Experiment 21 - Solubility and Molecular Structure

Part 1

In this part of the experiment you will compare the solubilities of various substances in two very different solvents: water and cyclohexane. Recall that water molecules are polar and can hydrogen bond. In contrast, molecules of cyclohexane are nonpolar and cannot form hydrogen bonds. Cyclohexane is a typical nonpolar solvent. The substances that you will try to dissolve will be of three bonding types: ionic, such as NaCl; nonpolar covalent, such as paraffin wax; and polar covalent, such as ethanol. Results will allow you to put all the substances in one of the following four groups: soluble in water (but not in cyclohexane); soluble in cyclohexane (but not in water); soluble in both; or soluble in neither. Recall also the concept "like dissolves like" in trying to understand the reasons behind the different solubilities of compounds.

Safety Precautions:

- Wear your safety goggles.
- When using cyclohexane, use it under the fume hood and avoid breathing the fumes.

Waste Disposal:

- All solutions containing cyclohexane and other organic compounds should be disposed of in the **organic waste** bottle (with a red label) in the hood.
- Waste that does not contain organic compounds should be placed in the **inorganic waste** bottle (which has a blue label) in one of the fume hoods.

Best way to shake a test tube and its contents:

Hold the tube firmly at the top with one hand, and tap the bottom of the tube with one or more fingers of your other hand. In this way you shake the contents thoroughly yet do no shake any out of the tube. No stopper is needed. (Stoppers are a nuisance because they frequently start to dissolve in the liquid and cause problems.) This method works quite well as long as the test tube has less than 5 to 10 mL of liquid in it.

Procedure - Part 1

1. Note: To avoid complications, it is best to use dry test tubes for testing with cyclohexane solvent. It is best to dedicate a group of test tubes to "cyclohexane-only" use, and a second set to "water-only" use. By using your tubes and those of your partner you may be able to collect enough tubes to do this. Label the test tubes accordingly and place them in your test tube rack. You will be testing the following substances: ethanol, naphthalene, NaCl, KI, $BaSO_4$, sucrose (sugar), vegetable oil, water, baking soda ($NaHCO_3$), paraffin wax, heptane, acetic acid, and butter.
2. Put 2 mL of water in one test tube and 2 mL of cyclohexane in a different test tube. If the substance you are to test is a solid, put 1 spatula tip-full (this is a very small amount) into each tube. If the substance is a liquid, put 10 drops of it in

each tube. Swirl the test tube vigorously to give the substance every chance to dissolve. Note that the most common mistake students make is not to mix the tube vigorously enough or long enough. If the sample dissolves (seems to disappear), consider it "soluble" in that particular solvent, and go on to other tests. But if some or all of the sample remains visible, hence undissolved, list the sample as "insoluble" in that solvent.

A solid that does not dissolve is easily seen lying on the bottom of the test tube. A liquid that does not dissolve is not so easily seen. It may be hard to see the second layer that it forms on the surface (if less dense than the solvent) or at the bottom (if more dense). Try watching for little bubbles that form on shaking, then disappear as they come together to form a layer when the shaking is stopped. You may want to dye the water layer with some dark blue $CuSO_{4\,(aq)}$, which will not color the nonpolar layer.

Cloudiness is a sign that a substance has not dissolved but is floating as tiny particles. Although in principle cloudiness is a sign of insolubility, in practice it may be due to traces of an impurity.

3. Test each substance listed on the report sheet to see whether they are soluble in water, in cyclohexane, or in neither. Record your results on the report sheet as you go.

Part 2

In Part 1 you found that one kind of alcohol, ethanol, is extremely soluble in water, thanks to its OH group. All alcohols contain OH groups; are they all equally soluble in water? You will test five other alcohols to find out. Notice that they vary in the number of C and H atoms, while all having just one OH group.

Procedure - Part 2

1. To 2 mL of deionized water in a clean test tube add methanol (methyl alcohol) dropwise, counting drops and shaking the test tube, to see how much will dissolve. If you can dissolve as much as 40 drops, just stop and call it "infinitely soluble."
2. Repeat, using fresh portions of water, for these alcohols: 1-propanol, C_3H_7OH; 1-pentanol, $C_5H_{11}OH$; 1-butanol, C_4H_9OH; and 1-hexanol, $C_6H_{13}OH$.
3. What do you conclude? Try to formulate a conclusion relating solubility and the "chain length" of these alcohols.

Part 3

You probably know that certain vitamins are water-soluble, while others are fat-soluble. (This relates to what happens to them in our bodies, and how long they may be stored, where they are stored, and so on.) You will test the solubilities of a few vitamins in water (which has about the same solvent properties as blood, so whatever dissolves in water should also dissolve in blood) and in vegetable oil (to represent the solvent power of body fat).

Procedure – Part 3

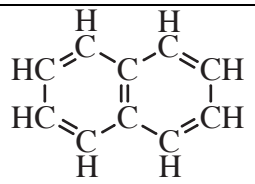
1. Put a **very small** amount of the first vitamin into two separate test tubes. Use the same amount of the vitamin for each tube. It is best if the test tubes are dry, and if

you put the sample directly in the bottom of the tube so that it doesn't stick to the side. Use the tip of the spatula provided to sample the solid vitamins. Use the end of the glass stirring rod provided to sample viscous liquids (such as vitamin E).

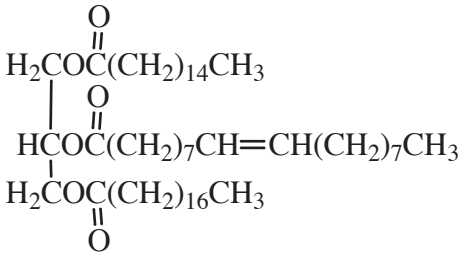
2. Add about 1 to 1.5 mL (20 to 30 drops) of water to the vitamin in one of the test tubes, and shake well to mix. Add the same amount (20 to 30 drops) of oil to the other test tube. Shake each tube well. Notice that we are not going to measure how much solvent is needed, but just to add the same amount of solvent to each. Decide whether the vitamin is more soluble in water ("water-soluble") or in oil ("fat-soluble").
   How can you tell if the vitamin has dissolved? If the vitamin is a liquid, look for cloudiness or a second layer as a sign that it has **not** dissolved. (When liquids dissolve in other liquids, you get just one clear solution, no second layer). On the other hand, if the vitamin is a solid, it is much easier to see the undissolved solid.
3. Repeat steps 1 and 2 with a different vitamin. Record all of your results on the report sheet. Test all of the available vitamins to determine whether each one is water-soluble or fat-soluble.
4. Look at the structural formulas of these four vitamins (below) and notice that the structures of the two water-soluble and the two fat-soluble vitamins are different. <u>How</u> do they differ?

Structures of Some Organic Compounds Used in this lab:

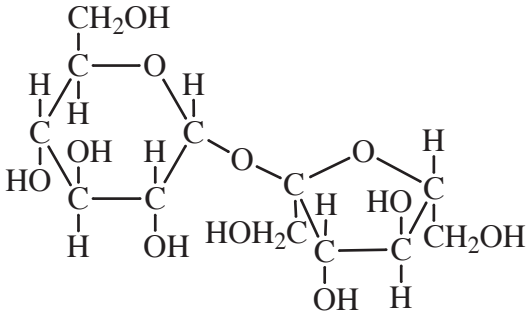
Methanol CH_3OH	**Ethanol** CH_3CH_2OH
1-Propanol $CH_3CH_2CH_2OH$	**1-Butanol** $CH_3CH_2CH_2CH_2OH$
1-Pentanol $CH_3CH_2CH_2CH_2CH_2OH$	**1-Hexanol** $CH_3CH_2CH_2CH_2CH_2CH_2OH$
Acetic acid (in vinegar) $CH_3\overset{\overset{\displaystyle O}{\|}}{C}OH$	**Naphthalene** (was used in mothballs)



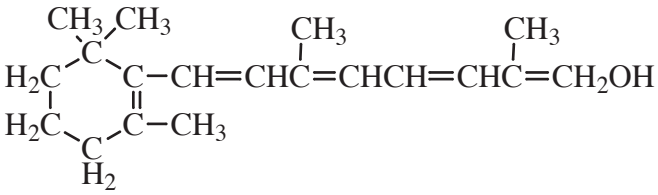
A **triglyceride** (in fats such as vegetable oil)



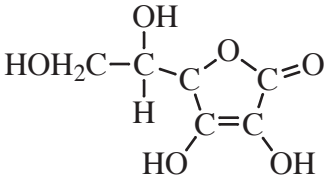
Sucrose (table sugar)



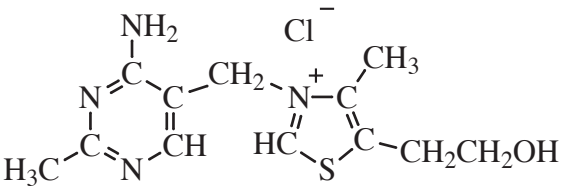
Retinol (Vitamin A)



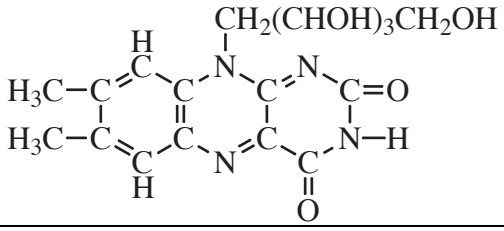
Ascorbic acid (Vitamin C)



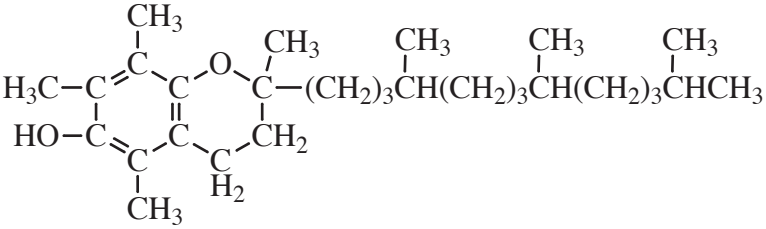
Thiamine (Vitamin B_1)



Riboflavin (Vitamin B_2)



α-Tocopherol (Vitamin E)



Questions

1. Look at your data for part 2 of this experiment. How does the solubility of an alcohol in water relate to the "chain length" of that alcohol?
2. Imagine you did an experiment in which you tested the solubility of various alcohols in cyclohexane (instead of in water). Which alcohol (of the ones you tested in part 2) would you expect to be the most soluble in cyclohexane? Which would be least soluble in cyclohexane? Explain your reasoning clearly.
3. How do the structures of the "fat-soluble" and the "water-soluble" vitamins differ?