Multistep synthesis of benzilic acid essay

The objective of the experiment was to prepare Benzilic acid by multistep synthesis starting with benzaldehyde. In this setup however, product of the first step, Benzoin, is provided thereby omitting the first step involving the conversion of benzaldehyde. For this experiment, the microscale techniques of reflux, crystallization, and melting-point determination were used. Utilizing these techniques a product yield of 93% for benzil and 57% for Benzilic acid was obtained, as well as a melting point range of 94. 8 – 95. 4oC for benzil and 152. 0 – 153. 9oC for Benzilic acid. With the literature melting-point value of benzil being 95oC (Pavia, 2012), the product was deemed of pure enough quality to be used in the preparation of Benzilic acid.

The melting point of Benzilic acid was taken twice, the first being155. 4 – 156. 5oC, and the second being 152 – 153. 9oC. Another melting point was taken due to suspicion of a wet product as suggested when compared to the literature value at 150oC (Pavia, 2012). The second melting point having decreased from the first indicated that the product was indeed wet however of pure quality nonetheless, concluding the reaction was overall a success.

Introduction: Overall Reaction:

Mechanism:

The oxidation of Benzoin to Benzil and conversion of Benzil to Benzilic acid are two of the main steps that make up this multistep reaction. This being a multistep reaction meant that the success of synthesizing the end product Benzilic acid would be completely dependent on the success of every step that came before it. Since Benzil is required to form Benzilic acid, it makes sense then that the first step be the production of Benzil, which was formed

from Benzoin. This reaction required the oxidation of the α – hydroxyketone, Benzoin, by Nitric acid. Both reactions in this experiment however required refluxing of the reaction to maintain the amount of solvent and allow the reaction to proceed at a high temperature (Pavias, 2011).

For this first step however, it was crucial to assure the continual evolution of the NO gas out of the air condenser which would shift the equilibrium to the side of the products. To ensure the highest yield in product, the reaction was carried out until no more NO gas was produced which indicated the completion of the reaction. The second major step involved the conversion of Benzil into Benzilic acid which began with refluxing of Benzil with potassium hydroxide. The hydroxide ion converted Benzil to the Benzilate by a simple rearrangement mechanism where a phenyl group bonds to the double bonded carbon triggered by the delocalized pairs of electrons on the oxygens, being pushed back and forth.

Formation of the salt acts as a driving force for the reaction for it is later combined with cold acid to protonate it to its Benzilic acid form. Cold acid was used as to aid in the precipitation of the crystal product as most solvents lose capacity at lower temperatures (about. com). In order to analyze whether the reactions were successful, a melting point determination was taken following each step as well as infrared spectroscopy to confirm the identity of the end products and gauge its purity. Performing a infrared spectrum on a successful production of Benzil following the first step wouldn’t yield a very interesting IR and would simply include a strong peak around ~1660 indicating strong presence of carbon double bonded oxygens and a small spike around ~ 3000 indicating sp2 carbons.

The infrared of a successful production of Benzilic Acid however would yield a more complex IR consisting of a hydroxyl peak ~3400, and broad peak at ~2800, and a strong peak at around ~1700 indicating a strong presence of carbon doubled bonded oxygen's. Melting point determination of a successful product of Benzil and Benzilic acid would result in a melting – point range that is small and narrow around the literate point of 95oC for Benzil and 150oC for Benzilic acid. A broad range melting point on the other hand coupled with an infrared spectrum with no similar key point when compared to a reference would indicate a different and impure product (Pavias, 2011). Procedure

To prepare Benzil, . 30g of Benzoin was placed in a 5-mL conical vial with 1. 5mL of concentrated nitric acid. With the addition of a spin vane and attachment of an air condenser, the mixture was heated and stirred for an hour in a hot water bath at 70oC. The red nitrogen oxide gases were allowed to evolve completely before the air condenser was detached and the mixture was transferred to a beaker containing 4mL ice-cold water then cooled via ice bath. The crude product was then collected and washed with 5mL cold water through vacuum filtration. The weighed product was then transferred and dissolved with hot 95% ethanol in another flask over light heat.

Once dissolved completely, the flask was removed from heat, seeded, and then allowed to cool first at room temperature, than in an ice-bath. Following crystallization, the product was collected and rinsed with 95% ethanol vacuum filtration for 5 minutes. After air-drying, the dry benzil was weighed and a melting point was taken. To rearrange into Benzilic acid, . 100g of benzil and 95% ethanol was mixed into a 3mL conical vial. Following the

addition of a spin vane and attachment of an air condenser, the mixture was heated with an aluminum block at 95oC. After dissolving completely, . 25mL of aqueous potassium hydroxide was added and gently boiled at 110oC with stirring for 15 minutes.

The brown mixture was transferred to a beaker, cooled and then bathed in ice-water for 15 minutes. The crystals were collected and washed with 3 portions ice-cold 95% ethanol under vacuum filtration. The product was then transferred into a flask containing 3mL of water at 70oC and then stirred. While swirling the flask . 50mL of 1M hydrochloric acid was added. Following a period of cooling, the product was then ice-bathed. The product then collected and washed with 3-4mL ice cold water through vacuum filtration. After air-drying for a few minutes, the weight was recorded followed with melting point characterization.