

Procedure

Short story (Racemic): Mix 2 compounds in a solvent at low temperature then quench and extract the product. Analyze the product via IR and polarimetry.

Racemic reduction:

1. Put the 1-inch stir bar in the 250 mL beaker and add 1.5 g of sodium borohydride followed by 25 mL of ethanol.
2. Add a solution of 5 g of methyl acetoacetate dissolved in 15 mL of ethanol.
3. Stir the reaction while heating to a gentle boil. Remove from the hotplate when all the ethanol has evaporated.
4. Place the beaker in ice-water bath and slowly add 30 mL of 1 M HCl to quench the reaction. **Bubbling may occur so be sure to add the acid dropwise initially.** Add 30 mL of dichloromethane and stir until dissolved.
5. Once all of the solid has dissolved and the bubbling has ceased, pour the solution into the separatory funnel and drain the organic layer (bottom) into a clean Erlenmeyer flask and set it aside.
6. Drain the aqueous (top) layer into a beaker. This will be waste.
7. To the organic layer add about 4 spatulas of anhydrous sodium sulfate and swirl for about 15 seconds.
8. Remove the sodium sulfate by filtration and collect the dichloromethane into a beaker. Set up the air tube to blow over the solution to evaporate the dichloromethane.
9. Weigh the final product to calculate a percent yield, obtain an IR spectrum, an H-NMR spectrum, and observe the optical rotation.