Melting Point Determination
Chemistry 233: Organic Lab I

Procedure:

Obtain from the stockroom your two unknowns, A and B. Be sure to obtain a matched pair: i.e. if you choose unknown 12A, also choose 12B. Record the unknown numbers in your notebook prior to actually performing melting point determinations. Grind each sample to me melted into a fine powder using a clean scoopula and a clean, dry watch glass.

I. Your first determination should be quick and preliminary to get an approximate idea of where both unknowns A and B melt.

1. Place both unknowns in the melting point apparatus and turn the voltage control to full power. Note the melting "point" of each sample.

2. As soon as the higher melting sample begins to melt, turn the voltage control to very low power, allowing the block to begin to cool down in preparation for more accurate determinations. As soon as the block has cooled to approximately 10^{o}C below the melting point of the higher melting sample, remove and dispose of the previously melted samples and insert a fresh sample of the higher melting material.

3. Increase voltage to establish a heating rate of approximately 1-2^{o} per minute and record the melting range of the higher melting sample. As soon as melting begins, decrease the voltage. Normally, there is ample heat in the block to complete the melting process for the sample, and cool down will have begun in preparation of the next determination as early as is practical.

4. Allow the block to cool down below the lower melting sample and determine the melting range of a fresh sample of the lower melting compound.

II. After determining the melting range of both sample A and B, you are to determine the melting point of mixtures of B with specific, pure known compounds.

1. Several known compounds will have been placed in the melting point room by the stockroom personnel. Each bottle is labeled (name of the compound and melting range). Choose the compound that melted closest to your sample B and prepare three melting point tubes as follows:

   Tube 1 – place pure known compound
   Tube 2 – place a 50/50 mixture of pure known compound and unknown B
   Tube 3 – place your unknown B

2. Place all three tubes in the melting point apparatus at the same time and determine their melting range.

3. If all three samples have approximately the same range (within about 2 degrees), the substances are identical samples of the same material. If Tube 2 (the mixture tube) melts over a broad range and this range is significantly below the melting range of the

other two samples, the known and unknown substances are not the same. Should this latter case prevail, proceed to choose another known substance and repeat the process until a mixture is found that does not result in melting range lowering (and broadening).

4. Fill out a single unknown card, being sure to include your (1) unknown number, (2) the melting range for sample A and (3) the identity and melting range of sample B.

   Example:
   12A – 121-112^{o}C
   12B – Oxalic Acid, 132-134^{o}C