Purification Of A Solid By Recrystallization
AND
Identification By Melting Point Determination

Refer back to your recrystallization and melting point experiments. In this experiment you must purify your solid substance from a mixture of solids by using their difference in solubilities in ethanol. Your material will be soluble in hot ethanol and insoluble in cold ethanol.

PROCEDURE

Place 10 mL 95% Ethanol into a 100 mL beaker, and place it atop your hot plate, which in turn is placed atop the base of a ringstand. Set the hot plate to “3”.

Seat a fluted filter paper in a stemless glass funnel, supported on an iron ring, over the 50 mL beaker - the funnel just barely above the top of the beaker.

In addition, place a 100 mL beaker containing 50 mL of 95% Ethanol on the hotplate.

Weigh about 1 - 2 gm. of your unknown in a 100 mL beaker, using the top-loading balances provided in the laboratory.

Add small amounts of boiling 95% Ethanol (no more than 30 mL total) to the impure solid in the beaker.

Place the beaker on the hot plate.

Stir the mixture with a glass rod as it is being heated. If all the solid does not dissolve in the boiling solvent after a minute or two, add another 5 mL of solvent. Heat the mixture for another minute or so (with stirring) and observe whether the solid has dissolved to any greater degree than previously observed.

After the funnel has been warmed by the vapors from the beaker, add your solution containing your material to the funnel. Only add enough solution to cover half the filter paper at one time. Once the solution has been filtered, add an additional aliquot of hot 95% Ethanol, to dissolve any crystals that might be present.

Discard the filter paper into the Recovered Organic Solids jar at the front of the lab.

Allow the beaker containing the clear filtrate to cool to room temperature on the lab bench. During this cooling period, place 10 - 20 mL 95% Ethanol in your 50 mL beaker and place the beaker in an ice-water bath.

Clamp your 125 mL suction flask to a ringstand with a three-prong clamp, and place the smallest Buchner funnel (with rubber stopper attached) into the neck of the flask. Attach a length of black vacuum tubing to the side-arm of the flask and to the house vacuum line (the valve with the Yellow cap), or to the water aspirator.
After the mixture has cooled to room temperature, place the beaker in the ice bath. Place a disk of 4.25 cm filter paper into your Buchner funnel and wet it with a small portion of ice cold ethanol. Remove the beaker from the ice bath, turn on the vacuum, and filter the precipitated crystals with suction.

    a) If there are no - or very few - crystals at this point, see the instructor.

    b) If there appears to be no liquid in the beaker and you have a solid mass, add another 5 - 10 mL ethanol and stir the mixture, while cooling it in an ice bath. Then, filter the solid.

After all the solution has drained from the funnel, wash the crystals with about 5 mL ice cold ethanol.

When all the liquid has again drained from the funnel, turn the vacuum off slowly, and remove the funnel from the flask. Pour the filtrate into the "Recovered Ethanol" bottle.

Return any excess unused ethanol to the plastic ethanol bottle.

    NOTE: Pay no attention to any solid that may have precipitated from the solution inside the suction flask. This is quite normal and results from the evaporation of some of the water from the saturated solution under the reduced pressure in the flask. Any such solid is impure and should not be recovered.

Put the top portion of your buchner funnel, containing the recrystallized product in your locker. Your product will dry in your locker overnight.

    Determination of Melting Point Ranges

    PART I

    Melting Ranges of Impure and Recrystallized Unknown Solids

    NOTE: The Purified Product MUST be completely dry before its melting point can be determined.

You are to determine the melting range of your recrystallization product using the following procedure:

    Capillary Tubes and Sample Preparation

Commercially available capillary tubes have one end already sealed and are open at the
other end to permit introduction of the sample. The sample is put into the tube as follows:

Place a small amount of the solid whose melting point you wish to determine (your recrystallized product) on a clean watch glass and tap the open end of the capillary tube into the solid on the glass so that a small amount is forced about 2mm - 3mm into the tube. You may have to grind your crystals first into a fine powder in order to get your material into the capillary tube, then to get the solid to the closed end of the tube, take a piece of glass tubing 1 meter long and place this tube vertically on the benchtop, then drop the capillary tube (sealed end down) through the tube several times.

**Melting Point Determination**

The determination of the melting point involves taking the capillary tube containing the purified unknown and placing them in the slots that are seen through the magnifying glass of your melting point apparatus. The melting point apparatus is then allowed to heat until the contents of both tubes dissolve. Best results are obtained by heating the sample at a rate of about two degrees per minute. Many organic compounds undergo a change in crystalline structure just before melting, usually as a consequence of the release of water of crystallization. The solid takes on a softer, perhaps "wet" appearance, which may also be accompanied by a shrinkage of the sample in the capillary tube. These changes in the sample should NOT be interpreted as the beginning of the melting process. Wait for the tiny drop of liquid to appear. Melting invariably occurs over a temperature range, and the melting point range is defined as the temperature at which the first tiny drop of liquid appears up to and including the temperature at which the solid has melted completely.

**PART II**

**Mixture-Melting Point Determination**

Prepare a capillary tube containing only your recrystallized product.

On a piece of glassine weighing paper (DO NOT use filter paper) place a small sample of your purified product and a small amount of known compound you believe to be your product. Make an intimate mixture of the two substances by mixing the solids together thoroughly with your spatula.

Prepare a capillary tube containing the mixture.

On another piece of glassine weighing paper place a small sample of your purified sample and a small amount of another known compound you believe your product could be.

Make an intimate mixture of these two substances by mixing them thoroughly with your spatula.

Prepare a capillary tube containing the mixture from step 6.

Measure the melting ranges of the 3 samples. The apparatus can accommodate three capillary tubes, so melting range determinations can be done simultaneously.

Package the remainder of your recrystallized product in an appropriately labeled sample vial.
NOTES:

a) The actual melting ranges may differ somewhat from the literature values that are given in the list. Do NOT determine the melting range of any of the standards.

b) The melting range of your purified benzoic acid may not be exactly the same as the literature value because: a) the solid is not completely pure b) the sample was heated too rapidly in the melting point apparatus c) or thermometer error, etc.

Solid Compounds and their Melting Points

**Group A**

Benzhydrol, 69°C  
Biphenyl, 69°C  
Stearic Acid, 70°C

**Group B**

Benzamide, 133°C  
Cinnamic Acid, 133°C  
Urea, 133°C  
Phenacetin, 134°C

**Group C**

Benzanilide, 163°C  
Itaconic Acid, 164°C  
Sulfanilamide, 166°C
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Lab Report

**TITLE:** Recrystallization of an impure solid compound and its Identification

**Name:** (Your Name)                        **Unknown #**

**Date:**

**Weight of crude Unknown:** gm.

**Weight of recrystallized Unknown:** gm.

**% recovery:** %

**Melting Range of Recrystallized Unknown:** °C

Melting Range of Unknown and Standard(s):

**Test 1:** Standard/MP __________________________ °C

**Test 2:** Standard/MP __________________________ °C

**Test 3:** Standard/MP __________________________ °C

**Test 4:** Standard/MP __________________________ °C

**Test 5:** Standard/MP __________________________ °C

Identify your unknown by name. Justify your reasoning.