Week 5
Purification & Characterization of Synthesized Chemicals

“Perfection of means and confusion of ends seem to characterize our age.” —Albert Einstein

Scientist

Making a chemical is only the first step in the synthesis process. Before that chemical can be used it will be necessary to confirm that the product of the reaction is actually what was wanted. There are many techniques available to characterize chemicals and they range from the very simple to the quite complex (and expensive). This experiment will introduce some simple methods for characterizing the products of a chemical synthesis.

Educational Objectives: A student who has successfully completed this experiment will be able to
- perform and interpret a melting point determination,
- characterize a reaction product using TLC, and
- quantify a reaction product using titration.

Experimental Objectives: A student who performs this experiment is asked to
- confirm the Title identity and determine the purity of the reaction product using melting point, TLC and titration.

Background
Government regulations require proof not only of purity but also of the identity of the chemicals used in drugs and other products marketed for human consumption. As with purification there are many varied techniques available to confirm the identity of synthesized chemicals.

The simplest, cheapest and most common method of identifying solid organic compounds is melting point which is discussed in chapter 6 of AHC (chapter 7 of GC). Another quick and easy assessment tool is thin layer chromatography (TLC) which you employed earlier this semester. This is discussed in chapter 8 of AHC (chapter 6 of GC).

Titration is a quantitative method for determining the amount of a substance present in a sample. You will perform a simple titration as described in chapter 10 of both lab books.

The Problem
You will confirm the identity of your aspirin by determining its melting point and comparing your value to the accepted literature value. Pure aspirin will always have the same melting point. Impure aspirin will have a different value.

You will also perform a thin layer chromatography (TLC). This technique is used to separate similar chemicals. If your aspirin is pure, you will see only one spot. If impurities are present, you’ll see more than one spot.
Finally, you will perform a **titration** to determine the purity of your aspirin. In a titration you perform a reaction and measure all quantities of materials used. By stopping the reaction at the point where exactly equal amounts of material have reacted, you can determine how much of an unknown material is present.

![Chemical reaction diagram](image)

**Procedure**

Before beginning any procedures, reweigh your aspirin product from last week.

**Recrystallization**

If you were not able to complete the recrystallization the first week you will need to do so before continuing with the characterization procedures.

**Melting Point Determination.**
- The commonly accepted melting point range for aspirin is 135 - 136 °C.
- Closed ended capillary tubes will be provided. Remember that they are glass waste. Dispose of them accordingly.
- Following a melting point measurement a damp sponge can be used to lower the temperature of the hot block so that more samples can be measured more quickly.

**Thin Layer Chromatography.**
- You will be provided with the following: a silica gel coated glass plate to use as the stationary phase and acidified ethyl acetate (0.1% acetic acid in ethyl acetate) to use as the mobile phase.
- Open ended capillary tubes are provided for spotting samples. Note that these are not the same as those provided for the melting point determination.
- You are to chromatograph four samples: pure aspirin, salicylic acid, an aspirin tablet and your aspirin product.
- Preparation of the aspirin tablet. Grind up the tablet. Transfer the powder to a small beaker. Add about 20 drops of a 50/50 mixture of ethanol and dichloromethane to the beaker containing the crushed tablet. Swirl the mixture and let it stand for about 10 minutes to allow the aspirin to dissolve and the insoluble materials to settle.
- Preparing your aspirin. Put some on a watch glass and add a couple of drops of the 50/50 mixture of ethanol and dichloromethane. Work the mixture with a spatula to dissolve the aspirin.
- Solutions of pure aspirin and of salicylic acid prepared by the preproom are available for your use.
- Because aspirin is colorless you will not be able to see it on the plate. You will use both methods described in the lab book to view the spots on the plate.

**Titration**
- You are to titrate a small portion of your aspirin sample. If you know the weight of the sample you can calculate the moles and compare this to the titration result.
- You will be provided with a pre-standardized solution of NaOH to use to titrate your aspirin. The concentration will be about 0.1 M.
Phenolphthalein will be provided to use as an indicator.

**Data Analysis**

- Using the mass of your recrystallized product and the mass of your crude product determine the % recovery for the recrystallization. Yield calculations are discussed in chapter 3 of AHC (chapter 2 in GC).

- Review your melting point values and discuss the apparent purity of your aspirin.

- Review the TLC data and discuss the apparent purity of your aspirin.

- Using the titration data calculate the moles of aspirin in the sample. Compare this to the moles from the mass of the sample and the molar mass of aspirin. Express the ratio as a percent yield.