2. Purification of Solids by Recrystallization


Techniques Checklist:
- Solubility tests
- Choosing a good solvent system
- Inducing crystallization
- Filtration

Pre-Lab Discussion and Required Reading:
- Theory of recrystallization: Zubrick Ch. 13, LLP Ch. 11.2

Digital Lab Techniques Manual:
- 9. Recrystallization

Equipment:
- Test tubes (five 13x100 mm)
- Erlenmeyer flasks (2x50-mL, 1x125-mL)
- Small magnetic stir bar
- Funnel
- Filter paper
- Büchner funnel and filter paper
- Magnetic stirring/hot plate
- Filter flask (250-mL)
- Rubber filter adapter
- Large vial with white cap
- Test tube rack
- Large crystallizing dish
- Desiccator
Goal:
You will be given 2.00 g of impure naphthalene (mothballs!). Your job is to purify the naphthalene by recrystallization without losing a significant amount of your sample!

Experiment Outline:
I. Solubility Tests

- Determine an appropriate solvent system for the recrystallization of naphthalene. For your tests try: water, methanol, acetone, hexane, and toluene. To understand how to find the appropriate solvent or solvent mixture for recrystallization, see pages 104–105 in Zubrick.

II. Recrystallization of Naphthalene

- Transfer the material to a 50-mL Erlenmeyer flask equipped with a stir bar. Add about 20 mL of the solvent (determined in Part I) and heat to boiling on a stir/hot plate.
- Remove any insoluble impurities by filtration, and recrystallize your product - see Two-Solvent Recrystallization Guide.
- Collect your crystals on a small Büchner funnel by vacuum filtration, and rinse with the cold solvent mixture.
- Your crystals should be colorless. If some orange or yellow color persists, wash your material with cold hexane. (Be careful: What is the solubility of naphthalene in hexane?).
- Dry your compound well - see Two-Solvent Recrystallization Guide for tips.
- Determine the yield, and obtain a melting point.

Results:

- To obtain your "CC Rating" in Purification of Solids by Crystallization, you must obtain colorless, well dried crystals weighing at least 1.30 g (no traces of yellow!). The purified material must melt over no more than three degrees with the lower range beginning no lower than 77 °C and the upper range ending no higher than 83 °C. This material must also be submitted to the TA for possible weight and melting point verification.

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