

## **Synthesis of Dibenzalacetone: Thin Layer Chromatography**

Review the procedure for TLC in this Manual. You will need a pencil to mark the TLC plates.

A TLC can be performed before the product is completely dry. In a small test tube, dissolve ~30 mg of the product in acetone. Do the same for benzaldehyde (convert 30 mg benzaldehyde to volume). In a third small test tube, combine a few drops of the solutions from the first two test tubes to make a mixture of benzaldehyde and dibenzalacetone.

Prepare a TLC silica gel plate by lightly drawing, with a pencil, a horizontal line about 1 cm from the bottom edge of the plate and marking 3 small x's equidistant from each other and the vertical sides of the plate.

Spot one lane with benzaldehyde ("B"); the second lane with the mixture ("M"); and the third with the product dibenzalacetone ("D"). Develop the plate using a 10:90 ethyl acetate:hexane developing solvent. Develop the chromatogram until the solvent is within 1.5 cm of the top of the plate. Remove the plate carefully from the chamber and immediately mark the solvent front line with a pencil. Allow the plate to dry on a paper towel at your bench. To visualize the spots, place the dry plate in an iodine chamber.

Remove the plate from the iodine chamber and immediately trace the outline of the dark spots with a pencil. Be careful not to chip the coating on the plate.

Measure the distance the spots traveled and then calculate the  $R_f$  for benzaldehyde and dibenzalacetone. Sketch your developed TLC plate in your notebook. Comment on the presence of any spots other than your product in the lanes where you spotted it.

Submit your chromatogram to your instructor.