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Preparation of p-Nitro Acetanilide from Acetanilide **Principle**: The nitration of aniline is challenging due to the oxidation of amino groups and formation of a mixture of products. Therefore, the amino group is first protected by acylation to form acetanilide, which is then nitrated to produce p-nitroacetanilide as the major product. **Chemicals Required**: Acetanilide, concentrated HNO3, concentrated H2SO4, CH3COOH, ethanol **Apparatus**: Conical flask, thermometer, beaker, funnel, measuring cylinder, filter paper The Preparation of p-Nitroacetanilide: A Step-by-Step Guide p-Nitroacetanilide is a crucial compound used as a starting material for various commercially valuable products, including dyes, drugs, and explosives. In this experiment, we will demonstrate the preparation of p-nitroacetanilide through the nitration of acetanilide. **Principle** The p-Nitroacetanilide is prepared via the nitration of acetanilide. The acetamido group (-NHCOCH3) in acetanilide is ortho and para directing, resulting in a mixture of o- and p-nitroacetanilide being formed. **Objectives** After performing this experiment, we should be able to: 1. Recrystallize the p-Nitroacetanilide 2. Prepare p-Nitroacetanilide 3. Determine the melting point of prepared p-Nitroacetanilide 4. Perform other compounds' nitration **Step-by-Step Procedure** HNO3 + H2SO4 + H2NO3 + HSO4 → NO2 - H2O Step 2: Attack on Electrophile HN N O HN NO2 H HN HN H O2N Ortho-substituion Para-substituion O O O O O N O O **SPP-1.8 (1.4A) Practical Organic Chemistry** DOS & R in Organic Chemistry, TUT Page 6 Step 2: Loss of proton and formation of product HN HN H O2N O O N O O HSO4 HN NO2 O **Chemical Tests** Reduction Test: Acetanilide does not undergo reduction with Sn and no dye formation (-Ve) p-Nitroacetanilide: Undergoes reduction with Sn and dye formation (+Ve) Diazotization Test: Acetanilide: Does not undergo reduction with Sn and no dye formation (-Ve) p-Nitroacetanilide: Undergoes reduction with Sn and dye formation (+Ve) **Melting Point** Theoretical Melting point: 214oC Experimental Melting point: [Insert experimental melting point] **Report** Structure and name of the compound: HN NO2 O p-nitro acetanilide Theoretical yield: [Insert theoretical yield] Practical yield: [Insert practical yield] Percentage yield: [Insert percentage yield] **References** 1. 2. Organic compound p-nitro acetanilide can be synthesized from acetanilide through nitration. When acetanilide is treated with a nitrating mixture, comprising sulphuric acid and nitric acid, it yields p-nitro acetanilide, accompanied by o-nitro acetanilide as a minor product. The latter's high solubility in alcohol facilitates the isolation of p-nitro acetanilide through crystallization. The chemical reactions involved are an electrophilic substitution reaction, where the electrophile -NO2 attaches to the para position due to the electron-releasing group -NHCOCH3. This form of reaction is used to prepare nitro anilines, as direct nitration of aniline would lead to amino group oxidation. Materials Required: - Acetic acid - Acetanilide - Fuming Nitric acid - Concentrated Sulphuric acid - Ethyl alcohol - Beaker - Conical flask - Filter paper - Dropping funnel - Buchner funnel - Glass rod - Pipette Procedure: 1. Dissolve 3 grams of acetanilide in glacial acetic acid at room temperature. 2. Warm the solution gently to ensure complete dissolution. 3. Cool the mixture and slowly add concentrated sulphuric acid while stirring. 4. Maintain the temperature below 20°C throughout the process. 5. Add fuming nitric acid dropwise through a dropping funnel, stirring constantly. 6. Once the addition is complete, remove the beaker from the ice-bath and let it stand at room temperature for 30 minutes. 7. Pour the mixture into 100 grams of crushed ice and stir well to obtain large crystals of p-nitro acetanilide. 8. Filter the crystals using filter paper and wash with cold water to remove excess acid. 9. Crystallize from ethyl alcohol, then dry the crystals in filter paper folds and weigh them to determine the yield. Observations: Color of the Crystals: Colorless Expected Yield: 4 Grams Melting Point: 214°C **Storage and Labeling** List quantities: * Acetanilide: 25 g * P-Nitroacetanilide: [amount not specified] Garage location: Not applicable (chemicals) Storage technique: * Label all boxes with the name, hazard information, manufacturing date, and statistics on the hazards associated with the chemical being stored. * Keep protection information sheets from chemical suppliers handy for reference. **Reaction Information** Principle: Prepare p-Nitroaniline from acetanilide via nitration and hydrolysis. Mechanism: 1. Electrophilic aromatic substitution (nitration) of nitronium ion towards para position of acetanilide due to steric reasons. 2. Hydrolysis of acetate ion from acetamido functional group in presence of concentrated sulphuric acid, resulting in p-Nitro aniline. **Safety Precautions** * Handle ethanol with care, as it is flammable. * Avoid direct or indirect contact and inhalation/ingestion of sodium acetate trihydrate, acetophenone oxime, acetophenone, hydroxylamine hydrochloride (defined as corrosive), and deuterochloroform (defined as toxic). * Handle all chemicals with caution. **Apparatus** Conical flask Beaker Pipette Glass rod Buchner funnel Add 150 ml of 70% H2SO4 (prepared by mixing 100 ml concentrated acid with 75 ml water) to a round-bottomed flask. Reflux the mixture for 20-30 minutes and then pour the hot solution into 1000 ml of cold water. Neutralize the mixture with 10% NaOH, cool it down, and filter the resulting yellow crystalline product on a Buchner funnel. Thoroughly wash the product with water. For recrystallization, use a mixture of equal volumes of rectified spirit and water or hot water. Calculation: The limiting reagent is acetanilide, so the yield should be calculated based on its amount. The molecular formula for acetanilide is C8H9NO, while that for p-nitroaniline is C6H6N2O2. The molecular weight of acetanilide is 135 g/mole, and that of p-nitroaniline is 138 g/mole. Theoretical yield: 25 g of acetanilide will form X (X) g of p-nitroaniline. Calculating the theoretical yield gives X = (138 × 25)/135 ≈ 25.55 g. Practical yield: ?? g Percentage yield: (Practical Yield)/(Theoretical Yield) × 100 P-nitroaniline was synthesized, and the percentage yield was found to be ???%.