

# Problem in diastereoselectivity



**ASSIGN  
BUSTER**

Purpose: The purpose of this experiment is to determine the stereochemical outcome of a reaction. A second chiral center is being formed in this experiment and two diastereomeric compounds are created.

Reagents: Safety Precautions or Hazards:

- Do not place pipettes in paper trash. Must be thrown away in the broken glass container.
- Take caution when adding HCl.
- CO<sub>2</sub> evolution may be vigorous so be sure to vent funnel.
- Never directly heat ethanol over a hot plate, use water bath.
- Conc. H<sub>2</sub>SO<sub>4</sub> is highly corrosive.

Equations for all Reactions: Mechanism(s):

Experimental Procedure: Part A:

1. Add a magnetic stir bar, 2g of benzoin, and 20 ml of absolute ethanol in a 125ml Erlenmeyer flask.
2. While stirring, (do not turn on the heater) carefully add 0.4 g (10.6 mmol) of sodium borohydride portion-wise to the mixture over 5 minutes. After the addition is complete, stir the mixture for another 15 minutes at room temperature. A white precipitate will form.
3. Cool the flask in an ice-water bath and decompose the excess sodium borohydride by first adding 30 mL of water followed by the careful and dropwise addition of 2 mL of 3M HCl. The HCl addition should be done quite slowly, no more than about 3 drops per minute (maybe 10-15 minutes total time). The mixture may foam uncontrollably if the acid is

- added too quickly. If the foam reaches the 100 mL mark on the flask, stop adding HCl until the foaming subsides.
4. When the HCl addition is complete, add another 10 mL of water and stir the mixture for 15 minutes.
  5. Collect the white precipitate by suction filtration. Wash the product diol with water on the suction funnel (about 150 mL) and allow the product to air dry on a filter paper. Safely store the product in your drawer until next week. The filtrate should be placed in the aqueous acidic waste container. It contains water,  $B(OH)_3$ , HCl, and ethanol.
  6. Then, record the mp when the sample is completely dry (mp ~136-137 °C). Discard filtrate in the organic waste container.
  7. Record the IR spectrum and compare it to the spectrum of the starting benzoin, noting the absence of the carbonyl-stretching band. Your product should be completely dry before going on next week to the next reaction.

#### Part B:

1. Dissolve 1 g (4.67 mmol) of the diol in 15 mL of anhydrous reagent grade acetone in a 50 mL round-bottomed flask fitted with a stir bar. Add 2 mL of acetone dimethyl acetal (2,2-dimethoxypropane). Immediately stopper the flask and cool it in an ice bath.
2. Remove the stopper and add 12 drops of concentrated sulfuric acid. (Caution: conc.  $H_2SO_4$  is highly corrosive).
3. Carefully replace the stopper and continue to stir the cooled flask for 20 minutes.

4. Then, transfer the cold reaction mixture to a separatory funnel. Rinse the reaction flask with two 25 mL portions of diethyl ether and add the rinses to the separatory funnel. Carefully add 30 mL of  $\text{Na}_2\text{CO}_3$  solution to the separatory funnel. Swirl the funnel gently before inserting its stopper. (Caution:  $\text{CO}_2$  evolution may be vigorous. ) Insert the stopper and immediately invert the funnel and vent it by opening the stopcock. With the stopcock open, swirl the funnel again to complete the neutralization of the acid and evolution of  $\text{CO}_2$ . Close the stopcock. Position the funnel upright and remove the stopper.
5. Drain off the lower aqueous layer and extract the remaining organic layer with two-30 mL portions of water and then one 30 mL portion of saturated sodium chloride solution.
6. Transfer the organic layer to an Erlenmeyer flask and dry the solution over  $\text{Na}_2\text{SO}_4$ . DECANT the dry solution into a round-bottomed flask and evaporate the solvent on a rotary evaporator (rotovap). Your TA will explain to you how the rotovap works.
7. The aqueous layer should be placed in the aqueous basic waste container.
8. Scrape your crystalline product out of the round-bottomed flask. Dry. If your crystals are sticky, your TA will assist you by washing with ice-cold pentane. Record the mp and IR of your product. Be sure to calculate a yield for steps 1 and 2 and an overall yield.

Results and Observations: I noticed that it was very hard to get the solid substance out of the Erlenmeyer flask without rinsing several times. I do not believe this gave me abnormal results or is a cause for skewed yield. In part

A I got a melting point of 122°C and collected 1.427g of product. In part B I got 4.857g of product. My melting point was 49°C and there was no IR taken because the machine was down.

Product Data: The product in part A is officially called erythro-1, 2-diphenyl-1, 2-ethanediol. It has a melting point of ~136°C. It has the chemical formula of C<sub>14</sub>H<sub>14</sub>O<sub>2</sub> and molecular mass of 214.64 g/mol. The product in part B is officially called meso-2, 2-dimethyl-4, 5-diphenyl-1, 3-dioxolane. The melting point for this is ~57°C and has a molecular weight of 254.329 g/mol. Its chemical formula is C<sub>17</sub>H<sub>18</sub>O<sub>2</sub>.

Discussion of Results: My yield was decent but because of the lower than normal melting points I've determined that my product is not as pure as I'd like it to be. Also, I am without an IR spectrum of my product in part B so I cannot analyze my own chemical stereochemistry.

Percent yield and mp: MP part

- A: 122°C MP part
- B: 49°C
- C % yield
- A: % yield
- B: % yield.