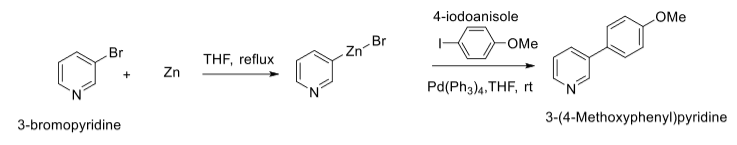
**CH 412 LA: INORGANIC CHEMISTRY LABORATORY (Spring 2021)**

**Title:** Synthesis of 3-(4-Methoxyphenyl)pyridine using Pd(Ph3)4

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

**The purpose of this experiment is to demonstrate the use of the Pd(Ph3)4 catalyst in coupling reactions to form 3-(4-Methoxyphenyl)pyridine.**

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)\* |
| 3-bromopyridine | 158.00 | 1.6570 g/cm3 | 15.8 grams | 0.1 | May be fatal if absorbed through skin. Flammable liquid and vapor. Causes eye irritation.  BP: 192-194°C | Reactant |
| Zinc | 65.38 | 7.133 g/cm3 | 9.81 g | 0.150 | Water reactive. Causes eye and skin irritation. Causes digestive and respiratory tract irritation.  MP: 419°C  BP: 908°C | Reactant |
| Tetrahydrofuran | 72.10 | 0.89 g/cm3 | -- | -- | Highly flammable. Causes eye and respiratory tract irritation.  MP: -108.5°C  BP: 66°C | Solvent |
| Pyridyl zinc bromide | 223.39 | 0.974 g/cm3 | -- | -- | Highly flammable liquid and vapor. Harmful if swallowed. Causes severe skin burns and eye damage. | Intermediate |
| 4-iodoanisole | 234.03 | 1.7 g/cm3 | 1.80 grams | 0.008 | May cause eye and skin irritation.  MP: 46-51°C  BP: 237°C | Reactant |
| Pd(Ph3)4 | 1155.58 | -- | 0.10 grams | 0.0000865 | Harmful if swallowed. May form combustible dusts.  MP: 105°C | Catalyst |
| 3-(4-Methoxyphenyl)pyridine | 185.22 | 1.077 g/cm3 | -- | -- | No data.  MP: 62-63°C  BP: 321°C | Product |

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

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

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

The limiting reagent is

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. |
| * Place 9.81 grams of zinc in a 250-mL round bottom flask with 100 mL THF. * Add 15.8 grams 3-bromopyridine via a cannula while stirring at room temperature. * Continue stirring mixture while refluxing. When reaction is finished, and settled down overnight, transfer supernatant to 500 mL bottle and dilute with fresh THF up to 200 mL. * In a 50 mL round bottom flask, add 0.10g Pd(Ph3)4. * Add 20 mL of 0.5 M 3-pyridyl zinc bromide in THF at room temperature. Add 1.80 g iodoanisol dissolved in 10 mL THF via a syringe. * Stir the resulting mixture at room temperature for 1 hour. * Quench reaction with saturated NH4Cl solution, then extract with 3 x 30mL ethyl acetate. Wash with NaHCO3 solution and brine, then dry over anhydrous MgSO4. |  |

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

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