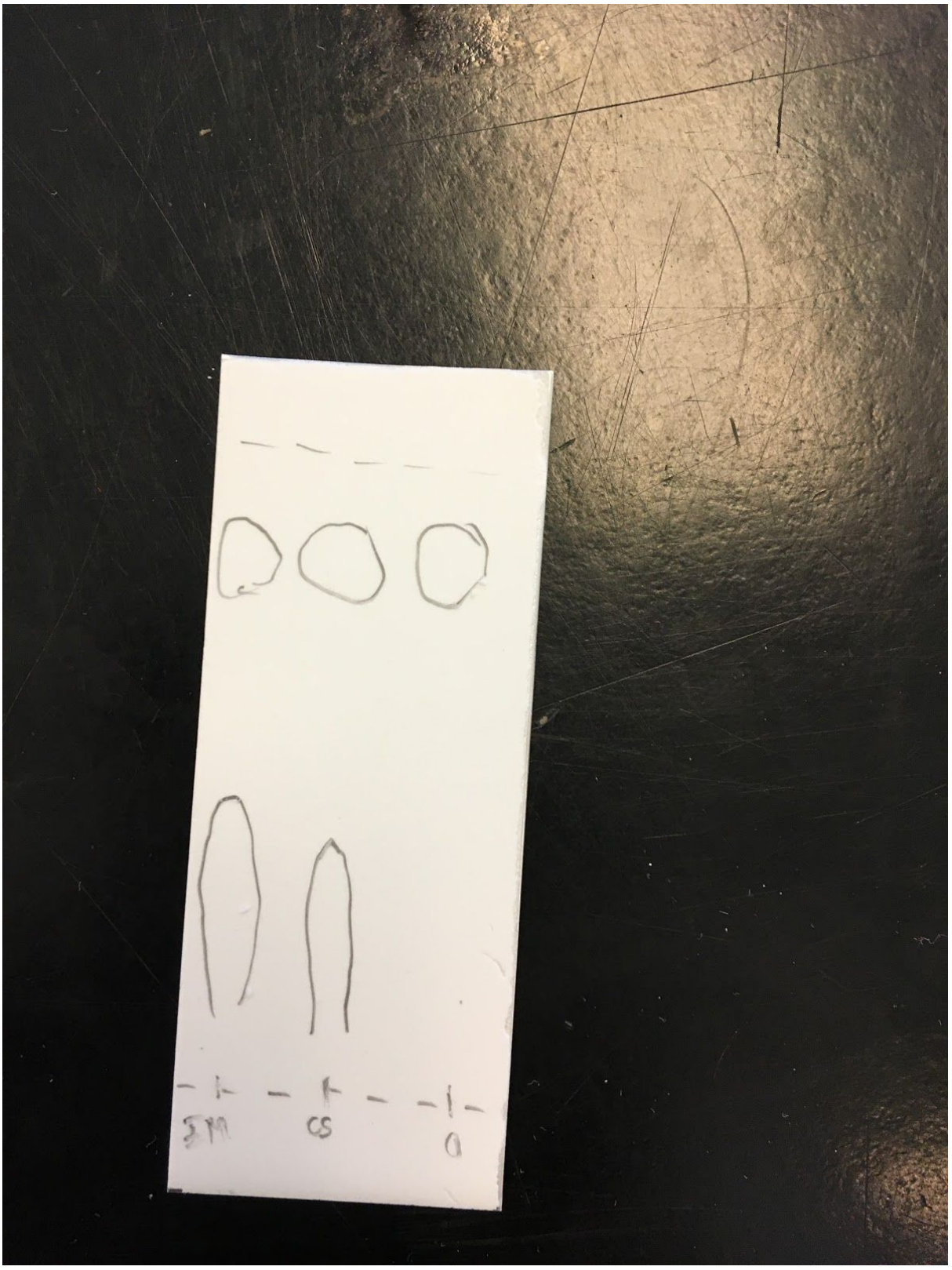
o o o
o o

TLC Plate #3

- says what the unknown is

o o
o o

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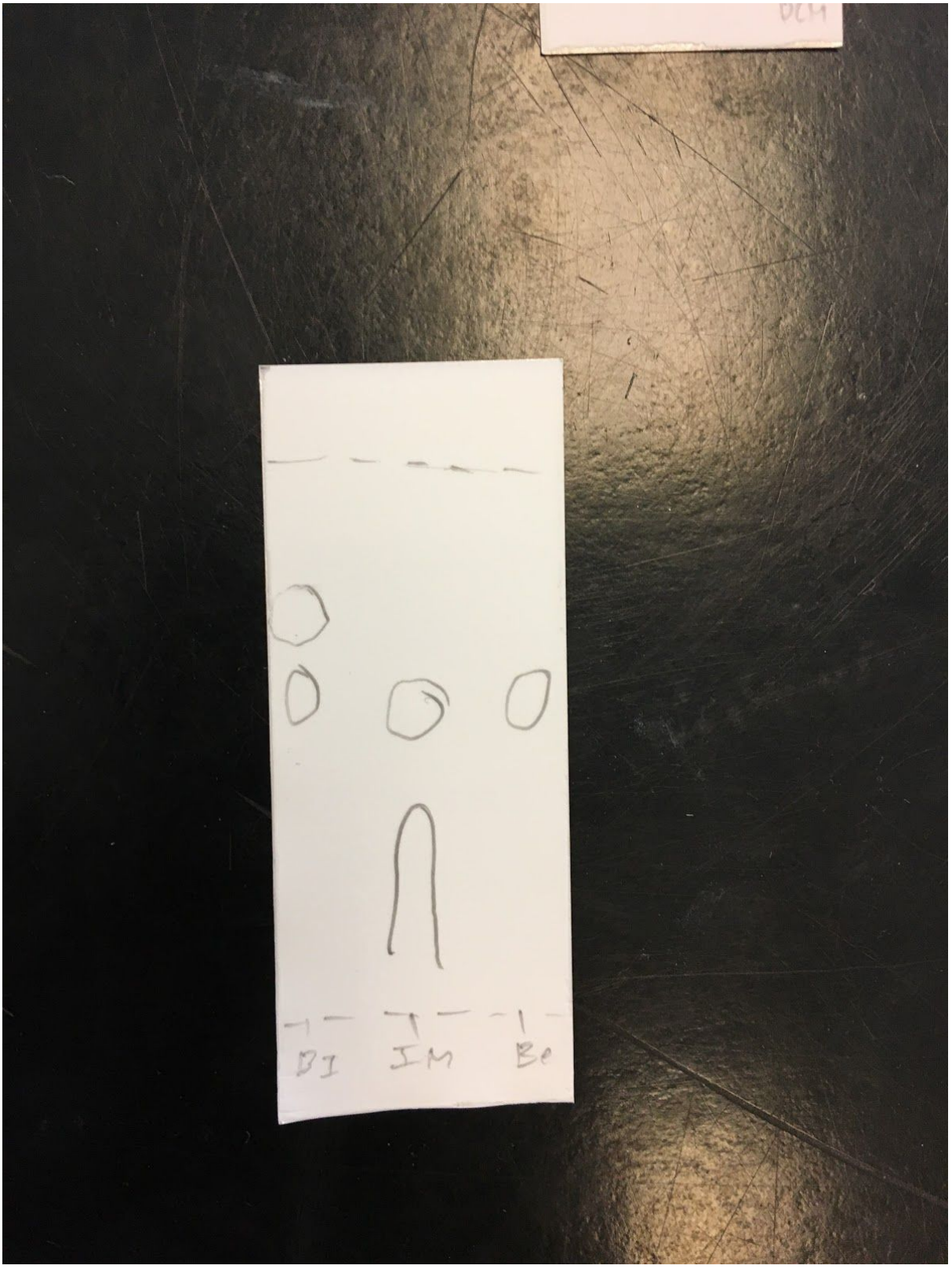
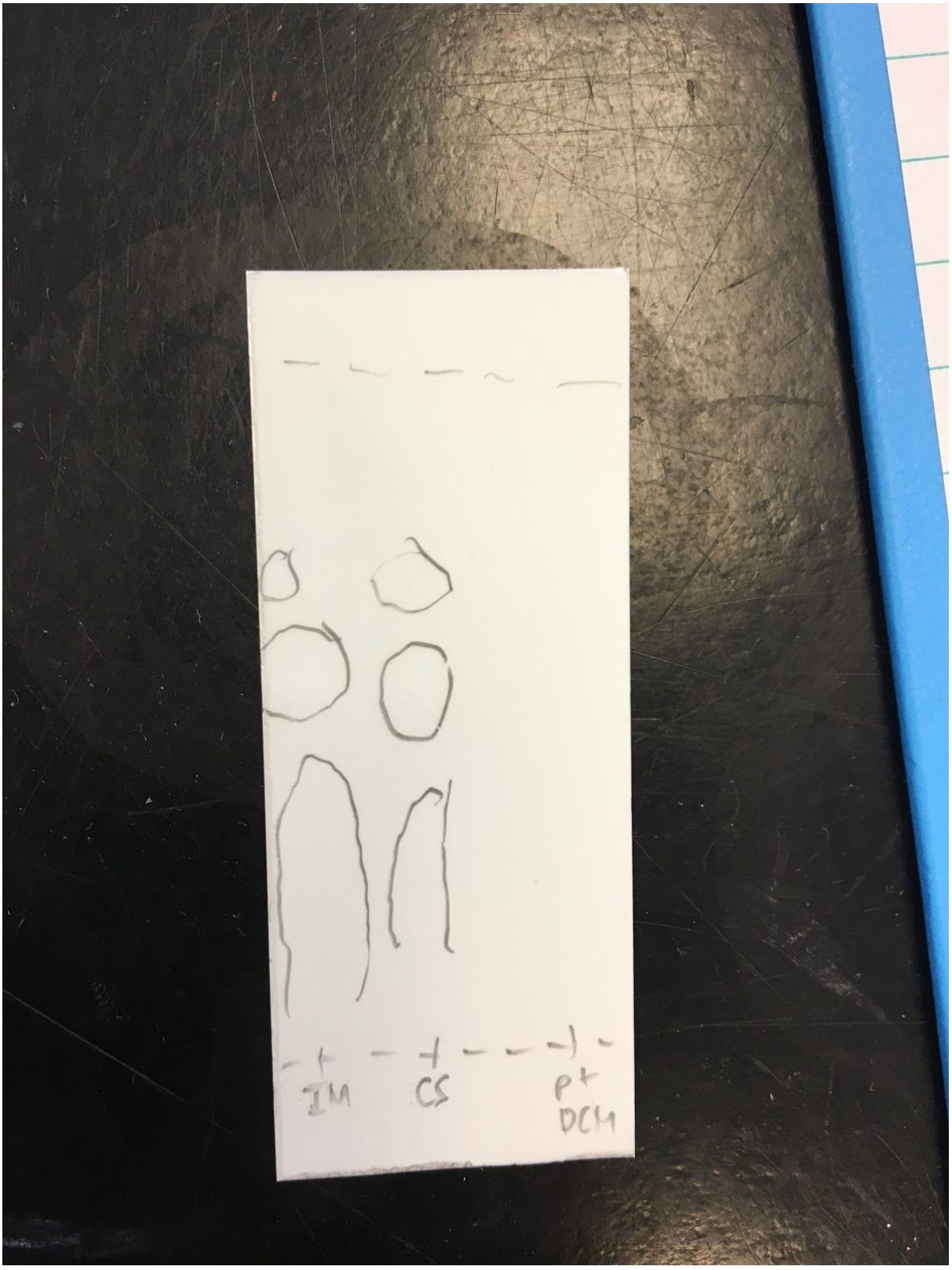

DCM
BI
IM
Be


IM
CS
p+
DCM