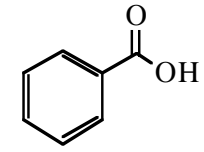
Melting Points

Introduction

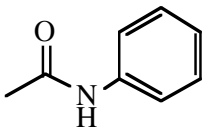
One of the most important identifying characteristics of a substance (especially before modern spectral techniques were advanced in the computer age) is its melting point. As organic chemistry is a science that deals with making and isolating compounds, it is important to be able to assess the relative purity of a molecule that you have just made or isolated in the lab. The melting point of a solid can be used for this purpose. In general, a pure crystalline solid will have a very sharp and narrow melting point range while an impure compound will have a more broad range. In addition, determining the melting point of an unknown compound is often the first step a chemist takes when attempting to identify it.

There are thousands of solid organic molecules in the world and most of these have melting points below 300 ˚C. So it is inevitable that several of them will have similar melting points. One common technique to distinguish between these compounds is to do a mixed melting point experiment. An unknown solid is mixed with a known compound with a similar melting point. If the two mixed compounds are identical, then the sample is a pure solid and it will have a narrow melting point. If the compounds are different, then the mixed sample is an impure solid and its melting point range will be lower and larger than **BOTH** of the compounds in the mixture. Therefore, these techniques are of great importance in an organic chemistry laboratory. These experiments are designed to familiarize you with these techniques.

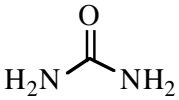
Structures (line drawings and condensed)



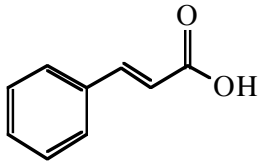
Benzoic Acid



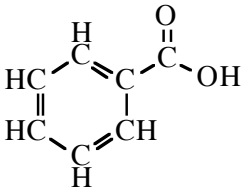
Acetanilide

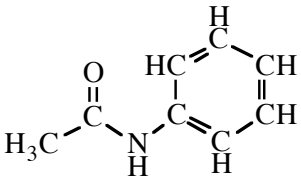


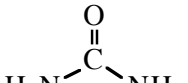
Urea

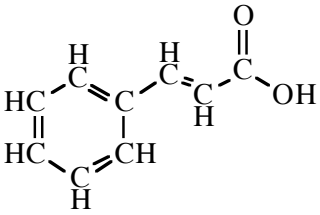


Cinnamic Acid









Experimental Procedure:

Melting points

You will be provided with pure samples of acetanilide, benzoic acid, cinnamic acid, and urea. All of these compounds have melting points between 105 - 150 ˚C. In addition, you will also be given three pre-made mixtures of urea/cinnamic acid (4:1, 1:1, and 1:4). Using either the Thomas-Hoover apparatus or the Mel-Temp apparatus you should determine the melting points of these 7 samples. Don't worry, you can do two or three at one time.

To do this, you will use the capillary tube that is closed at one end. Place a small amount of the solid on a watch glass and push the open end of the capillary tube into the solid several times until about 3-5 mm of solid is trapped in the open end of the tube. Then invert the tube and tap it on the bench top until the solid falls to the bottom, closed end of the tube (Note: to do a mixed melting

point, simply mix the two solids on the watch glass before tapping the capillary tube into the mixture). Then place this tube in the mel-temp apparatus and begin raising the temperature. Make sure that the mel-temp is at least 15 ˚ below the melting point of your solid. You may raise the temperature fairly quickly until you are about 15-20 ˚ below the melting point of your compound. Then lower the heating so that the temperature only raises one degree per minute. Then record the temperature at which the solid melts. Remember, you should always record the melting point as a **RANGE** of temperatures! Do not just list one temperature as the melting point. The beginning of the melting point range should be when you see the first drop of liquid in your capillary tube. Many compounds will appear to "sweat" (you'll know it when you see it) before they melt. Do not take the first appearance of this "sweat" as the start of your melting point range. Wait for the first drop! The end of the melting point range should be when all of the solid has become a liquid.

After determining the melting point ranges of these 7 samples, obtain an unknown solid from your lab instructor and record its melting point range. You should identify the unknown solid. It will be one of the 4 compounds listed above. The compounds listed above should have some similar melting points. So how will you be able to **prove** which of them is your unknown?