

## Purification of Biphenyl – 2A: Solubility and Recrystallization

### Question:

What solvent can be used for an effective recrystallization of biphenyl? How well does it work?

### Procedure:

1. Using test tubes, test the miscibility of 0.5 mL of each of the following solvents with water: methanol, acetone, dichloromethane, toluene, and hexanes. If immiscible, determine which layer is the top layer in each case.
2. Based on Technique 15.4 from the Mohrig textbook, test how suitable each of the following solvents are for recrystallizing biphenyl: water, methanol, acetone, dichloromethane, toluene, and hexanes.
3. Weigh about 0.5 g of crude biphenyl, which contains methyl orange dye as an impurity, into an Erlenmeyer flask with a stirbar. Determine the exact mass of biphenyl used. Add hot hexanes until the no more solid dissolves and only impurities remain. Filter the impurity using suction filtration into a 125 mL sidearm flask (or talk to your AI about alternative filtration procedure). Transfer the liquid into a small beaker, and evaporate the hexanes by blowing air or nitrogen over the solution until only the partially purified biphenyl is left.
4. Using the solvent you determined in the previous experiment, barely cover the solid biphenyl in the hot solvent. Be sure to maintain the temperature of the hot solvent while allowing the crystals to dissolve. Add more of the hot solvent dropwise to the biphenyl is completely dissolved. Remove the flask from the heat and allow to cool to room temperature. (Note: if you chose a low-boiling solvent, you will not want to let it cool at r.t. for very long, as you will lose solvent due to evaporation). Place the flask in ice to maximize recrystallized product.
5. Isolate the recrystallized biphenyl by suction filtration. Use ice cold recrystallization solvent to rinse the flask and wash the recrystallized biphenyl. Pull air through until the crystals are dry. Obtain a mass of recrystallized biphenyl and save it for next lab period in a labeled container.

**Observations and Results:** Make observations of chemicals used and what you see happening during the experiment. Record all masses with adequate labels so that you know what you weighed. Use tables where appropriate (i.e. miscibility and solubility, but not a single mass number)

**Notebook Discussion Comments:** Comment on the water solubility of the organic solvents you tested. Considering the structure of biphenyl, was its solubility in the solvents what you would expected based on theory? Which solvent did you choose as a recrystallization solvent? Why?

What was the purpose of adding the hot hexanes to the crude product? Based on its structure, why did the dye not dissolve? How effective was the recrystallization in terms of percent recovery?

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

**Reference(s)** References for any necessary authentic data, such as densities

**Lab 2a assignment:** Turn in a hardcopy of your lab notebook (carbon copy or photocopy)

- Due at the beginning of lab next week
- 25 pts based on in-lab performance, completion of all sections, correctness of content
- No electronic submission to turnitin.com