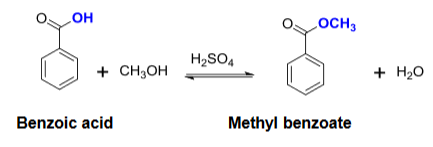
**CH 246: ORGANIC CHEMISTRY II LABORATORY (Spring 2021)**

**Title:** Preparation of Methyl Benzoate from Benzoic Acid

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

**To synthesize methyl benzoate from benzoic acid via Fischer Esterification and monitor the reaction with thin layer chromatography.**

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



**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)\* |
| Benzoic Acid | 122.12 | 1.27 g/cm3 | 2.5 grams | 0.0205 | Causes eye, skin, and respiratory tract irritation. Harmful if swallowed.  MP: 122.4°C  BP: 249.2°C | Reactant |
| Methanol | 32.04 | 0.791 g/cm3 | 8 mL | 0.198 | Poisonous. Harmful vapor. Flammable. Causes eye, skin, and respiratory tract irritation.  MP: -98°C BP: 64.7°C | Reactant |
| Sulfuric Acid | 98.07 | 1.83 g/cm3 | 0.6 mL | 0.0112 | Very strong acid. Causes eye, skin, digestive, and respiratory tract burns.  MP: 10°C  BP: 290-338°C | Catalyst |
| Dichloromethane | 84.93 | 1.33 g/cm3 | N/A | N/A | Harmful if swallowed. Causes skin, eye, and respiratory tract irritation.  MP: -97°C BP: 40°C | Solvent |
| Methyl Benzoate | 126.0548 | 1.08 g/cm3 | N/A | N/A | May cause eye, skin, respiratory tract irritation. Combustible.  MP: -12.2°C  BP: 150°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:

Benzoic Acid

The limiting reagent is

2.58 grams

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. |
| * Measure out 2.5 grams of benzoic acid and 8 mL methanol, and place into 25-mL round bottom flask. * Carefully pour 0.6mL concentrated sulfuric acid down the wall of flask. (Take 0 min TLC sample) * Add boiling chips and reflux mixture for 1 hour with heating mantle. (Take 30 min TLC and 1 hour TLC samples during heating) * After reflux, cool mixture down to room temperature and pour in separatory funnel containing 5 mL of water and 5 mL of dichloromethane. * Rinse the flask with 2-3 mL of dichloromethane and pour into funnel. * Shake the mixture and separate organic layer from aqueous. * Wash organic layer with 5 mL of water followed by 5 mL of 5% sodium carbonate. * Separate and pour organic layer into Erlenmeyer flask. (Take final TLC sample)   **TLC:**   * Obtain 1 mL of each sample to be taken for TLC. * Take 2 TLC plates. With pencil, draw a line 1 cm from both the top and bottom of the plate. * On the first plate, make four spots of 0 min, 30 min, 60 min, and after washing samples of organic solution. * On the second plate, make three spots of known benzoic acid solution, methyl benzoate solution, and organic solution after carbonate wash. * Let dry for a few minutes, then put each plate in TLC chambers for development. * For first plate, calculate Rf of each spot. For second plate, compare experimental spot with known spots to ensure synthesis of methyl benzoate. |  |

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

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