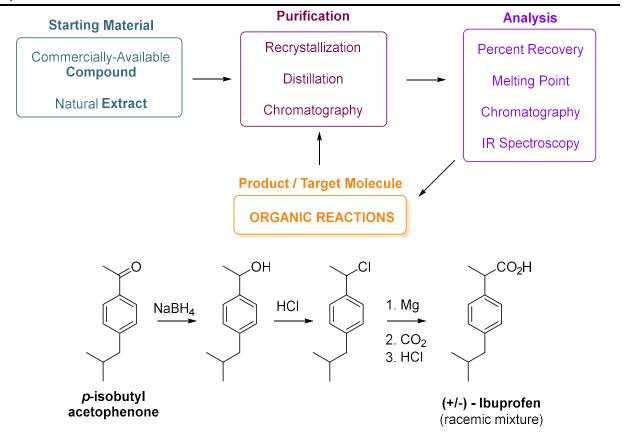
### CHEM 8L, Lecture 1 - Recrystallization

Course Introduction & Policies
Experiment 1 – Purification of Acetanilide



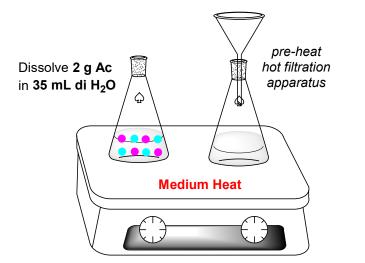
Kjonaas, R. A., et. al. J. Chem. Ed. 2011, 88, 825 – 828.

### **RECRYSTALLIZATION** → purification of solids based on solubility

- Dissolve solid in solvent, remove insoluble (  $I_i$  ) and soluble (  $I_s$  ) impurities
- Precipitate (crystallize) solid from solution & isolate via filtration
- Analyze by percent recovery and melting point (Lecture 2)

### **Experimental Procedure**

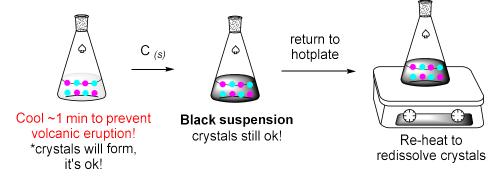
- (1. Choose recrystallization solvent...WATER!)
- Best theoretical % recovery (Lecture 2): high solubility in hot solvent, low solubility in cold solvent
- 2. Dissolve sample in *minimum volume* of hot deionized water (di H<sub>2</sub>O)



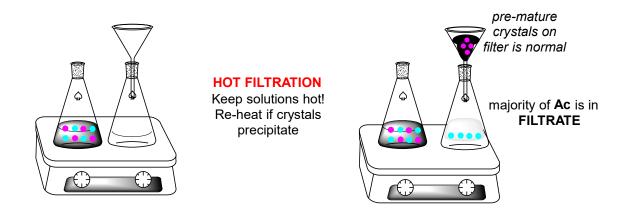
## **SAFETY FIRST**

Use 2 hot mitts to handle hot glassware

3. Add activated charcoal, C<sub>(s)</sub>



4. Remove I<sub>i</sub> via hot filtration

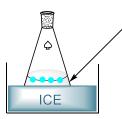


# 5. Cool to induce crystallization



Multi-task!
Set up cold filtration
apparatus while
crystals form

Cool on bench maybe crystals, maybe not



Cool in ice bath

CRYSTAL LATTICE formation, based on hydrogen-bonding patterns (H-bond)

6. Remove Is via cold filtration

- 7. Wash with cold di H<sub>2</sub>O
- 8. Dry to remove solvent from solid

**COLD FILTRATION by vacuum**Keep solutions cold!



9. Percent Recovery & Melting point analysis (Lecture 2)

### **Recrystallization Procedure Overview**

### This Week = First Lab Meeting

- Arrive on time & dressed for lab (see dress code in SAFETY RULES online)
  - 15+ min late or not dressed = dropped from course 🔾
- No experimentation, no assignments due before lab
- Intro Packet: Safety, Writing, & Error Analysis
  - o Provided in lab, skim online before lab to prep
- Optional, encouraged: Bring your blank notebook to start preparing for Exp 1

Exp 1 performed week 2 - pre-lab Q's & notebook due

 Exp 1 (intro, procedure, pre-lab, post-lab Q's, report cover page) on <u>acrochem.sites.ucsc.edu</u> > CHEM 8L

Please read **syllabus** and go to '*Preparing for the First Day*' tab of acrochem site for answers to questions on course policies, wait-lists, late adds, etc.