SEPARATION TECHNIQUES

ATUL SINGH ARORA

Chemistry

Dr. R Vijaya Anand
Indian Institue of Science Education and Research, Mohali

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Every honest researcher I know admits he's just a professional amateur. He's doing whatever he's doing for the first time. That makes him an amateur. He has sense enough to know that he's going to have a lot of trouble, so that makes him a professional.

— Charles F. Kettering (1876-1958) (Holder of 186 patents)

ACKNOWLEDGEMENTS

I express my sincere gratitude to our instructors, Dr. R Vijaya Anand, for bringing the subject to life and helping us discover, in depth, the science behind the procedures.

I also thank Prashansa Gupta and Srijit Mukherjee for their contribution to this report as my lab-partners, who made the task of performing experiments immensely comfortable and productive at the same time.

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THIN LAYER CHROMATOGRAPHY (INTRODUCTORY)

January 14, 2013

1.1 AIM

TO find the R_f values of the following compounds:

1. Naphthalene (least polar)
2. Benzophenone (partially polar)
3. Aniline (polar)

1.2 CHEMICALS REQUIRED

1. Naphthalene
2. Benzophenone
3. Analine
4. Hexane
5. Ethyl Acetate
6. Iodine
7. Silica

1.3 THEORY

Thin Layer Chromatography is a separation technique that involves the use a rigid porous structure (like that of a silica layer on a glass slide). The mixture is dissolved in a suitable solvent (the precise meaning of suitable will be made clear in a specific example) and a spot of this mixture is made near one end of the structure. This end is now dipped in a suitable solution (again suitable will be defined later), and due to capillary action, this solution begins to move up. When it comes in contact with the spot, the constituents of the spot, differentially move up the structure, allowing us to separate them.

For viewing the different components, some of the techniques used are as follows:

1. UV

2. Iodine Staining
3. $KMnO_4$ Staining

We now define an R_f value for a given concentration of the solution

$$R_f = \frac{\text{Distance travelled by the compound}}{\text{Distance travelled by the solution}} \tag{1}$$

1.4 PROCEDURE

1. Preparing the TLC plates
   a) Prepared a silica slurry, using silica and ethyl acetate
   b) Dipped two glass slides, held together, into the solution, to coat about eighty percent of it with the slurry.
   c) Allowed them to dry (could blow air on it to accelerate the process) and them separated them.
2. Visibility Chamber
   a) Added a few granules of Iodine with silica granules dominant in number, in a beaker, covered with a watch glass.
3. 10% Ethyl Acetate Soln. in Hexane
   a) Using a measuring cylinder, measured 1 mL Ethyl Acetate and the made the volume 10 mL using Hexane. Transferred the contents in a suitable beaker and covered it with a watch glass.
4. Prepare a solution of the given compounds in Ethyl acetate and using a capillary tube, put a spot on the TLC plate, near the silica coated edge. Also, marked physically, by a method suitable, the position of the spot.

In our experiment, we scratched off some silica to mark, using the capillary tube

5. Placed the TLC place, carefully (it's fragile) inside the the 10% Ethyl Acetate Soln., such that the spot is above the level of the solution initially and covered it again, with the watch glass.

We actually had to repeat the experiment as we broke the silica coating!

6. Kept a watch on the TLC and removed it as soon as the solvent crossed about 90% of the height of the silica coating and placed it cautiously in the Visibility Chamber, until the spots became visible.
7. Now marked the lower portion of the visible spots (should ideally be only one, excluding the Ethyl Acetate Solution) and the Ethyl Acetate solution and measured their distances from the initial position of the spot marked earlier.

1.5 OBSERVATIONS AND RESULTS

1. Napthalene: $\frac{4.2}{4.4} = 0.954$
2. Benzophenone: $\frac{3.3}{4.2} = 0.785$
3. Aniline: $\frac{0.7}{4.4} = 0.159$

For details, please refer to

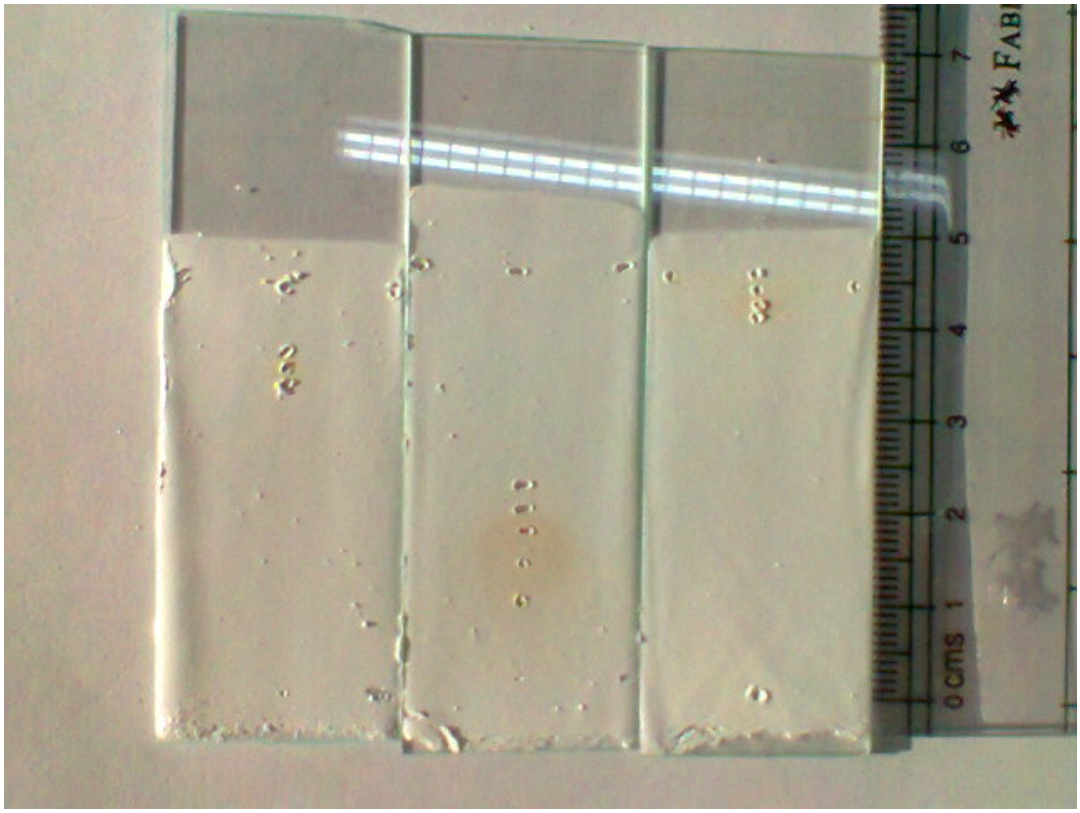

Figure 1

The R_f value decreases with increase in polarity of the compound being analysed.

1.6 PRECAUTION

1. The slurry shouldn't be very thick
2. Cover the beakers with a watch glass to ensure there's no loss of volatile substances (minimal that is)
3. The coating is very fragile, thus the TLC plates must be handled with caution

1.7 ACKNOWLEDGEMENTS

I thank Dr. R Vijaya Anand for his guidance during the experiment. I also acknowledge the contribution of my lab partners, Prashansa and Srijit for performance of the same.

2

THIN LAYER CHROMATOGRAPHY (CONSTITUENT DETECTION)

January 21, 2013

2.1 AIM

To find the constituents of the mixture samples given, which contain two of the following each:

A) Resorcinol

B) p-Hydroxybenzaldehyde

C) 2,4 - Dinitrophenylhydrazine

D) p-Nitro Aniline

and to find the corresponding R_f values, using suitable solvent systems.

2.2 CHEMICALS REQUIRED

1. Resorcinol
2. p-Hydroxybenzaldehyde
3. 2,4 - Dinitrophenylhydrazine
4. p-Nitro Aniline

2.3 THEORY

The theory here is rather straight forward. We already know that the sample contains two components. We, on a TLC, make three spots, the extreme right comprising of only, say A, the middle comprising of A and the mixture, and the extreme left spot, comprising of only the mixture. Now as shown in the diagram, we'll obtain three spots in the middle (called the co-spot), if A is not already present in the compound. If A is present, then only two spots will appear in the middle.

It is the same as saying that the extreme left should have fewer spots than the co-spot if A is not present, in general, and the 'extra spot' in the co-spot, must match with the spot of A on the extreme right.

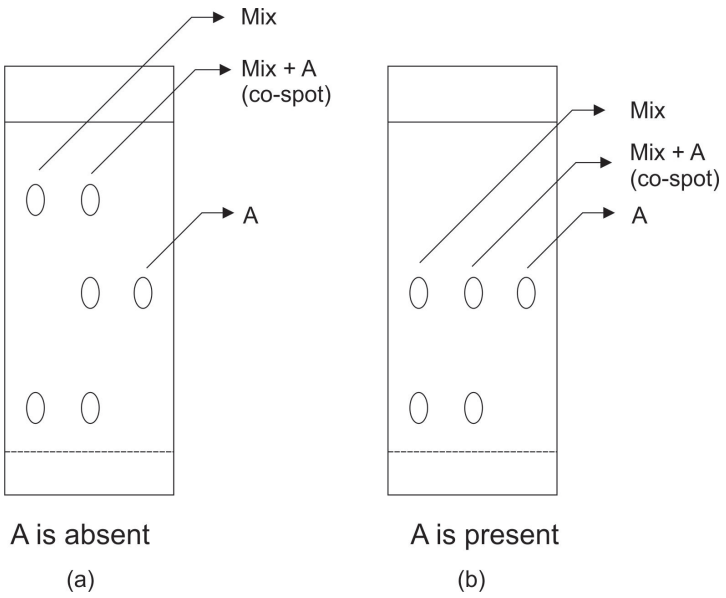

Figure 2

2.4 PROCEDURE

1. Preparing the TLC plates (same as the previous experiment, repeated here for convenience)
   a) Prepared a silica slurry, using silica and ethyl acetate
   b) Dipped two glass slides, held together, into the solution, to coat about eighty percent of it with the slurry.
   c) Allowed them to dry (could blow air on it to accelerate the process) and them separated them.
2. Visibility Chamber (again, same)
   a) Added a few granules of Iodine with silica granules dominant in number, in a beaker, covered with a watch glass.
3. Various concentrations (5%, 10% and 20%) Ethyl Acetate Soln. in Hexane
   a) Using a measuring cylinder, measured the required volume of Ethyl Acetate and the made the volume 10 mL using Hexane. Transferred the contents in a suitable beaker and covered it with a watch glass.
4. Diluted the given solutions of A, B, C, D, Mixture 1 and Mixture 2 appropriately and using a capillary tube, put the spots as described in the theory, using compounds A, B, C and D, one after the other, on the TLC plate, near the silica coated edge. Also, marked physically, by a method suitable, the position of the spot.

Our dilutions were not sufficient the first time we attempted this and we had to redo the entire procedure

5. Placed the TLC place, carefully (it's fragile) inside the Ethyl Acetate Soln. (its concentration was changed progressively in accordance with displacement of the spots), such that the spot is above the level of the solution initially and covered it again, with the watch glass.

We once ended up dipping the spots in the solution itself, which lead to trouble later

6. Kept a watch on the TLC and removed it as soon as the solvent crossed about 90% of the height of the silica coating and placed it cautiously in the Visibility Chamber, until the spots became visible. This is the same as before, except that we setup two chambers simultaneously to speeden up the process.

7. Now marked the positions of all the spots, deduced presence of the substance taken and also calculated the R_f value for A, B, C and D.

2.5 OBSERVATIONS AND RESULTS

Mixture 1 was found to be constituted of compounds A and B, viz. Resorcinol and p-Hydroxybenzaldehyde. In a 20% system, we observed:

1. Resorcinol : $R_f = 0.129$
2. p-Hydroxybenzaldehyde : $R_f = 0.477$
3. 2,4 - Dinitrophenylhydrazine : $R_f = 0.028$
4. p-Nitro Aniline : $R_f = 0.202$

Mixture 2 was found to be constituted of compounds C and D, viz. 2,4-Dinitrophenylhydrazine and p-Nitro Aniline. In a 20% system, we observed:

1. Resorcinol : $R_f = 0.129$
2. p-Hydroxybenzaldehyde : $R_f = 0.422$
3. 2,4 - Dinitrophenylhydrazine : $R_f = 0.085$
4. p-Nitro Aniline : $R_f = 0.259$

For details, please refer to Figure 3 and Figure 4

2.6 PRECAUTION

Precautions are same as those in the previous experiment, viz.

1. The slurry shouldn't be very thick
2. Cover the beakers with a watch glass to ensure there's no loss of volatile substances (minimal that is)
3. The coating is very fragile, thus the TLC plates must be handled with caution

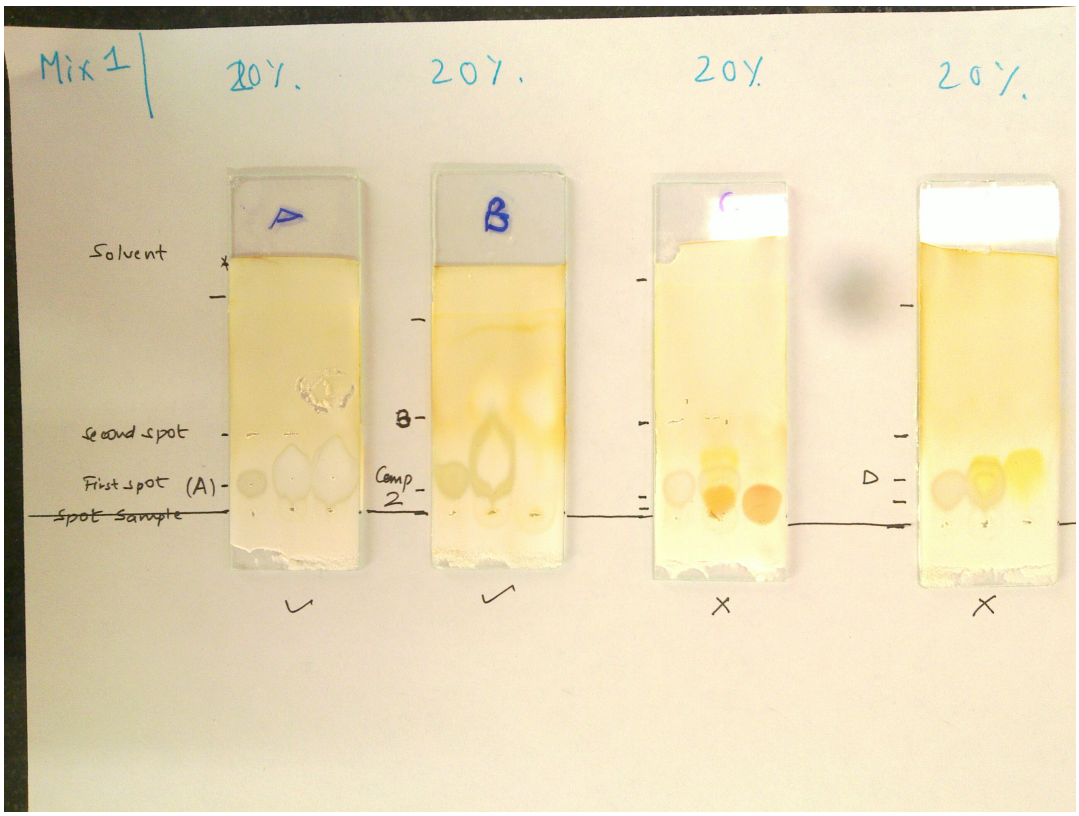

Figure 3

2.7 ACKNOWLEDGEMENTS

I thank Dr. R Vijaya Anand for his guidance during the experiment. I also acknowledge the contribution of my lab partners, Prashansa and Srijit for performance of the same. I especially thank them for maintaining their calm while we were forced to repeat the experiment numerous times.

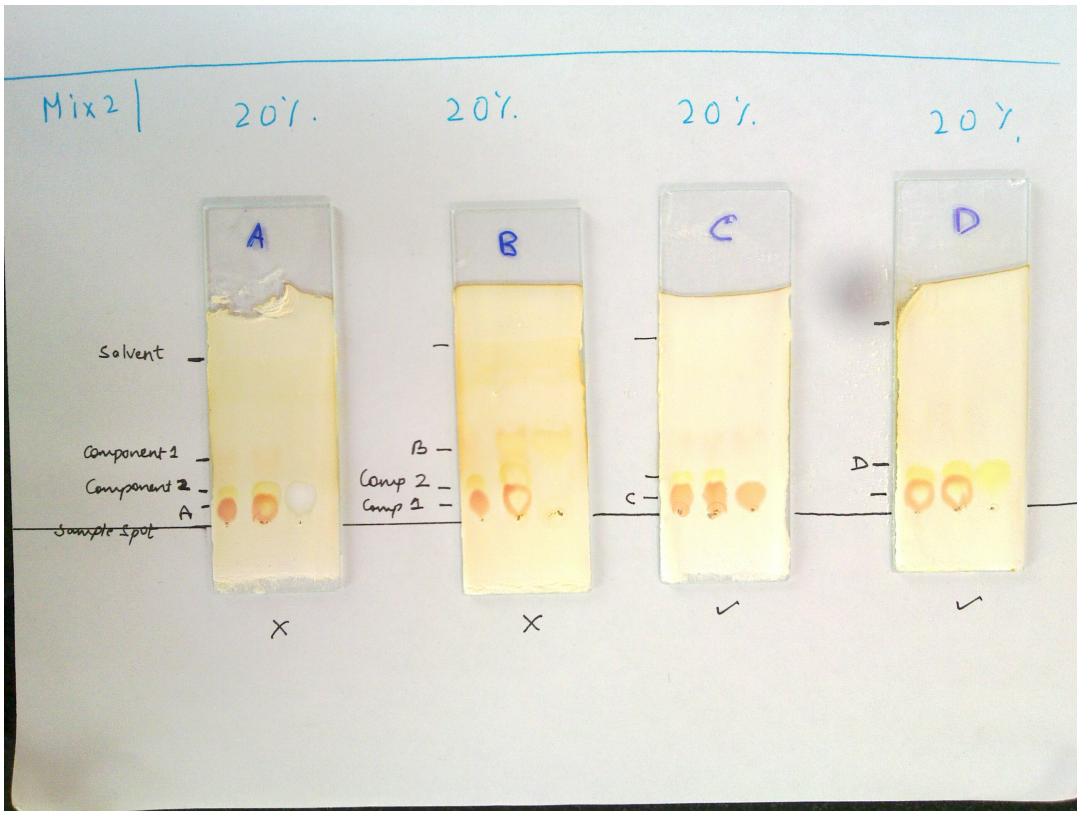
Mix 2
20%.
20%.
20%.
20%.
A
B
C
D
Solvent
Component 1
Component 2
A
Sample Spot
B
Comp 2
Comp 1
C
D
X
X
✓
✓

Figure 4

COLOPHON

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The style was inspired by Robert Bringhurst's seminal book on typography "*The Elements of Typographic Style*".

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