

Solubility

Reading *Mohrig* Essay- Intermolecular Forces in Organic Chemistry (pp. 127-131) and Section 10.2. See also, *Klein* Sections 1.12 (Intermolecular Forces) and 1.13 (Solubility).

General

Understanding the solubility characteristics of organic solids and liquids is vital to gaining experience in handling and purifying organic compounds in the chemistry laboratory. Also vital is learning the appropriate terms applied to the observation of mixing different materials: soluble, insoluble, partially, or soluble, and miscible vs. immiscible. This experiment explores the solubility behavior of a selection of different organic compounds, liquids and solids, each with a variety of functional groups.

Prelab

Include your **Name**, the **Date**, a **Title** for the experiment (i.e., Solubility), a **Purpose**, and a brief **Outline** of the essential procedure for each part (see below for the procedure). Prepare tables in your notebook to enter your observations for each part of the experiment. Read through the procedures below to see how to prepare each table - these must be in your notebook before you start the experiment.

Procedure

Work in pairs (no more than two students). Follow the procedures for Parts A through D below. Note your observations as you proceed with each part *directly into your notebook into the table you prepared in the prelab*.

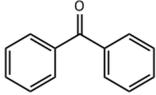
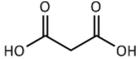
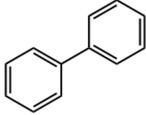
Part A -Solubility in Organic Solids

Place about 40 mg (0.040 g) of benzophenone into each of four dry test tubes. (Don't try to be exact: You can be 1–2 mg off and the experiment will still work.) Label the test tubes and then add 1 mL of water to the first tube, 1 mL of methyl alcohol (methanol) to the second tube, and 1 mL of hexane to the third tube. The fourth tube will serve as a control. Determine the solubility of each sample in the following way: Using the rounded end of a micro-spatula stir each sample continuously for 60 seconds by twirling the spatula rapidly. If the solid dissolves completely, note how long it takes for the solid to dissolve. After 60 seconds (do not stir longer), note whether the compound is

soluble (dissolves completely),
insoluble (none of it dissolves),
or partially soluble.

Compare each tube with the control sample in making these determinations. You should state that a sample is partially soluble only if a significant amount (at least 50%) of the solid has dissolved. If it is not clear that a significant amount of solid has dissolved, then state that the sample is insoluble. If all but a couple of granules have dissolved, state that the sample is soluble. Record these results in your notebook in the form of a table, as shown on this page. For those substances that dissolve completely, note how long it took for the solid to dissolve.

Now repeat the directions just given, using the solids malonic acid and biphenyl. Record these results in your notebook, using a table such as the one below to record your data – if the compound is soluble, be sure to also include the time (in seconds) it took to dissolve.

| Compound | water | methanol | hexane |
|---|-------|----------|--------|
| benzophenone  | | | |
| malonic acid  | | | |
| biphenyl  | | | |

Part B – Solubility of Liquids

For each solubility test (see list of compounds below), start by adding 1 mL of solvent (water or hexane) to a screw-cap vial. Then add one of the alcohols, dropwise. Carefully observe what happens as you add each drop. If the liquid solute is soluble in the solvent, you may see tiny horizontal lines in the solvent. These mixing lines indicate that solution is taking place. Cap and shake the vial after adding each drop. While you shake the vial, the liquid that was added may break up into small balls that disappear in a few seconds. This also indicates that solution is taking place. Continue adding the alcohol with shaking until you have added a total of 20 drops. If an alcohol is partially soluble, you will observe that at first the drops will dissolve, but eventually a second layer of liquid (undissolved alcohol) will form in the test tube. Record your results (soluble, insoluble, or partially soluble) in your notebook in table form.

methanol
1-butanol
1-octanol

Part C – Miscible or Immiscible Pairs

For each of the following pairs of compounds, add 1 mL of each liquid to the same screw-capped vial. Use a different vial for each pair. Cap the vial and shake for 10–20 seconds, then let the vial sit for 10–20 seconds to determine whether the two liquids are miscible (form one layer) or immiscible (form two layers). Record your results in your notebook.

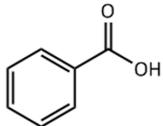
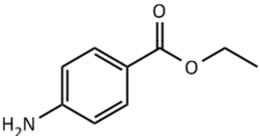
Water and ethyl alcohol
Water and diethyl ether
Water and methylene chloride
Water and hexane
Hexane and methylene chloride

Part D – Solubility of Organic Acids and Bases

Place about 30 mg (0.030 g) of benzoic acid into each of three dry test tubes. Label the test tubes and then add 1 mL of water to the first tube, 1 mL of 1.0 M NaOH to the second tube, and 1 mL of 1.0 M HCl to the third tube. Stir the mixture in each test tube vigorously with a micro-spatula for 10–20 seconds (be sure to clean off the micro-spatula thoroughly between each tube). Note whether the compound is soluble (dissolves

completely) or is insoluble (none of it dissolves). Record these results in the table. Now take the second tube containing benzoic acid and 1.0 M NaOH. With stirring add 6 M HCl dropwise until the mixture is acidic. Test the mixture with pH paper to determine when it is acidic. When it is acidic, stir the mixture for 10–20 seconds and note the result (soluble or insoluble) in the table.

Repeat this experiment using ethyl 4-aminobenzoate and the same three solvents. Record the results. Now take the third tube containing ethyl 4-aminobenzoate and 1.0 M HCl and with stirring, add 6 M NaOH dropwise until the mixture is basic. Test the mixture with pH paper to determine when it is basic. Stir the mixture for 10–20 seconds and note the result in a table like the one shown below.

| Compound | water | 1.0 M NaOH | 1.0 M HCl |
|--|-------|---------------|----------------|
| benzoic acid  | | | |
| | | add 6.0 M HCl | |
| ethyl 4-aminobenzoate  | | | |
| | | | add 6.0 M NaOH |

To Complete the Experiment – Template Report

Once you have completed all parts of the experiment, all the tables in your notebook should be completely filled in. Download the template from the class website and then fill in the appropriate fields, type your conclusion and summarize the results from each part using words. Answer the questions for each part, then print. You can draw the chemical equation asked for in Part D after you print the completed template. Attach your notebook pages to the completed template and turn in.