**CH 246: ORGANIC CHEMISTRY II LABORATORY (Spring 2021)**

**Title:** Cannizzaro Reaction

1. **Purpose: (1 point)**

**­­­The purpose of this experiment is to demonstrate the Cannizzaro Reaction by preparing p-chlorobenzoic acid and p-chlorobenzyl alcohol from p-chloro benzaldehyde.**

1. **Drawing of structure of the main compound or balanced chemical equation if synthesis is performed: (1 point)**

Diagram

Description automatically generated

**3. Reagents and the major product (up to 5 points)**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Name** | **M.W.**  (0.5 pts) | **Density**  (0.5 pts) | **Amount (grams/mL)**  (0.5 pts) | **Moles**  (0.5 pts) | **Hazards/Precautions**  **(MSDS data) and melting point or boiling point** (2 pts) | **Role of the reagent** (1 pts)\* |
| p-chloro benzaldehyde | 140.57 | 1.196 g/cm3 | 2.0 grams | 0.0142 | Causes eye and skin irritation. May be harmful if swallowed.  MP: 46-48°C  BP: 214°C | Reactant |
| Potassium hydroxide | 56.1 | 2.04 g/cm3 | 3.2 grams | 0.0570 | Corrosive. Water reactive. Causes severe eye and skin burns. Causes severe digestive and respiratory tract burns.  MP: 360°C  BP: 1320°C | Reactant |
| Methanol | 32.04 | 0.792 g/cm3 | 4 mL | 0.0988 | Poisonous. Flammable liquid and vapor. Harmful if swallowed, inhaled, or absorbed through skin. Causes eye, skin, and respiratory tract irritation.  MP: -98°C  BP: 64.7°C | Solvent |
| Dichloromethane | 84.93 | 1.33 g/cm3 | 50 mL | 0.783 | Harmful if swallowed. Causes eye, skin, and respiratory tract irritation.  MP: -97°C  BP: 40°C | Solvent |
| Sodium Bicarbonate | 84.01 | 2.2 g/cm3 | 10 mL 10% solution | -- | Causes eye and skin irritation. May cause respiratory tract irritation.  MP: 270°C | Solvent |
| Magnesium sulphate | 120.37 | 2.65 g/cm3 | 1 gram | 0.0083 | May cause eye, skin, and respiratory tract irritation. Hygroscopic.  MP: 1124°C | Drying agent |
| Hydrocholoric acid (38%) | 36.46 | 1.19 g/cm3 | 10 mL | 0.326 | Causes eye and skin burns. Corrosive. Causes digestive and respiratory tract burns.  MP: -66°C  BP: 83°C | Solvent |
| p-chlorobenzoic acid | 156.57 | 1.57 g/cm3 | -- | -- | Causes skin irritation. May cause eye and skin irritation. May cause respiratory and digestive tract irritation.  MP: 240-242°C | Product |
| p-chlorobenzyl alcohol | 142.58 | 1.2 g/cm3 | -- | -- | May cause eye and skin irritation. May cause digestive and respiratory tract irritation.  MP: 70-72°C  BP: 234°C | Product |

**\*** Mention role as either reactant, solvent, catalyst or product

**4. Calculations: (1 point)**

Show each calculation for moles of reagents and for theoretical and actual yield. Fill in the box with the limiting reagent and theoretical yield:

p-chloro benzaldehyde

The limiting reagent is

1.113 g p-chlorobenzoic acid

1.014 g p-chlorobenzyl alcohol

The theoretical yield is

**5. Procedure (up to 2 points)**

|  |  |
| --- | --- |
| **Procedure** | **Observations and Lab Data** |
| A summary of the procedure done with bullet points) | Color changes, exothermic or endothermic reactions, gas generation, etc.; tare weights for flasks, etc. |
| * Dissolve 3.2 g of potassium hydroxide in 8 mL of water in an Erlenmeyer flask. Cool to room temperature. * Add slowly, with swirling, a solution of 2.0 g p-chlorobenzaldehyde in 4 mL of methanol. Swirl the flask gently during addition. Add a stir bar. * Warm to 55-60°C for at least 45 minutes while stirring. The mixture should turn into a cloudy solution or slurry. * Cool the solution and extract with two 20mL portions of dichloromethane. If reaction mixture still contains solid, wash with extra 5-10 mL DCM. Combine DCM extracts. Save aqueous layer. * Wash the combined organic layer once with 10% sodium bicarbonate solution and then with 10 mL of water. Dry using 1 g magnesium sulfate. * Distill off the DCM using distillation apparatus. Cool the residue remaining in boiling flask and then in ice to crystallize. * Collect the crude p-chlorobenzyl alcohol, record yield and melting point. * Recrystallize with 5% acetone-hexane mixture if necessary. * Acidify the aqueous layer with conc. HCl (8-10 mL). Check pH of solution using pH paper (should be ~1). A thick precipitate will be formed. * Collect the precipitate with suction filtration. Wash thoroughly with cold water. Dry and record crude yield and melting point of p-chlorobenzoic acid. * Recrystallize if necessary from methanol. |  |

**6.** Results; include actual yield in grams and % yield.

**Results (need to get signed by instructor or TA):**