CHEM–333: Lab Experiment 1:
# Synthesis of N-Acetylanthranilic Acid: A Triboluminescent Material:

In addition to the experiment you perform this week, you will also need to check into your lab drawer and receive Right-to-Know and safety training. This will make for a full lab period, so work efficiently. <u>As always, bring your safety goggles and wear proper lab attire.</u>

<u>IMPORTANT:</u> Pre-lab assignment: YOU MUST READ all of Chapter 1 as a general introduction to the organic chemistry laboratory including very important safety information. There are many safety and procedural issues that are different than you may have had for general chemistry. For this experiment, read Chapters 2 and 3.

In this experiment you will learn how to perform your first organic reaction. At this point the mechanism likely is a bit advanced but you will learn some fundamental lab techniques such as suction-filtration and how to take an accurate melting point. You'll even learn what triboluminescence means first hand! Come prepared, relax and enjoy! -JK

Introduction:

The product, *N*-acetylanthranilic acid is prepared from reaction of anthranilic acid with acetic anhydride to produce an intermediate, benzisoxazinone (not isolated) which is immediately hydrolyzed to form the crystalline product.
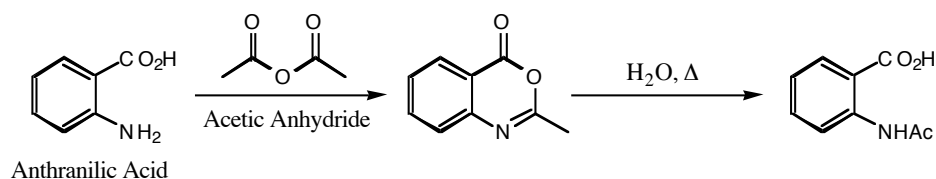
The product crystals, once isolated and dried, should exhibit the property of triboluminescence. It is left to you to find out what this property is and to come up with an idea of what may cause it, to include in your lab notebook write-up.

Procedure:

1. Weigh 2 g of anthranilic acid (FW = 137 g/mole) in the fume hood and place it in a 50 ml Erlenmeyer flask along with 6 ml of acetic anhydride. The mixture may solidify. Add a boiling stone and warm the mixture to a gentle boil on the hot plate for 15 minutes. Allow the reaction mixture to cool, then add 2 mls of distilled water. Warm the mixture until it nearly boils, then allow it to cool SLOWLY to room temperature. Crystals of the product, *N*-acetylanthranilic acid (FW = 179 g/mole), should form during this slow cooling period. Generally, the slower the cooling, the better the crystal growth.

2. Isolate the crystals by vacuum filtration using a small buchner funnel. With the vacuum on, transfer the crystals to the vacuum filtration apparatus with distilled water from your wash bottle. Wash the crystals with a <u>small</u> amount of chilled methanol, and suck them thoroughly dry. (Careful: Too much methanol will dissolve your product.)   Weigh your product. Calculate and report your percent yield. Obtain and report the melting point range (see notes below) for your product.

3. In the dark room provided, crush crystals of your product between the two watch glasses, looking for evidence of triboluminescence. You may want to give your eyes time to adjust to the darkness to better observe the effect. Report your observations. [Modified from: *J. Chem. Ed.*, Erikson, J. ; p. 688 (1972)]

<u>Notes</u>:

•   You will be using a Laboratory Devices Meltemp as picture on page 20.

•   You need only a small amount of sample (about 1/4 of a micro-spatula) for these measurements. Put only what is needed onto some weighing paper, and fill the melting-point capillary as instructed.

•   It is very important that the temperature of the Meltemp be well below the melting point of the sample (10 - 20 °C). Allow enough time between samples for the block to cool.

•   The dial on the Meltemp reads in volts. For these samples you should never need to go above 60 volts while for melting points around 100°, 50-55 volts gives a fast rise in temperature. For a slow rise in that range, 36 to 38 should be optimum close to the melting point.

•   The melting point of an organic compound is always reported as a range, not a single temperature. This range starts at the temperature at which the first drop of liquid appears and ends at the disappearance of the last trace of solid. If the sample melts <u>very</u> quickly at a single temperature, the melting point may be reported as 112.5° *sharp*, for example.