Acetylsalicylic acid synthesis

ASSIGN BUSTER

Experiment # 3 Acetylsalicylic Acid Introduction: The purpose of this experiment is to create and isolate pure acetylsalicylic acid from the substances salicylic acid and acetic anhydride. Then one will find the melting point to determine purity. Procedure: Make a hot bath. Weigh some salicylic acid and place in conical vial. Add . 480mL of acetic anhydride and a drop of concentrated phosphoric acid. Drop in a magnetic spin vane and attach air condenser to vial. Partially submerge it in hot water. Stir with spin vane to dissolve salicylic acid. After mixture dissolves, heat for 8 to 10 minutes. Crystallize the Acetylsalicylic acid by cooling the mixture. Then add 3. 0mL of water and stir. Set up a Hirsh funnel for vacuum filtration. Add 1mL of cold water to help transfer all of crystallized substance. Rinse crystals with water once all collected in funnel. Let crystals dry in funnel. Let finish drying on a watch glass for the week. After a week take the product, and weigh it in a pre-weighed beaker. Calculate the percent yield. Test for Purity. Take 3 test tubes filled with water and dissolve a little bit of crystals in the first 2 tubes. Add 1 drop of 1% ferric chloride solution to each tube. Shake and note color. If impure, red to violet color will appear. Melting point can be used to test for purity. Melting point from a dry sample should be 135-136°C for pure aspirin. There are impurities if much less than that. Data and Calculations: Amount of Salicylic Acid – 0. 216g Amount of Acetic Anhydride – 0. 480g Amount of final product (acetylsalicylic acid) – 0. 144g Percent Yield: 0. 144g Acetylsalicylic Acid 0. 216g Salicylic Acid X 100% = 66. 667% Melting Point: 120-123°C Ferric Chloride Test: Test Tube Content Results Salicylic Acid Deep Purple Acetylsalicylic Acid (Product) Light Lilac Water (control) Clear& Colorless *All tubes contain 0. 5mL of water Results and Discussion: This procedure produced 0. 144g (66. 67% yield) of acetylsalicylic acid. It was a white sold

crystal substance. The melting point was lower than the literature mp of 135-136°C. The temperature recorded for this substance was 120-123°C indicating that it was an impure substance. The exact temperature is not certain but it was within that range. The temperature was changing during the melting process and the whole substance melted while the temperature was within that range. The substance that could have been left over was salicylic acid which did not react fully at the beginning of the reaction. Other impurities could have been from unclean containers or from the environment. However, that is probably not the case. Impurities also could have not been dissolved fully by the water so remained. To find out if salicylic acid, a phenol, was the impurity present in the product the Ferric Chloride test was performed. It showed that the when a substance has salicylic acid it turns purple. When it is a pure substance it does not change and is clear and colorless. The product which was mostly acetylsalicylic acid did have some salicylic acid in it because it turned a light lilac/ purple color. Some sources of error and reason for the low percent yield could be from many different things. During the filtration of the substance some of the substance could have got through and was lost in the filter flask solution. The transferring of product from the filter paper to the watch glass to beaker could have caused more of the product to be lost. Another error could have been caused by using different scales to weigh the original salicylic acid and the final product. The purpose of the concentrated phosphoric acid was to have act as the catalyst for the reaction. If it were left out the reaction would probably not occur or take a long time for it to occur. References: 1. Pavia, D. L., Lampman, G. M., Kriz, G. S., Engel, R. G., Organic Laboratory Techniques,

a Microscale Approach, Third Edition. Philadelphia: Sanders, 1999, pp. 102-105.