# Rowan College at Burlington County

# CHE 241

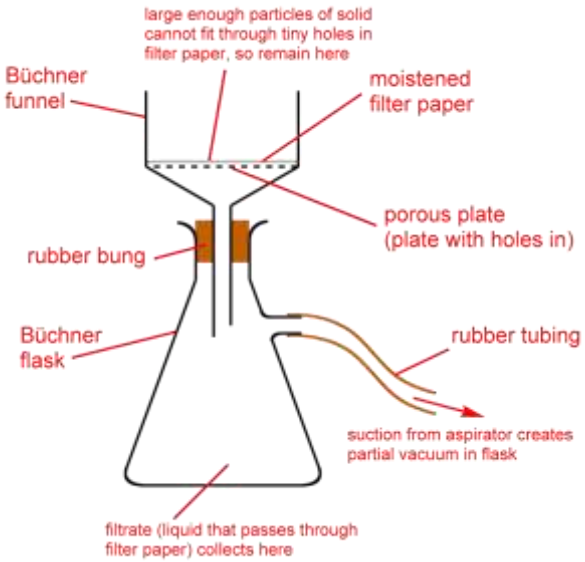
# Lab 2: Recrystallization and Melting Point Determination

**Objectives:**

1. Recrystallize a solid from a single solvent.
2. Recrystallize a solid by decolorization and a single solvent.
3. Recrystallize a solid from a mixed solvent.
4. Determine the melting point range of a pure substance.
5. Determine the melting point range of an impure substance.

**Benzil:**

Weigh approximately 1 g of benzil (record actual weight in the Data Table) into a 125 mL Erlenmeyer flask, then dissolve in hot ethanol by gradually adding small quantities to the flask containing the benzil. After the initial addition, the flask containing the hot ethanol and benzil should also be kept warm until all the crystals have dissolved. The solution is then allowed to cool to room temperature and then placed in ice. The crystals are filtered by vacuum through a Buchner funnel (see below). The crystals are removed from the funnel, allowed to dry until next class, and saved for melting point determination. The flask can be washed with cool solvent to assist in crystal transfer. The filtrate is placed in the waste ethanol container.




**Acetanilide:**

The starting material is crude and must be decolored using charcoal. Weigh approximately 1 g of acetanilide into a 150 mL beaker, add an initial 20 mL of hot water then gradually add hot water to the acetanilide, keeping hot until the crystals just dissolve. When the acetanilide is dissolved, add a slight excess of hot solvent to prevent crystallization during decoloring. The solution is then removed from the hot plate, a pinch of charcoal is added to the solution, and the solution returned to the hot plate for 5 minutes. Make sure that an excess of solvent is still present (no crystals present). To remove the charcoal, the solution is filtered through two fluted filter papers in a stemless funnel, which has been preheated, into a small beaker on the hot plate. Excess solvent is evaporated off if necessary. The filtrate is cooled and the crystals are vacuum filtered as before. The crystals are removed from the funnel, allowed to dry until next class, and saved for melting point determination. The filtrate may be discarded down the sink drain.

Example of hot filtration set-up.

**2-naphthol:**

The pure compound is colorless while the technical grade is purple. Recrystallization removes much of this color. Treatment with charcoal would remove even more, but is not required in this experiment. The solubility of this compound in water (0.0876 g/100 mL of solution at 29.55 ºC) is too low to permit recrystallization from a convenient volume; the solubility in ethyl alcohol (55 g/100 mL at 5.5 ºC) and most other solvents is too high. The best approach is to use a mixture of water and ethyl alcohol.

Weigh approximately 1 g of technical 2-naphthol into a 50 mL beaker and then dissolve in 5 mL of hot ethyl alcohol. Test the suitability of this solvent for recrystallization by cooling the solution. Re-warm the solution to room temperature and slowly add cold water dropwise until the solution becomes slightly cloudy and remains cloudy upon swirling (like a titration endpoint). Place the solution back on the steam bath until the cloudiness disappears. Remove the solution from the steam bath and cool immediately in an ice bath. Scratch the sides and bottom of the beaker with a glass stir rod to promote crystallization. Vacuum filter the crystals as in previous procedures. The crystals are removed from the funnel, allowed to dry until next class, and saved for melting point determination. The filtrate is placed in the waste ethanol container.

[End week 1 after completing all recrystallizations. Week 2 will require samples to be dried prior to melting point determination.]

**Procedure for Melting Point Determination:**

The Mel-Temp apparatus is used to determine the melting point of your solids. A ground sample of your substance is placed in a capillary tube. Grind the crystals to a powder with a mortar and pestle. Push the powder into a small pile and then push the open end of a melting point capillary tube into the powder so that some solid is forced into the opening of the tube. The sample is then shaken down into the closed end of the capillary by gently tapping the tube on the counter. If necessary, push more crystals into the open end and pack it down into the closed end so that you have no more than 3 mm column of solid at the bottom of the capillary tube, and no less than 2 mm. When a melting point apparatus becomes available be sure that it has cooled to below 70 °C prior to using.

The "mixed melting point" is of particular help in the identification of an unknown. When enough information has been gathered to permit an educated guess, a quick test may be made by intimately mixing the unknown with a known sample of the substance. If the two are the same, there will be no change in the melting "point"; if they are different, the mixture will have a lower (and broader) melting range. Refer to the introduction in the manual in terms of phase diagrams and effect of impurities on melting points.

The material tested should be ground so that fine, uniform powder is used, and the capillary tubes for multiple trials should be packed with equal amounts of material in order to give reproducible results. Care must be taken to use the correct amount of material in the capillary tube; too much material will give an inaccurate, broad, high melting point range; too little will give irreproducible results. Care must also be taken with the rate of heating at the melting point, too fast a rate will again give an inaccurate, broad, high melting point range. The Mel-Temp apparatus should automatically raise the temperature $1^{o}$ C per minute. You should set the “start” temperature at about $5^{o}$ below the literature value of the chemicals melting point. A melting point range must be taken, with both a beginning melting point and a final total melting point recorded.

**Notes:**

1. All flammable solvents are contained in Erlenmeyer flasks and heated on steam baths.

2. When using a Buchner funnel during vacuum filtration, the filter paper is always wet with cold solvent and suction applied to seal the system before filtration.

3. Solids are placed in wide mouth jars, or spread out on large watch glasses. The jar should be labeled prior to weighing and weighed empty. When the crystals are dry (allow at least two days for drying) reweigh the wide mouth jar with the crystals and determine the mass of the recrystallized product: this is the number of grams of recovered crystals used to determine the percent recovery.

$$\% \text{ recovery} = \frac{\text{mass of recovered crystals}}{\text{mass of crude crystals}} \times 100\%$$

4. Water may be heated on a hot plate and worked with in beakers since it is non-flammable.

CJF

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