

## Purification of Biphenyl – 2B: Chromatography and Analysis

**Question:** What is a good solvent system for purification of biphenyl by column chromatography? Is column chromatography a more or less effective purification technique than recrystallization for our biphenyl mixture?

**Procedure:**

Thin Layer Chromatography (Read Technique 18 thoroughly) In the first part of the lab, you will analyze a crude sample of biphenyl by TLC and determine an appropriate solvent system for purification of biphenyl by column chromatography. You will be given 6 TLC plates for the entire lab (both parts). This is enough plates, but since this is the first time many of you have performed this technique, I suggest that you work in groups of two or three for this part of the lab, in case any TLC's need to be repeated. *Make sure to draw all of your group's developed TLC plates in your lab notebook so that you can calculate  $R_f$  of both compounds in all three solvent systems.*

Devise your own procedure for this part, and make sure you do this before entering the lab (see Technique 18.7). Determine  $R_f$  values for biphenyl and the impurity in each of the following solvent systems: 100% hexanes, 100% ethyl acetate, and 4:1 hexanes:ethyl acetate. Together with your group, determine an appropriate column chromatography solvent system.

Column Chromatography (Read Technique 19.5a thoroughly) Using a long Pasteur pipet (220 mm), insert a plug of glass wool loosely, but firmly in the tip of your pipette column. Fill the pipet column about 2/3 full with silica gel, then add eluent and allow it to drain through the silica gel until it is evenly wet. Dissolve 50 mg of impure biphenyl in 2 drops of acetone and apply them to the top of the column. Once the sample is drained onto the column, fill the pipet with eluent solvent and collect four 1 mL fractions. (You can speed up the process by gently applying air pressure with a pipet bulb—ask your AI for details.) Test for biphenyl in the fractions by running a TLC plate against authentic biphenyl using the appropriate eluent solvent. If you see no biphenyl-containing fractions, or if your biphenyl is still coming off the column (present in the 4<sup>th</sup> fraction), continue to elute and collect fractions.

Isolation of purified biphenyl For every fraction that contains only biphenyl, evaporate by pouring onto a *pre-weighed* watchglass in the hood. Once one fraction is evaporated, add the next pure fraction until all the biphenyl is dry on the watchglass. Obtain a final mass of isolated biphenyl and calculate your percent recovery.

Melting point data Obtain three melting points: impure biphenyl, recrystallized biphenyl, and column purified biphenyl. Record these in your notebook.

TLC data Spot a TLC plate with four lanes: authentic biphenyl, impure biphenyl, recrystallized biphenyl, and column purified biphenyl. Develop the solvent in 4:1 hexanes/ethyl acetate. Visualize the spots using UV light. Draw your developed TLC plates in your notebook.

**Observations and Results:** Make observations of chemicals used and what you see happening during the experiment. Include the following: tabulated  $R_f$  data; tabulated melting point data; drawing of TLC plate from column chromatography; drawing of TLC plate with four samples (authentic biphenyl, impure biphenyl, recrystallized biphenyl, and column purified biphenyl); mass of purified biphenyl and percent recovery (including calculation)

**Discussion Comments:** How effective was the column at purification in terms of recovered percent yield and level of purity of the sample? What data support your conclusions? How is melting point data useful? How is TLC data useful?

What are the pros and cons of recrystallization versus column purification? Which gave a higher percent recovery? Which gave a purer final sample? Which accommodated a larger sample? Which took longer? Are there any other considerations when choosing between recrystallization and column chromatography?

**Conclusion:** Write two sentences about the effectiveness of your recrystallization versus column chromatography.

**Reference(s):** References for any necessary authentic data, such as melting points

**Lab 2b assignment: Turn in a hardcopy of your lab notebook and “Tables and Figures” in a formal format**

Due at the beginning of lab next week

- 25 pts based on in-lab performance, notebook, and formal Tables and Figures
- Data for your figures and tables can be taken from experiments 2a and 2b. Choose sets of data that would be effectively communicated in these three formats”
  - A figure (Labeled “Figure 1” underneath) with a caption. This figure should be of your TLC plate, made with ChemDraw. See instructions on the website for making TLC plate figures with ChemDraw.
  - A scheme (Labeled “Scheme 1” above) with effective title. This Scheme shows structures of the compounds with which you worked. Label compounds with bold numbers underneath. Make sure that the scheme communicates an idea—it is not enough to simply draw all the structures. For instance, are there structural features of the solvents you tested for recrystallization that group them into “effective” and “ineffective” solvents?
  - Two tables (labeled “Table 1” and “Table 2” above) with an effective title and headings. These tables should efficiently and effectively show sets of data that would be communicated with a table. For instance, don’t
- See “Writing a Formal Report” for more information
- There is no electronic submission for this week.