

## **TLC analysis of benzhydrol produced by a Grignard reaction**

Before recrystallizing your product from hexanes, be sure you save a small amount of the crude product (tip of a small spatula) to run the TLC experiment below and to measure the melting point.

TLC (thin layer chromatography) experiment:  
reading: technique 10 (p 813-826)

- Dissolve a small amount of your crude product in about 1 mL (1/2 pipette full) of diethyl ether. Do the same with your recrystallized product.
- Get 3 TLC plates and draw a line approximately 1 cm from the bottom of each plate with a pencil.
- On each plate you will make 4 spots: biphenyl standard, benzhydrol standard, crude benzhydrol product, and recrystallized benzhydrol product.
- To make a sample spot, take a capillary tube and place it in your solution. A small amount of the solution will go into the capillary. Lightly touch the capillary containing your sample to the TLC plate and remove it quickly. Blow on the plate to dry and repeat 1-2 times. Use the UV lamp to be sure visualize the spots. If your spot is faint, repeat spotting a few more times. Your spots should be placed on the line you drew and be as small as possible.
- Place hexanes in a beaker that is taller than your TLC plates. The hexane should be enough to cover the bottom of your beaker, but not deep enough to be above the line on your TLC plates. Stand a piece of filter paper in the beaker (see book). Cover the beaker with a watch glass and wait for a minute.
- Carefully place one of your TLC plates in the beaker with the spots towards the bottom. Allow the solvent to travel almost to the top of the plate, but not all of the way. When the solvent is near the top, remove your plate and quickly mark the top of the solvent front. Mark the TLC plate with the solvent you used--hexanes.
- Repeat the above process with the second TLC plate using a 9:1 mixture of hexanes and ethyl acetate. The mixture is most easily made in a 10 mL graduated cylinder. Again mark the solvent front and label the TLC plate with the solvent system.
- Finally run your third TLC plate, as above, but using a 1:1 mixture of ethyl acetate and hexanes.
- Use a UV lamp to visualize the spots. The spots will be blue on a green background. You may also treat the plate with iodine vapor (place it in the iodine chamber) visualize the spots. Circle each spot you observe with a pencil.
- Keep the TLC plates and attach to your report.
- Determine the R<sub>f</sub> value for each component and report the results in your lab report.