

Separating a Mixture of Biphenyl, Benzhydrol and Benzophenone by Thin-Layer
Chromatography
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➤ Introduction:

The goals of this experiment was to select an appropriate solvent to separate a mixture of biphenyl, benzhydrol, and benzophenone by thin-layer chromatography. Additionally, identified the specific compounds in an unknown mixture containing any combination of biphenyl, benzhyrol and benzophenone.

Thin-layer chromatography(TLC) is a simple and inexpensive analytical technique that can quickly and efficiently separate quantities of less than ten micrograms of material. TLC is also used for the rapid analysis of reagent and product purity, or to quickly determine the number of compounds in a mixture. Also, by comparing an unknown compound's behaviour to the behaviors of known standard compounds, mixture compounds can be tentatively identified. In TLC, capillary action allows a liquid(mobile phase) to ascend a solid (stationary phase) coated on a support plate. A sample of the compound mixture is applied near the bottom of a dry TLC plate. The plate is placed into a developing chamber, a covered container with a shallow layer of mobile phase liquid in the bottom. As the mobile phase moves to different extents, due to differences in their relative attractions for the mobile and stationary phases. After the separation is complete, the TLC plate is called a chromatogram. During the TLC process, the solid stationary phase, called the eluent, travels up over the adsorbent the compounds within the mixture move at different rates. The ratio of the distance that a compound moves to the distance that the eluent front moves is called the retention factor, denoted as R_f . A calculation for R_f is shown in Equation 1.

$$R_f = \text{Distance traveled by compound, mm} / \text{Distance traveled by eluent front, mm}$$

The first step of this experiment was to obtain 3 beakers and to pour the ethyl acetate, hexane and toluene into the beakers. Put 3 TLC plates into the beakers after labeling the name and the origin points. Obtained the stock solution mixture containing biphenyl, benzhydrol, and benzophenone. The solution was spotted onto the TLC plate for both side. After the eluent level had reached the top, pulled the TLC plates out and let them dry in the air. Examine the developed TLC plates under an ultraviolet light after they were all dry in order to choose the right solvent.

After the appropriate solvent was chosen. Pour the beakers with the solvent and spotted the unknown sample on the TLC. Besides, three different known compounds which were biphenyl, benzhydrol, and benzophenone should also be spotted on the TLC with the unknown sample in each beaker. After the eluent traveled to the top and dried in the air. The TLC plates were examined under the UV lights. The travel spots and the distance from the eluent front to the origin points was measured so that the values of R_f for each compound could be calculated. The calculated values of R_f was used to identify the unknown compound by comparing the R_f value of the unknown compound with that of the known compounds.

➤ Data

Table 1: The separate situation of the ethyl acetate, hexane and toluene as solvent.

Ethyl acetate	Not separate
Hexane	Partly separated
Toluene	Well separated

Table 2: The A and S value of Biphenyl, benzhydrol and benzophenone

Biphenyl

S	A ₁	A ₂	R _{f1}	R _{f2}
6.5cm	6.1cm	6cm	0.9385	0.9231

Benzhydrol

S	A ₁	A ₂	R _{f1}	R _{f2}
6.3cm	6.5cm	3.2cm	1.032cm	0.508cm

Benzophenone

S	A ₁	A ₂	R _{f1}	R _{f2}
6.05cm	5.8cm	1.8cm	0.96	0.2975

➤ Results and Discussion

As shown in Table 1, Toluene was chosen to be the appropriate solvent because it best separates biphenyl, benzhydrol, and benzophenone among the three tested solvents. In the second part of the experiment, biphenyl was determined to be the identical compound to the unknown because the R_f value of the unknown was very close to the R_f value of biphenyl, with an error percentage of 1.64%. The most likely explanation for the difference in R_f values is that the travel spots were not regular circles, so the middle points were not indicated accurately.

➤ Post-Lab Questions:

3. (a) The eluent was harder to travel so that the R_f value may be smaller than it should be.
(b) The eluent can be traveled too much than it should be.
(c) The eluent may overcome the length of TLC which will let the R_f value be smaller than it should be.
(d) The eluents may have not been separated yet. The R_f value may be smaller than it should be.
(e) The eluents may be evaporate. The eluents may not travel as far as it should be. In that case, the R_f value is smaller than it should be.
4. Using appropriate solvent which can separate benzophenone and benzhydrol. Spot the sample which needs to be tested and the known benzhydrol. If the R_f value of the unknown compound was the same with the pure benzhydrol, it means that the reaction has been done.