Recrystallization and Melting Point Determination

Introduction

RECRYSTALLIZATION is a standard and efficient process for PURIFICATION of solid substances. In this process, the IMPURE solid is first dissolved in HOT water. INSOLUBLE CONTAMINANTS can then be removed by GRAVITY FILTRATION of the hot solution through ordinary filter paper. The solution that passes through the filter, called the FILTRATE, is then cooled to room temperature (or below) to permit CRYSTALLIZATION of the desired material. Water is then removed from the crystals of the desired material by SUCTION FILTRATION. The damp crystals are then dried in the air over several days to remove residual water.

The MELTING POINT is a CHARACTERISTIC property of a pure crystalline substance and may be used as a point of IDENTIFICATION for the substance. PURE substances usually melt SHARPLY (or over a very narrow range of temperatures).

The presence of a dissolved IMPURITY almost always has TWO EFFECTS on the melting point: The MIXTURE of chemicals (major substance plus impurity) tends to melt at a LOWER TEMPERATURE INITIALLY, and over a much BROADER RANGE OF TEMPERATURES overall.

In this experiment we determine accurately the melting point of a recrystallized UNKNOWN chemical, and then identify the unknown by COMPARING its melting point with the melting points of KNOWN chemicals. A MIXTURE of the UNKNOWN chemical is then made with the KNOWN chemical it is believed to be, and the melting point of the mixture is determined. If the known and unknown chemicals are IDENTICAL, the mixture should melt at the SAME TEMPERATURE. If the known and unknown are different, the melting point of the mixture should show a large depression (10°C or greater) and will melt over a broad temperature range.

Summary

An unknown substance is purified by recrystallization from water and is allowed to dry. Its melting point is then determined, and its identity confirmed by mixed melting point.

Supplies

Capillary tubes; rubber bands

CHEMICALS

Acetanilide, toluic acid, benzoic acid, p-toluene sulfonamide and salicylic acid are all organic flammable solids.

Acetanilide—irritant
Benzoic acid—irritant, toxic by ingestion
p-Toluene sulfonamide—irritant
o-Toluic acid—irritant, toxic by ingestion
Salicylic acid—toxic by ingestion

CAUTION! WEAR SAFETY GLASSES AT ALL TIMES!!

Procedure

(1) Recrystallization

Obtain a melting point unknown from the lab and **RECORD THE UNKNOWN NUMBER** in both your lab notebook and on the Experiment 2 report sheet.

Inspect the sample apparatus for hot GRAVITY filtration and for SUCTION filtration.

Weigh an empty piece of filter paper or weighing paper. Transfer your unknown to the filter or weighing paper and reweigh.

Transfer your unknown to a 250 mL Erlenmeyer flask and add about 100 mL of water.

Return your empty plastic container to the tray you took it from before you leave the lab.

Heat the water to boiling and stir the mixture occasionally with a glass rod during the solution process. (The insoluble impurity, carbon, will not dissolve.)

Filter the hot solution of the unknown through a filter paper in the gravity funnel into a 250 mL beaker. CAUTION: YOU WILL NOT BE ABLE TO HANDLE THE FLASK WITH YOUR BARE HANDS. YOU WILL NEED TO USE YOUR CRUCIBLE TONGS OR SEVERAL PIECES OF PAPER TOWEL.

Dispose of your filter paper into the beaker labeled used filter paper.

Heat the solution in your 250 mL beaker until the volume is reduced to between 40 and 50 mL. Cool the filtrate in the beaker by immersion in an ice/water bath.

When crystallization is complete (the filtrate reaches 0°C), filter the crystals by suction, using a water aspirator, suction flask, Buchner funnel, and adapter. Remove as much of the liquid as possible by suction (press down on the Buchner funnel with the palm of your hand to ensure a tight seal). Make sure you take the proper size filter paper to fit your Buchner funnel. It should cover all the holes and be flat on the bottom. If it goes up the side of the funnel, the filter paper is too big for your funnel.

Weigh a clean empty watch glass and record its mass in your lab notebook.

Transfer the moist crystals to a clean watch glass and allow them to dry until the next lab period. Before you leave the lab, place the watch glass flat on top of your 600 mL beaker in your locker.

Dispose of your filter paper in the Buchner funnel in the beaker labeled used filter paper.

Dispose of the liquid in your filter flask down the drain.

(2) Melting Point Determination

Weigh your watch glass with the crystals.

There are two methods for determining the melting point of your unknowns. The first is using an electronic device called a Digital Melting Point Apparatus. The second is to use a mineral oil bath to melt your crystals. Directions for using both are listed below.

DIRECTIONS FOR USING DIGITAL MELTING POINT APPARATUS

1. Your lab instructor will show you how to fill the melting point capillary. The column of crystals in the bottom of the capillary tube should only be about l cm high. Place the capillary tube in the back of the eyepiece.
2 Turn on the apparatus. It is the black switch on the back left of the transformer. Do not change the settings on the red switch.
3. The device will beep and should read between 20°C and 25°C.
4. Hit the up arrow ? symbol to set the desired temperature. (The machine moves temperature upward in 10°C increments.) Push button for every 10°C increase desired. The symbol is located on the bottom of the control area (next to the down symbol and the clear symbol).
5. When the desired set temperature is reached push the ? symbol. This starts the instrument heating.
6. When it reaches the set temperature, the instrument will beep three times. If your crystals have not melted, increase the set temperature.
8. To increase the set temperature, push the clear symbol and the push the up arrow symbol until the new set temperature is reached and shows in the window.
9. Push the ? symbol to start the unit heating again to attain the new set temperature.
10. The recommended procedure is to set the device initially to 100°C. If your crystals have not melted by this temperature, increase the temperature by 10°C and reheat. Use 10°C increases until the crystals melt.
11. After finishing your melting point, it will be necessary to cool down the instrument to below 100°C before the next person can melt their crystals.
12. To cool down the instrument, push the clear symbol and then the down ? symbol until the desired temperature is reached (probably you should set it to 90°C for the next person). Note the apparatus changes temperature settings downward in 1°C increments.

MIXED MELTING POINT DETERMINATION

From the stockroom obtain a small sample of the pure compound you believe your unknown to be. Mix together THOROUGHLY roughly equal amounts of your recrystallized unknown and the known compound in the plastic container that you obtained from the stockroom that contains the known compound.

Determine the melting range of the mixture carefully. If there is no significant melting point depression, the identity of your unknown is confirmed. If you observe a large depression in the mixed melting point, try a different substance and repeat this procedure. Continue trying different substances until you find a substance that does not depress the melting point.

Dispose of your crystals in the WASTE EXPERIMENT 2 CRYSTALS beaker.
Dispose of your plastic boats into the plastic boats beaker.
Dispose of your capillary tubes into the capillary tubes beaker.

REMEMBER TO TURN IN THE YELLOW COPY OF YOUR LAB NOTEBOOK TO YOUR TA.

POSSIBLE UNKNOWNS AND MELTING RANGES

o-Toluic acid	100-106°C
Acetanilide	110-117°C
Benzoic acid	120-125°C
P-Toluene sulfonamide	133-140°C
Salicylic acid	150-155°C

Recrystallization and Melting Point Determination Report Sheet (30 Points)

Name _____ Date_____

Lab Instructor _____ Lab Day _____

Report

(1) Unknown number _____

Mass of filter or weighing paper _____ g

Mass of unknown and paper _____ g

Mass of unknown _____ g

Mass of empty watch glass _____ g

Mass of watch glass and crystals _____ g

Mass of crystals recovered _____ g

Percent recovery _____ g

Approximate melting point _____°C

Accurate melting point (range) _____°C

(2) Mixed melting point of unknown with _____ melted at _____ °C

Mixed melting point of unknown with _____ melted at _____°C

(3) The recrystallized unknown substance is _____

Recrystallization and Melting Point Determination
Post-Lab Questions (40 points)

Name _____ Date_____

Lab Instructor _____ Lab Day _____

1. Describe the process of recrystallization (not a definition).

2. Why does one boil the charcoal–unknown–water mixture as opposed to using only cold water?

3. Why is it necessary to wait until the following week before determining the melting point of your unknown?

4. What is a mixed melting point? How is it used to confirm the identity of an unknown substance?

5. Describe the process of suction filtration and explain why you might use this technique.

6. Calculate the percent recovery of your purified crystals.

Recrystallization and Melting Point Determination Preliminary Questions (10 Points)

Name _____ Date_____

Lab Instructor _____ Lab Day _____

(Show calculations on the back of the page.)

1. Define solubility

2. A substance has a solubility of 12.5 g per 100 mL of solvent at 100°C and 2.45 g per 100 mL solvent at 10°C. How many grams of the substance can dissolve in 25 mL of solvent at 100°C? How many grams of the substance can dissolve in 25 mL of solvent at 10°C?

_____ grams can dissolve in 25 mL at l00°C

_____ grams can dissolve in 25 mL at l0°C

3. What hazards are associated with the unknowns?

4. What is the proper disposal of all filter paper used in this experiment?

5. A student began with a 3.12 g sample that contained their unknown and charcoal. After recrystallization the mass of dried crystals recovered was 2.61 g. What was the percent recovery?