

CHM 3343

Organic Chemistry II

Isolation, Characterization, Oxidation, and Reduction of Cinnamaldehyde

TUD Department of Chemistry
Spring 2018
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Isolation

Insert steam distillation narrative here

Reduction

Place 100 mg of t-cinnamaldehyde in a 10 mL Erlenmeyer flask, add 0.5 mL methanol and stir to dissolve. Cautiously add, in small portions, 100 mg of NaBH₄. After all of the NaBH₄ is added, gently boil the solution (very low hot plate setting) for 2 minutes. Add methanol drop-wise to replace any lost by evaporation. Cool the flask and slowly add 4 mL of cold water. Transfer the solution to a centrifuge tube and extract several times with MTBE. (Product will be in the ether phase). Dry the ether with several spatula fulls of anhydrous Na₂SO₄ and then transfer the ether to a clean, dry 25 mL suction flask. Wash the remaining drying agent with a couple of mL of MTBE and add the wash to the previous ether. Evaporate the MTBE under vacuum. Weigh, calculate the yield, obtain an IR spectrum, and run a GC/MS analysis on the product.

Oxidation

Dissolve 500 mg of t-cinnamaldehyde in 2 mL of glacial acetic acid in a 50 mL Erlenmeyer flask. Add 10 mL of Clorox (contains about 5% NaOCl) drop-wise over a period of several minutes. Swirl occasionally and keep the

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reaction near room temperature by cooling in an ice bath if necessary. Allow the mixture to stand at room temperature for one hour while continuing to swirl occasionally. The solution should remain faintly yellow. Add aqueous NaHSO_3 drop-wise until the yellow color is discharged and pour the solution over ice-cold brine in a 150 mL beaker. Vacuum filter the solid and wash it with saturated aqueous NaHCO_3 until the washings are neutral. Continue suction and press the solid dry on the funnel. Transfer the product to a 50 mL Erlenmeyer flask and dissolve in about 20 mL of methyl t-butyl ether (MTBE). Dry the solution with anhydrous CaCl_2 and decant the liquid into a suction flask. Rinse the drying agent with a few mL of MTBE and add the rinse to the suction flask. Remove the MTBE under vacuum. Collect the solid, determine the yield, and characterize by melting point, FTIR, and GC-MS.