Chemistry 260 Summer 2024

**Experiment 13: Green Chemistry Considerations for an Amide Coupling Reaction**

**<<This report will be completed as an “in-lab assignment” that you will work on and hand in at the end of the period. You can do some of the work (e.g., calculations, some of the Analysis sectoin) in advance and bring your own copy of this template if you wish, but copies will be provided in the lab.>>**

Name: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Section:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Date: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

**Reaction Scheme (1 mark)**

**Observations (2 marks)**

**Reagents and Products Tables (2 marks)**

Table 1. Reagents for the amide coupling reaction is attached as appendix A.

Table 2. Product of the amide coupling reaction

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Compound | Physical Description | MW (g/mol) | Amount Isolated | mmol | % yield |
| N-benzyl-3-phenylpropanamide |  |  |  |  |  |

**Results**

**Percent Yield Calculation: (1 mark)**

**IR Data: (2 marks)**

Table 3. **KEY** signals in the IR of the product (only list key peaks used for identification). Attach the printout as Appendix B.

|  |  |  |
| --- | --- | --- |
| Wavenumber (cm-1)  | Strength (s/m/w) | Assignment and/or Comment |
|  |  |  |
|  |  |  |
|  |  |  |

**Analysis (In-Lab Assignment):**

 **Use the below table to summarize the data collected by your group of 4: (2 marks)**

Table 4. Summary of group results

|  |  |  |  |
| --- | --- | --- | --- |
| Group Member | Conditions tested  | Yield (%) | Comment on purity (colour, IR)  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |

**With your group, complete the portion of the table on the next page for your assigned method. Point form notes are fine. Include in brackets which Principle(s) of Green Chemistry applies to each point [e.g., ‘the reaction requires high pressure to work (Principle 6)’ would go in the ‘Con’ column]. You will also make a large form summary of your assessment to present to the class during the tutorial period using the large paper and markers available in the lab. Your large form summary should include which set of conditions you have determined is the best from those you tested. You will be asked to take notes to fill in the analysis of the method that the other group tested during their presentations.**

**(10 marks)**

Table 5. Summary of class analysis

|  |  |  |  |
| --- | --- | --- | --- |
|  |  | **Pros** | **Cons** |
| **Method 1** | **toluene** |  |  |
| ***p*-xylene** |  |  |
| **boric acid** |  |  |
| **workup** |  |  |
| **overall** |  |  |
| **Method 2** | **THF** |  |  |
| **2-MeTHF** |  |  |
| **CDI** |  |  |
| **LAG** |  |  |
| **workup** |  |  |
| **overall** |  |  |

**Of the conditions your group tested, which would you select as the best conditions for a scaled-up reaction, keeping in mind the 12 Principles of Green Chemistry? Explain why. (5 marks)**

**Of the conditions the whole class tested (i.e., consider both methods), which would you select as the best conditions for a scaled-up reaction, keeping in mind the 12 Principles of Green Chemistry? Explain why. (5 marks)**

Appendix A: Table of reagents (only fill in the ones you used).

| Compound | MW (g/mol) | Used | mmol | Physical and Safety Data |
| --- | --- | --- | --- | --- |
| hydrocinnamic acid | 150.175 |  |  | Irritant. M.p. 45 – 48 °C |
| benzylamine | 107.15 |  |  | Flammable, corrosive. density 0.981 g/mL |
| boric acid | 61.83 |  |  | Reproductive toxin (harmful to the unborn child.  |
| 1,1′-carbonyldiimidazole (CDI) | 162.15 |  |  | Corrosive, m.p. 115 – 122 °C |
| toluene | 92.14 |  |  | Flammable liquid; toxic. Bp 110 °C. Density 0.865 g/mL. |
| *p*-xylene | 106.17 |  |  | Flammable; harmful; Bp 138.4 °C density 0.861 g/mL |
| hexanes | 86.18 |  |  | Flammable; highly toxic; density 0.672 g/mL; bp 68-70 °C |
| tetrahydrofuran  | 72.11 |  |  | Flammable. Bp 65-67 °C; density 0.889 g/mL |
| 2-methyltetrahydrofuran | 86.134 |  |  | Flammable. Bp 78 °C; density 0.854 g/mL |
| ethyl acetate | 88.11 |  |  | Flammable, irritant. Bp 76.5-77.5 °C; density 0.902 g/mL |
| citric acid (aq) | N/A |  |  | Irritant. |
| sodium bicarbonate (aq) | N/A |  |  |  |