2. Purification of Solids by Recrystallization

2.1. Competent Chemist Rating: *"How do you recrystallize a Mothball?"*

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:

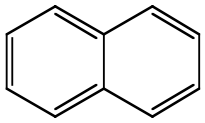
- 9. Recrystallization

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![1]



Naphthalene

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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[1]Adapted from Fieser, L. F.; Williamson, K. L. *Organic Experiments;* 7th ed.; D. C. Heath and Company: Lexington, MA, 1992; p. 40.