From: Intro to Org. Lab. Tech: A microscale approach, 4e
Pavia, Lampman, Kriz, Engel

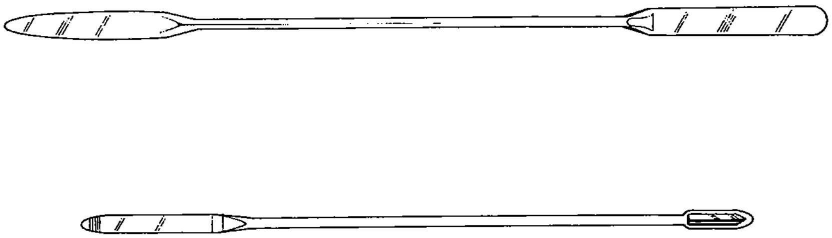

Figure 10
Microspatulas.

started in the laboratory. Additional techniques are described fully in Part Six of this textbook, and Experiments 2 through 17 expose you to the most important of them.

Some other practical hints need to be introduced at this point. The first of these involves manipulating small amounts of solid substances. The efficient transfer of solids requires a small spatula. We recommend that you have two **microspatulas,** similar to those shown in Figure 10, as part of your standard desk stock. The design of these spatulas permits the handling of milligram quantities of substances without undue spillage or waste. The larger style (see Fig. 10) is more useful when relatively large quantities of solid must be dispensed.

A clean work area is of utmost importance when working in the laboratory. The need for cleanliness is particularly great when working with the small amounts of materials characteristic of microscale laboratory experiments.

NOTE: You must read Technique 1 "Laboratory Safety." In preventing accidents, there is no substitute for preparation and care.

With this final word of caution and advice, we hope you enjoy the learning experience you are about to begin. Learning the care and precision that microscale experiments require may seem difficult at first, but before long you will be comfortable with the scale of the experiments. You will develop much better laboratory technique as a result of microscale practice, and this added skill will serve you well.

2

EXPERIMENT 2

Solubility

Solubility
Polarity
Acid-base chemistry
Critical thinking application

Having a good comprehension of solubility behavior is essential for understanding many procedures and techniques in the organic chemistry laboratory. For a thorough discussion of solubility, read the chapter on this concept

(Technique 10, p. 637) before proceeding because an understanding of this material is assumed in this experiment.

In Parts A and B of this experiment, you will investigate the solubility of various substances in different solvents. As you are performing these tests, it is helpful to pay attention to the polarities of the solutes and solvents and to even make predictions based on this (see "Guidelines for Predicting Polarity and Solubility," p. 639). The goal of Part C is similar to that of Parts A and B, except that you will be looking at miscible and immiscible pairs of liquids. In Part D, you will investigate the solubility of organic acids and bases. Section 10.2B on page 642 will help you understand and explain these results.

REQUIRED READING

New:	Technique 5	Measurement of Volume and Weight
	Technique 10	Solubility

SUGGESTED WASTE DISPOSAL

Dispose of all wastes containing methylene chloride into the container marked for halogenated waste. Place all other organic wastes into the nonhalogenated organic waste container.

NOTES TO THE INSTRUCTOR

In Part A of the procedure, it is important that students follow the instructions carefully. Otherwise, the results may be difficult to interpret. It is particularly important that consistent stirring be done for each solubility test. This can be done most easily by using the larger-style microspatula shown in Figure 10 on page 13.

We have found that some students have difficulty performing Critical Thinking Application 2 (p. 17) on the same day that they complete the rest of this experiment. Many students need time to assimilate the material in this experiment before they can complete this exercise successfully. One approach is to assign Critical Thinking Applications from several technique experiments (for example, Experiments 1–3) to a laboratory period after students complete the individual technique experiments. This provides an effective way of reviewing some of the basic techniques.

PROCEDURE

NOTE: It is very important that you follow these instructions carefully and that consistent stirring be done for each solubility test.

Part A. Solubility of Solid Compounds

Place about 40 mg (0.040 g) of benzophenone into each of four *dry* test tubes.[1] (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

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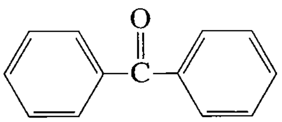
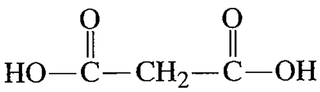
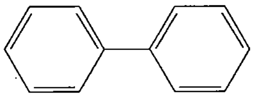
[1] *Note to the instructor:* Grind up the benzophenone flakes into a powder.

alcohol 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 microspatula (the larger style on p. 13), stir each sample continuously for 60 seconds by twirling the spatula rapidly. If a 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. You should compare each tube with the control 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. An additional hint for determining partial solubility is given in the next paragraph. 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.

Although the instructions just given should enable you to determine whether a substance is partially soluble, you may use the following procedure to confirm this. Using a Pasteur pipet, carefully remove most of the solvent from the test tube *while leaving the solid behind.* Transfer the liquid to another test tube and then evaporate the solvent by heating the tube in a hot water bath. Directing a stream of air or nitrogen gas into the tube will speed up the evaporation (see Technique 7, Section 7.10, p. 611). When the solvent has completely evaporated, examine the test tube for any remaining solid. If there is solid in the test tube, the compound is partially soluble. If there is no, or very little, solid remaining, you can assume that the compound is insoluble.

Now repeat the directions just given, substituting malonic acid and biphenyl for benzophenone. Record these results in your notebook.

Solvents

Organic Compounds	*Water (highly polar)*	*Methyl Alcohol (intermediate polarity)*	*Hexane (nonpolar)*
Benzophenone 			
Malonic acid  $HO-\overset{O}{\overset{\|}{C}}-CH_2-\overset{O}{\overset{\|}{C}}-OH$			
Biphenyl 			

Part B. Solubility of Different Alcohols

For each solubility test (see table on p. 16), add 1 mL of solvent (water or hexane) to a test tube. 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. *Shake the tube after adding each drop.* While you shake the tube, 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.

Solvents

Alcohols	*Water*	*Hexane*
1-Octanol $CH_3(CH_2)_6CH_2OH$		
1-Butanol $CH_3CH_2CH_2CH_2OH$		
Methyl alcohol CH_3OH		

Part C. Miscible or Immiscible Pairs

For each of the following pairs of compounds, add 1 mL of each liquid to the same test tube. Use a different test tube for each pair. Shake the test tube 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 with a microspatula for 10–20 seconds. Note whether the compound is soluble (dissolves completely) or is insoluble (none of it dissolves). Record these results in table form. Now take the 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 litmus or pH paper to determine when it is acidic.[2] 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 tube containing ethyl 4-aminobenzoate and 1.0 *M* HCl. With stirring, add 6 *M* NaOH dropwise until the mixture is basic. Test the mixture with litmus or pH paper to determine when it is basic. Stir the mixture for 10–20 seconds and note the result.

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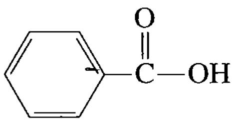
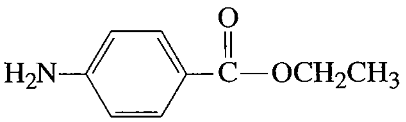
[2] Do not place the litmus or pH paper into the sample; the dye will dissolve. Instead, place a drop of solution from your spatula onto the test paper. With this method, several tests can be performed using a single strip of paper.

Solvents

Compounds	*Water*	*1.0 M NaOH*	*1.0 M HCl*
Benzoic acid 			
		Add 6.0 *M* HCl	
Ethyl 4-aminobenzoate 			
			Add 6.0 *M* NaOH

Part E. Critical Thinking Applications

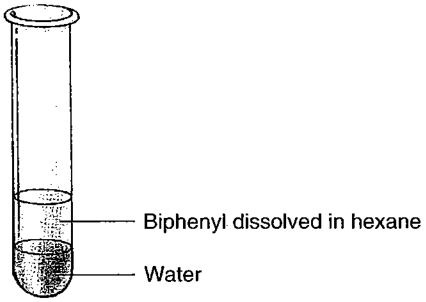
1. Determine by experiment whether each of the following pairs of liquids is miscible or immiscible.

   Acetone and water

   Acetone and hexane

   How can you explain these results, given that water and hexane are immiscible?

2. You will be given a test tube containing two immiscible liquids and a solid organic compound that is dissolved in one of the liquids.[3] You will be told the identity of the two liquids and the solid compound, but you will not know the relative positions of the two liquids or in which liquid the solid is dissolved. Consider the following example, in which the liquids are water and hexane and the solid compound is biphenyl.



   a. Without doing any experimental work, predict where each liquid is (top or bottom) and in which liquid the solid is dissolved. Justify your prediction. You may want to consult a handbook such as *The Merck Index* or the *CRC Handbook of Chemistry and Physics* to determine the molecular structure of a compound or to find any other relevant information. Note that dilute solutions such as 1 *M* HCl are composed mainly of water.

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[3] The sample you are given may contain one of the following combinations of solid and liquids (the solid is listed first): fluorene, methylene chloride, water; triphenylmethanol, diethyl ether, water; salicylic acid, methylene chloride, 1 *M* NaOH; ethyl 4-aminobenzoate, diethyl ether, 1 *M* HCl; naphthalene, hexane, water; benzoic acid, diethyl ether, 1 *M* NaOH; *p*-aminoacetophenone, methylene chloride, 1 *M* HCl.

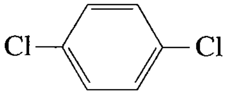
b. Now try to prove your prediction experimentally. That is, demonstrate which liquid the solid compound is dissolved in and the relative positions of the two liquids. You may use any experimental technique discussed in this experiment or any other technique that your instructor will let you try. In order to perform this part of the experiment, it may be helpful to separate the two layers in the test tube. This can be done easily and effectively with a Pasteur pipet. Squeeze the bulb on the Pasteur pipet and then place the tip of the pipet on the bottom of the test tube. Now withdraw only the bottom layer and transfer it to another test tube. Note that evaporating the water from an aqueous sample takes a very long time; therefore, this may not be a good way to show that an aqueous solution contains a dissolved compound. However, other solvents may be evaporated more easily (see p. 643). Explain what you did and whether or not the results of your experimental work were consistent with your prediction.

3. Add 0.025 g of tetraphenylcyclopentadienone to a dry test tube. Add 1 mL of methyl alcohol to the tube and shake for 60 seconds. Is the solid soluble, partially soluble, or insoluble? Explain your answer.

REPORT

Part A

1. Summarize your results in table form.
2. Explain the results for all the tests done. In explaining these results, you should consider the polarities of the compound and the solvent and the potential for hydrogen bonding. For example, consider a similar solubility test for *p*-dichlorobenzene in hexane. The test indicates that *p*-dichlorobenzene is soluble in hexane. This result can be explained by stating that hexane is nonpolar, whereas *p*-dichlorobenzene is slightly polar. Because the polarities of the solvent and solute are similar, the solid is soluble. (Remember that the presence of a halogen does not significantly increase the polarity of a compound.)



***p*-Dichlorobenzene**

3. There should be a difference in your results between the solubilities of biphenyl and benzophenone in methyl alcohol. Explain this difference.
4. There should be a difference in your results between the solubilities of benzophenone in methyl alcohol and benzophenone in hexane. Explain this difference.

Part B

1. Summarize your results in table form.
2. Explain the results for the tests done in water. In explaining these results, you should consider the polarities of the alcohols and water.
3. Explain, in terms of polarities, the results for the tests done in hexane.

Part C

1. Summarize your results in table form.
2. Explain the results in terms of polarities and/or hydrogen bonding.

Part D

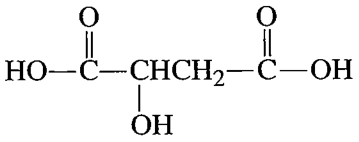
1. Summarize your results in table form.
2. Explain the results for the tube in which 1.0 *M* NaOH was added to benzoic acid. Write an equation for this. Now describe what happened when 6.0 *M* HCl was added to this same tube and explain this result.

3. Explain the results for the tube in which 1.0 *M* HCl was added to ethyl 4-aminobenzoate. Write an equation for this. Now describe what happened when 6.0 *M* NaOH was added to this same tube and explain.

Part E Give the results for any Critical Thinking Applications completed and answer all questions given in the Procedure for these exercises.

QUESTIONS

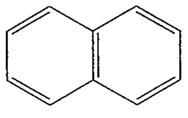
1. For each of the following pairs of solutes and solvent, predict whether the solute would be soluble or insoluble. After making your predictions, you can check your answers by looking up the compounds in *The Merck Index* or the *CRC Handbook of Chemistry and Physics*. Generally, *The Merck Index* is the easier reference book to use. If the substance has a solubility greater than 40 mg/mL, you may conclude that it is soluble.
   a. Malic acid in water



$$HO-\overset{\overset{O}{\|}}{C}-\underset{\underset{OH}{|}}{C}HCH_2-\overset{\overset{O}{\|}}{C}-OH$$

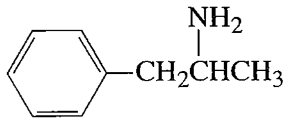
Malic acid

   b. Naphthalene in water



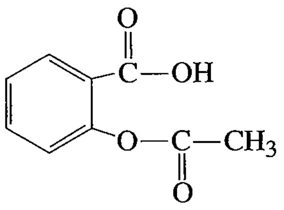
Naphthalene

   c. Amphetamine in ethyl alcohol



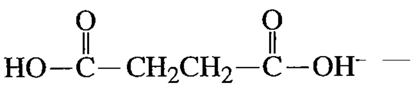
Amphetamine

   d. Aspirin in water



Aspirin

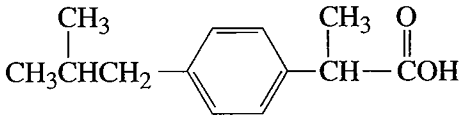
   e. Succinic acid in hexane (*Note:* The polarity of hexane is similar to petroleum ether.)



$$HO-\overset{\overset{O}{\|}}{C}-CH_2CH_2-\overset{\overset{O}{\|}}{C}-OH^-$$

Succinic acid

f. Ibuprofen in diethyl ether



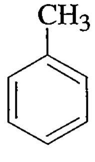
Ibuprofen

g. 1-Decanol (*n*-decyl alcohol) in water

$CH_3(CH_2)_8CH_2OH$

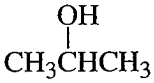
1-Decanol

2. Predict whether the following pairs of liquids would be miscible or immiscible:
   - **a.** Water and methyl alcohol
   - **b.** Hexane and benzene
   - **c.** Methylene chloride and benzene
   - **d.** Water and toluene



Toluene

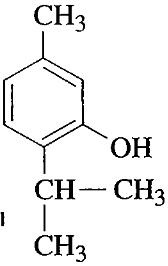
e. Ethyl alcohol and isopropyl alcohol



$CH_3CH(OH)CH_3$

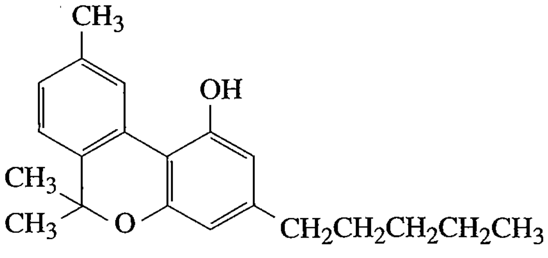
Isopropyl alcohol

3. Would you expect ibuprofen (see 1f) to be soluble or insoluble in 1.0 *M* NaOH? Explain.
4. Thymol is very slightly soluble in water and very soluble in 1.0 *M* NaOH. Explain.



Thymol

5. Although cannabinol and methyl alcohol are both alcohols, cannabinol is very slightly soluble in methyl alcohol at room temperature. Explain.



Cannabinol