

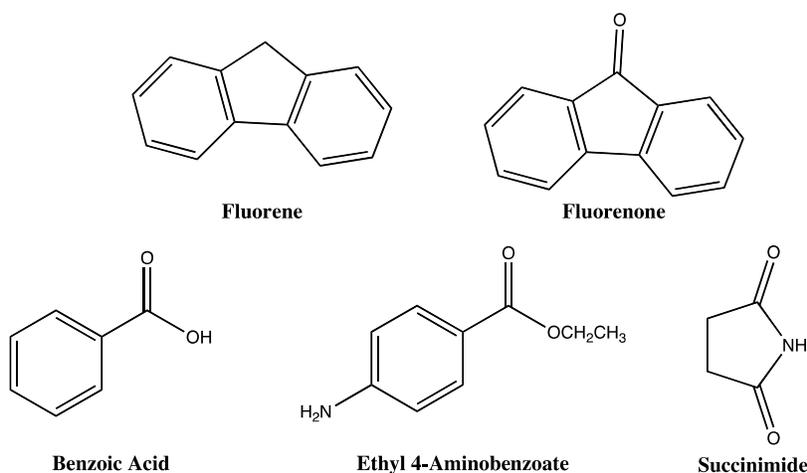
### S343: Principles of Extraction

**Question:** How does the identity of an impurity in a sample affect the choice of solvent to be used in extraction? When is it appropriate to use an acid extraction versus a base extraction?

#### Background:

Extraction can be an effective purification technique in many cases. Based on different solubilities of compounds, extraction is used to dissolve an impurity into a different liquid phase from the compound of interest. The success of this procedure depends on the two compounds having significantly different solubilities in the two phases. Toward this end, the chemist must choose the type of aqueous solution to use in the extraction. It is possible to use an acidic, basic, or neutral aqueous phase. Depending on the impurity, one of these may or may not be preferential.

In this experiment, you will be attempting to purify fluorene. Each of your team members will work with a different sample of fluorene contaminated with ethyl 4-aminobenzoate, benzoic acid, fluorenone, or succinimide. For each you will test an acid, base, and neutral extraction and determine which is most effective.



#### Procedure:

1. Each member of the team of four students should obtain one of the following impure fluorene samples: fluorene/ethyl 4-aminobenzoate; fluorene/benzoic acid; fluorene/fluorenone; or fluorene/succinimide. (Each sample is a 50/50 mix, except for fluorene/ethyl 4-aminobenzoate, which is 80/20.) Every team member should have a different impure sample.
2. Place about 0.5 g of your impure sample in 50 mL beaker and **record the mass**.
3. Add about 10-15 mL of ether to a separatory funnel and add the solid sample to the sep funnel. Swirl to dissolve the solid as much as possible.
4. Add 10 mL of distilled water to the separatory funnel. Shake the separatory funnel, **venting often**, until thoroughly mixed.
5. Drain the aqueous layer from the separatory funnel. At this stage err in allowing too little liquid out of the separatory funnel rather than too much.
6. Add another 10 mL of distilled water. Shake the separatory funnel, venting often, until thoroughly mixed.
7. Drain the aqueous layer from the separatory funnel. At this stage err in allowing too much liquid out of the separatory funnel rather than too little.
8. Pour the ether layer out the top of the separatory funnel into a clean, dry 50 mL Erlenmeyer flask. Rinse the separatory funnel out with ~5 mL of ether and transfer this to the Erlenmeyer as well.

9. Dry the ether with sodium sulfate for at least 5 minutes and decant into a clean, dry beaker. Rinse the Erlenmeyer flask out with ~5 mL of ether, let this sit for about a minute, and pour this into the beaker as well.
10. Remove the ether by blowing nitrogen over it.
11. Transfer the resulting solid to a labeled watch glass.
12. Repeat steps 2-11 two more times, using 1 M HCl and 1 M NaOH in place of distilled water.
13. After the purified fluorene is thoroughly dried, **obtain melting point data for each sample**.
14. Once you have completed the experiment, write your data on the board.

**Observations and Results** Include any observations and the following data in well-organized table(s). You should include all your team's data in one set of tables and the average class data in another set. Be sure to label the class data as such so that it does not infer that you collected all the data!

- Melting point ranges of each purified sample after:
  - HCl extraction
  - NaOH extraction
  - Water extraction

**Discussion Comments:**

Which, if any, extraction method effectively purified fluorene from ethyl 4-aminobenzoate? Provide specific data and explain any discrepancy between your group's data and that of the lab section. Provide a chemical explanation for why this extraction method was so effective or why none of the methods was effective. Repeat the same question for the other three impurities.

**Conclusion:** When choosing an extraction solvent for purifying a sample, one should...

**References**

**Lab 3 assignment: Turn in a hardcopy of your lab notebook and a formal Results and Discussion. Submit your Results and Discussion through Turnitin.com**

- Due at the beginning of lab next week
- 25 pts based on in-lab performance, notebook, and formal results and discussion
- The Results and Discussion section is the "meat" of the paper. Your goal is to convey your main scientific findings, supported by data, in such a way that it convinces your audience that you are correct in your conclusions. Include Figures and Tables as appropriate.
- See "Writing a Formal Report" for more information
- Make an electronic submission of your Results and Discussion section through Oncourse PRIOR to attending lab.