**Using the Melting Point to Identify Unknown Organic Compounds**

The melting point of a pure substance is the temperature at which the solid and liquid phases are in equilibrium under one atmosphere of pressure. There are literally millions of organic compounds and most of them have a melting point less than 300°C. Obviously, two or more compounds may have the same melting point. However**, if two compounds have different melting points, then they are different compounds.** Sometimes melting points may differ by only a fraction of a degree which means that obtaining a melting point must be done carefully. A mp can be used to help identify an unknown substance simply by finding a literature melting point value that matches your sample. Verification is done with a **mixed melting point** obtained by mixing your sample with a known compound and observing the mp of that mixture. If the mp range is broad and lower, the two compounds are not the same since one compound behaves as an impurity within the other.

**Using the Melting Point to Determine Purity of an Organic Compound**

The melting point of a pure sample is characterized by a **sharp melting point**. A sharp mp means the phase transition occurs over a small temperature range- typically a 1-2°C range is considered a sharp mp. A **broad melting point range** means that the melting occurs over a larger temperature range- greater than a 3°C mp range, for example. The presence of impurities affect the behavior of the sample undergoing a phase transition. **Impurities lower the mp and broaden the range.** Thus, even an unknown sample can be deemed pure or impure based on its mp range, i.e. sharp or broad mp range. A melting point is only one piece of data, but often it is a good clue regarding the purity of any sample.

The **goal** in this experiment is to learn how to obtain accurate melting points of organic compounds. The **objective** is to identify three unknown compounds by their melting point. Students will be working in small groups for this experiment and each group will be assigned 3 unknown compounds. Ideally, each student in the group will be working with at least one unknown compound and sharing the results with other members of the group. **Notebook organization:** Before starting the procedure, construct a simple data table in your notebook to record the data that you expect to obtain in this experiment. Record the number for each unknown and the name of the compound once you have identified it.

## Procedure

Be sure to record each melting point determination as a melting point range.
Each student must obtain a melting point for at least one unknown; multiple measurements should be consistent. Data should be shared with your group members.

1. Accurately determine the melting point range for your unknown compound. Multiple trials should be consistent. Data should be shared with your group members. Once you are confident that you have an accurate mp for your unknown, consult with the instructor and compare your melting point data with a list of possible known compounds.

2. Proceed to verify your initial assignment by performing mixed melting points of each unknown with the known compounds that you suspect are a match for your compound. For each unknown, obtain a mixed-melting point with at least one known compound. You may have to measure the mixed-melting point of your unknown with two or more known compounds until you make your identification. Repeat this step for each unknown until you are confident that you have found a match with a known compound. Record all determinations in your notebook.

**MiniReport**

Turn in lab notebook pages that contain your data table and other observations.
Review the Procedure so that your data entries are recorded properly:

- Identify each substance as unknown (number) or by its chemical name
- Record mp as a range. When multiple mp values are obtained for each compound, label each result as trial 1, trial 2, etc.
- Identify each mp trial as a single substance or a mixed melting point trial
- Do not average mp values when multiple trials are obtained.

**Exercises- Turn in this page along with your answers to the questions below.**

NAME _____ Section _____

1. Define melting point.

2. What is meant by a sharp melting point?

3. What is the effect of an impurity on the melting point of a compound?

4. A student prepares a melting point tube using a stir rod to grind a pure compound on a filter paper. The measured melting point range is broad and approximately 7°C below the literature value. Explain.

5. Define a eutectic mixture.

6. A student measures the melting point of an unknown organic solid and observes a melting point range of 122.5-123°C. Does this result guarantee that the unknown substance is a pure compound? Explain.

7. A student finds a white powder on the lab bench next to 4 bottles of organic compounds. The labels contain the following compounds and melting point information:

Salicylic acid mp 159°C
Sorbic acid mp 132-135°C
Benzamide mp 130-132°C
Benzoic acid mp 121°C

A mixture of the white powder with benzamide melts at 117-128°C; a mixture of the white powder with sorbic acid melts at 129-131°C and a mixture with salicylic acid melts at 128-136°C. Identify the white powder and explain your reasoning.

**Additional Resources:**

1. Read Operation 33 in your lab text for a complete background on melting point measurements

2. Visit this webpage to see a good example of melting point determination from The College of Southern Idaho: http://www.csi.edu/ip/physci/faculty/rex/MPTips.htm

## MELTING POINT TIPS AND GUIDELINES

(Exceptions are rare.)

### General Guidelines

A pure organic compound usually melts over a range of two degrees or less.

If the melting point of a pure compound is within a degree of the value found a lab handbook it is presumed to be pure.

A sample is impure if it has a melting point range that is lower and/or wider than that the literature value. More impurities increase this effect.

In the organic lab, unless something is wrong with the procedure or the equipment, a substance generally cannot be observed to melt at a higher temperature than its melting point!

Experimental melting points should be always reported as a range, for example, 42-44°.

A given sample is only used once for mp determination. Always use fresh samples for additional trials.

Dispose of used capillary tubes in the proper receptacle, not in the wastebasket.

### Melting Point Tips

Most errors come from heating the sample too fast. A heating rate of 1 to 2 degrees per minute is will give good results. Going faster than five degrees per minute virtually guarantees poor results in most cases.

TIME-SAVING TIP: If a compound has a high mp, it can take a long time to reach it at 1 or 2 degrees per minute! Unless you have prior knowledge of the expected melting point, it is advisable to have extra samples prepared ahead of time. Run the first sample at a high rate of heating to get an approximate mp range. Then repeat the procedure but slow down the rate of heating as you approach the expected melting point.

Too much or too little sample can lead to errors. Generally 3-4 mm in the bottom of a capillary tube works well.

The sample should be firmly packed in the bottom of the tube to insure efficient heat transfer.

**SAMPLE SIZE**

See the Picture (Right)

The smaller sample is probably too small to properly observe the mp range. The larger sample may be too large, resulting in a mp range that is too wide and possibly high. The middle sample is about right but it is near the maximum size that should be used. Note that it is firmly packed.

**More Melting Point Tips**

·If you have trouble packing a tube, consider discarding it (proper receptacle) and starting over. Difficult cases are often not worth the struggle and mp capillaries are inexpensive.

·When it doubt about a result, check your technique by running a sample of a pure known substance with a similar melting point.

·Samples that melt below 50° require extra care in order to get good results. Slow heating is critical. Also, small amounts of impurities seem to have a greater effect on these low melting substances.

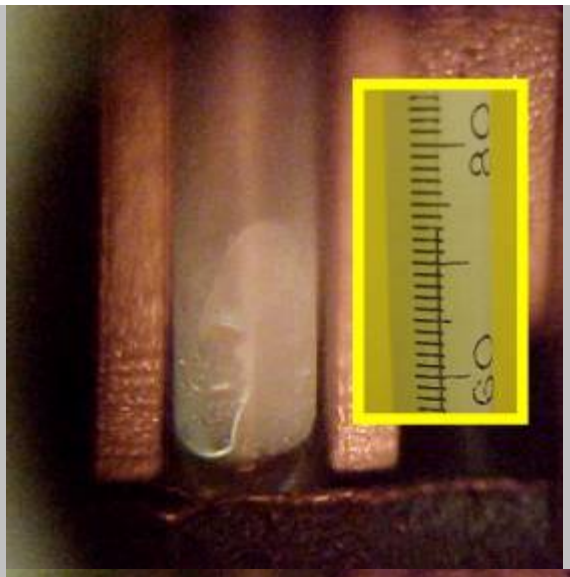
·**Mixed Melting Points –** The identity of two samples that have the same melting point can be determined by taking a mixed melting point. Grind roughly equal portions of the two samples together to mix well and take a melting point of the mixture. If the two substances are identical the melting point should be the same as that of either sample. If the two substances are not identical, then the melting point will be depressed.

WHAT YOU MAY SEE

THIS IS A SAMPLE OF IBUPROFEN ISOLATED FROM COMMERCIAL TABLETS.

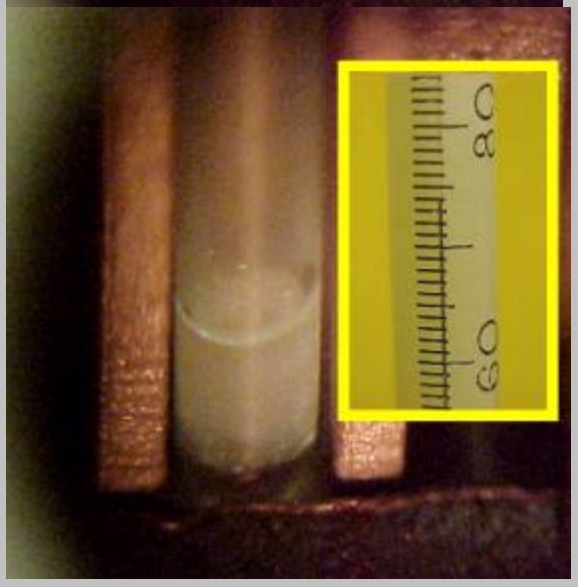
TINY DROPLETS APPEAR AT THE START OF THE MELTING POINT RANGE. OFTEN THE SAMPLE APPEARS TO “SHRINK” AWAY FROM THE SIDES OF THE TUBE. HERE, AS SHOWN IN THE INSET (YELLOW BOX), THE SAMPLE HAS STARTED TO MELT JUST ABOVE 72°.

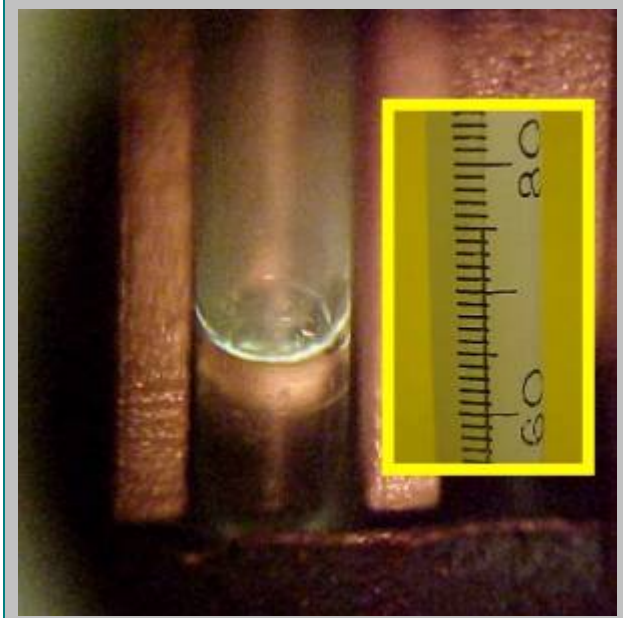
AS THE TEMPERATURE RISES TO 73° THE LIQUID PHASE BECOMES CLEARLY VISIBLE.



(RIGHT) AT 74° THERE IS A SLUSHY MIXTURE OF CRYSTALS AND LIQUID.

(BELOW) FINALLY AT A TEMPERATURE OF 75°, ALL OF THE SOLID HAS MELTED AND A CLEAR LIQUID REMAINS.THE MELTING POINT IS REPORTED AS 72-75°.





THE LITERATURE VALUE (*The Merck Index, 12th Ed.)* FOR IBUPROFEN IS 75-77°.

THE EXPERIMENTAL RESULT SUPPORTS THE PRESENCE OF IBUPROFEN. BUT THE MP RANGE IS WIDER AND LOWER THAN EXPECTED, PROBABLY DUE TO SMALL AMOUNTS OF IMPURITIES.