Synthesis of aspirin and oil of wintergreen essay sample

Literature, Russian Literature

Organic molecules have a wide range of applications occur both intracellular as well as in many different industries. The reactions use the reactivity of functional groups attached to organic molecules, as well as general chemistry concepts such as Le Chatelier's Principle (). The synthesis of Aspirin (its chemical name being acetylsalicylic acid) and of oil of wintergreen (with its chemical name as methyl salicylate) both occur by addition of an ester to the molecules.

Both syntheses have salicylic acid on the reactants side, however differ in the second reactant that ' esterifies' the salicylic acid. This is a vital process in the creation of Aspirin which is not only serves as an analgesic but also is used as a temporary blood thinner for patients at-risk for cardiac abnormalities. Following completion of the reaction forming oil of wintergreen, this shows how organic chemistry is a strong base in synthesizing different odors. Following the procedures to induce the reactions, other procedures included purifying the products, as well as testing the purity of each product, as well as the percent yield.

I. Introduction

Since the ' carbon backbone' (1) is generally unreactive, it is the specific polarity of the functional group (s) attached to the carbon backbone that allows organic molecules to react with a polar or nonpolar molecule. These reactions often depend on the electronegativity of the reacting components, as well as the number of free electrons available to react. As with all chemical reactions, organic reactions have optimal conditions (such as temperature) that allow the reaction to occur the quickest.

In addition, they often involve catalysts which help the reaction overcome an activation energy barrier, and in essence get the reaction going. In this experiment an acid catalyst was used, which donates a positively-charged hydrogen to one of the functional groups on the salicylic acid molecule, thus making the reacting portions of the molecules more likely to react by further separating their electronegativity's. The synthesis of acetylsalicylic acid (Aspirin) was very important in the pharmaceutical industry, by creating a way in which humans can tolerate the naturally-occurring salicylic acid molecule (1).

Likewise, the creation of different odors such as perfumes and mint is an enormous industry, which has a base in organic chemistry reactions. This laboratory experiment involves the synthesis of acetylsalicylic acid in one part and the oil of wintergreen in another, which both incorporate salicylic acid as the molecule being esterified. These reactions differ, however, in the functional group attached to the carbon backbone in salicylic acid that is taking part in the esterification process. In the formation of acetylsalicylic acid, a lone pair of electrons on the oxygen of acetic anhydride attacks the alcohol group on salicylic acid;

whereas in the creation of the oil of wintergreen, a lone pair of electrons on the oxygen in methanol attacks the carboxylic acid oxygen on the salicylic acid molecule.

The concept of Le Chatelier's Principle is also put to use in the second experiment (listed as Part 5) where oil of wintergreen was created. The

reactants and products in this reaction form an equilibrium prior to all of the reactants being used. For reactions at equilibrium, adding a reactant forces more product to be produced, since the reaction will find a new equilibrium by utilizing the excess reactant.

In the creation of oil of wintergreen, adding excess methanol after the initial reaction serves this function. In many industries, and especially in pharmaceuticals, a pure product is a necessity. There are a multitude of methods used to separate the different compounds in a crude product, and include taking advantage of the differing solubility's and boiling points of the compounds. Testing procedures are also available to verify a sample is composed of the pure compound of interest, which further points to the importance of purity.

II. Experimental Procedure (2)

Two separate reactions were performed in this laboratory experiment, both involving similar mechanisms of salicylic acid (an organic molecule) being ' esterified' by an electron-rich oxygen atom. In the formation of acetylsalicylic acid (Aspirin), salicylic acid and acetic anhydride were made to react in the presence of an acid catalyst, namely sulfuric acid. In the formation of methyl salicylate (oil of wintergreen), salicylic acid and methanol were reacted together in the presence of the same acid catalyst, sulfuric acid.

The designation of Part 1 for this experiment was used for the synthesis of Aspirin. To prepare the heating mechanism for the experiment, 250mL of tap

water was poured into a 400mL beaker, and this water was then heated on a hot plate to 85? C. As the water was heating, the mixture to be heated was concocted. First, 2. 595g of salicylic acid was weighed by first tarring a plastic weigh boat on a scale, and then adding the salicylic acid powder-like solid. This amount was then poured into a 125mL Erlenmeyer flask. Next, 5mL of acetic anhydride was measured using a 10mL graduated cylinder, which was then added to the same Erlenmeyer flask. Lastly, 5 drops of the concentrated sulfuric acid catalyst were added to the Erlenmeyer flask, and this was mixed for 5 seconds using a swirling motion.

The temperature of the water being heated in the beaker was measured using a digital thermometer, and once the water reached 85? C, the Erlenmeyer flask that contained the mixture was submerged in the water, occurring at 11: 26am. So that the flask could remain submerged without tipping, the neck of the flask was grasped using a utility clamp that was attached to a ring stand. The mixture remained submerged for 20 minutes.

During this waiting period, any used glassware was cleaned using a basic soap. In addition, 2mL of de-ionized water was measured in a 10mL graduated cylinder during this time. At the completion of the 20 minutes, the hot plate was turned off, and the Erlenmeyer flask was removed from the heated water and the 2mL de-ionized water was added. This water was used to wash away the acetic acid from the crude product. The next step in this reaction was to have the sample cool to 45? C by letting it rest on the counter. The temperature was monitored using a digital thermometer.

Next, 30mL of de-ionized water was measured in a 50mL graduated cylinder, and then this water was added to a 100mL beaker. Once the product finished cooling to 45? C in the Erlenmeyer flask, this was then added to the water in the beaker. There was some crystal formation following this addition.

To allow for further crystal formation, the 100mL beaker was then placed in an ice bath in an 800mL beaker for 20 minutes. This portion began at 11: 54am. During this time, Part 5 of the experiment was started, the synthesis of methyl salicylate (oil of wintergreen). To create this reaction, 1. 015g salicylic acid was weighed by first tarring a plastic weigh-boat on a scale, followed by adding the powdered solid. This reactant was then poured into a large test tube obtained from the student lab bench drawer. 5mL of methanol was measured in a clean 10mL graduated cylinder and was added to the large test tube. In order to jump-start the reaction 5 drops of the acid catalyst, concentrated sulfuric acid, was then added to the large test tube using a disposable plastic pipette.

Similar to the procedure from part 1, 250mL of water was poured into a 400mL beaker, and was heated on a hot plate until 70? C was reached. The temperature was measured using a clean digital thermometer. Once the water reached 70? C, the large test tube containing the reactants was submerged in the water at 11: 57am, and was held in place using a utility clamp to hold the test tube, which was attached to a ring stand. While the test tube remained submerged for 15 minutes, at 12: 07pm the solution in the test tube began to boil, due to methanol having a boiling point of 65? C.

Another 1mL of methanol was measured in a clean 10mL graduated cylinder, and was added to the test tube in order to generate more products.

At the completion of 15 minutes, the test tube was removed from the water at 12: 12pm and allowed to cool in an empty 400mL beaker until the product returned to room temperature. During this waiting period, Part 1 (the synthesis of acetylsalicylic acid), was revisited. Following the 20 minutes of the 100mL beaker cooling in the ice bath, the beaker was removed at 12: 14pm. During this time, larger crystals had formed along with a greater number of crystals, as compared to the crystals that formed following the cooling of the mixture to 45? C, as mentioned above.

The next step was to dry the product, however the house vacuum was not functioning properly. In order to begin drying, a 250mL filter flask was fitted with a Buchner funnel. In between the connection of the Buchner funnel and the filter flask, a rubber gasket was used to help maintain the seal. In addition, a round piece of filter paper was placed at the bottom of the Buchner funnel.

The product, which contained mostly solid crystals and some liquid, was emptied into the filtration set-up. Since there were still leftover crystals in the 100mL beaker, and the procedure called for extra washing of the crystals with de-ionized water, the leftover crystals was rinsed with about 1mL of de-ionized water and poured into the Buchner funnel. Though the procedure called for 5mL of water to be added, the product already contained a large

amount of moisture and the drying process available would likely not be able to remove the moisture completely.

The partially-dried product was then poured over a larger piece of filter paper, and was pressed between this and another piece of filter paper, being cautious not to contaminate the product. After allowing to dry for about 10 minutes, the product yield was weighed by first tarring a clean plastic weigh-boat on a scale and then adding the product to the weigh-boat. The yield was found to have a mass of 5. 492g, which ideally would contain pure acetylsalicylic acid. The percent yield of this product was then to be determined, using the formula: Percent yield = (actual yield/theoretical yield) x 100.

The actual yield was the mass found experimentally of 5. 492g, while the theoretical yield (shown in Table 1) below, was calculated using the number of moles of the limiting reagent, salicylic acid, which was 1. 879 X 10 -2moles salicylic acid. In order to find the amount of acetylsalicylic acid that could theoretically be produced, this number was multiplied by the molecular weight of acetylsalicylic acid, namely 180. 15g/mol. The theoretical yield was therefore calculated to be 2. 595g. Therefore from the equation of percent yield, this value was found to be 211. 6% acetylsalicylic acid. This sample calculation is displayed below under Calculation 4.

As to complete this portion of the experiment, a test of purity of the product was performed. First, 2 medium-sized test tubes were obtained from the front of the laboratory, cleaned using the basic soap and water, and dried. A

pea-sized portion of the product was added to the first test tube, using a clean scapula. The same sized amount of salicylic acid added to the other test tube using a separate clean scapula. Each test tube was then labeled with the contents it contained. 5mL of iron (III) chloride ($FeCl_3$) was then measured in two separate clean graduated cylinders (5mL in each cylinder) and 5mL was added to each test tube. Following the addition, a lighter magenta color resulted in the test tube with the experimental product yield, when compared to the tube containing the salicylic acid, which turned a darker shade of purple.

As this purity test completed the acetylsalicylic acid portion of the experiment, Part 5 of the experiment was returned to, to complete the formation of oil of wintergreen. The large test tube should have cooled to room temperature at this point, and now 3 drops of iron (III) chloride was added to the large test tube using a disposable plastic pipette. These drops were added one at a time in order to observe the results after each drop. After the addition of the first drop the solution immediately turned a significantly dark shade of purple. Following the addition of the next two drops, the solution became a dark shade of purple throughout.