SEPARATION TECHNIQUES

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Chemistry

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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.

CONTENTS

LIST OF FIGURES

LIST OF TABLES

LISTINGS

ACRONYMS

1

THIN LAYER CHROMATOGRAPHY (INTRODUCTORY)

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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

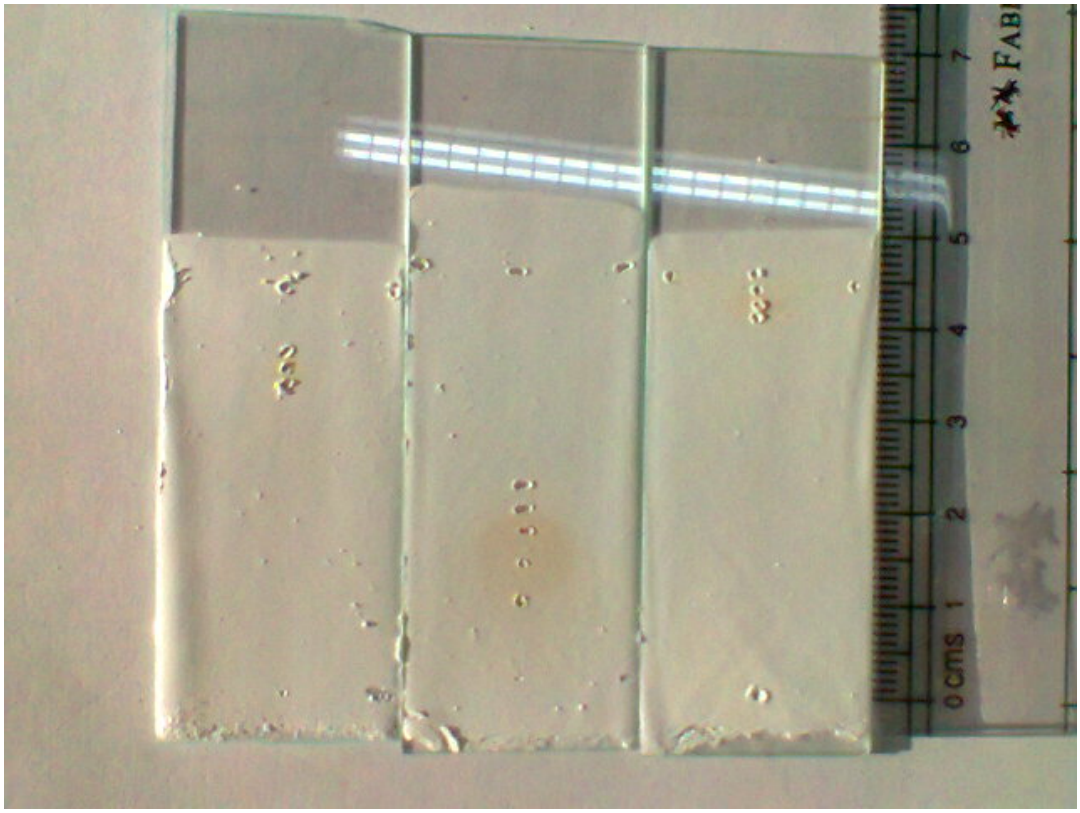

Figure 1

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

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COLOPHON

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https://github.com/toAtulArora/IISER_repo