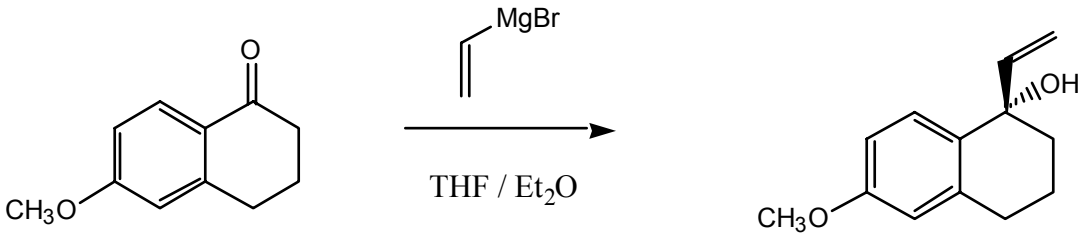
STEP 1:

1-HYDROXY-1-VINYL-6-METHOXY TETRALIN



1. Procedure

Equip a 500-mL, three-necked round-bottomed flask with a reflux condenser, a 250-mL dropping addition funnel, a magnetic stirbar, and a vacuum adapter (note 1, Figure 1). Cool the apparatus by purging the system with a stream of dry nitrogen. After the apparatus has cooled to room temperature, remove the vacuum adapter, replace it with a septum, and add a drying tube filled with calcium chloride to the top of the condenser. Charge the dropping addition funnel with 6-methoxy-1-tetralone (7.03 g, 40.0 mmol), dry tetrahydrofuran (25 mL), and dry ethyl ether (25 mL). Transfer a solution of vinyl magnesium bromide in tetrahydrofuran (1.0 M, 100 mL, 100 mmol, 2.5 equiv) to the round-bottomed flask via cannula with a forced nitrogen flow (note 2). Add the tetralone solution dropwise over 10 minutes to the vinyl magnesium bromide solution (note 3). After the addition is complete, rinse the dropping addition funnel with a small amount of diethyl ether in order to effect quantitative transfer of the starting material. Remove the dropping addition funnel, replace it with a septum, affix a heating mantle, and gently reflux the mixture for 30 min (note 4). Monitor the reaction by thin layer chromatography.

Cool the reaction mixture in an ice-water bath. Check the reaction for disappearance of the starting material using thin layer chromatography. Reinstall the

dropping addition funnel. **SLOWLY** add a solution of saturated aqueous ammonium chloride (35 mL) to the reaction mixture (notes 3,5). Add additional water (100 mL) to aid in the dissolution of the magnesium salts. Transfer the resulting biphasic mixture to a 1000-mL separatory funnel and allow the layers to separate. Remove the organic phase and extract the aqueous phase with ethyl ether (2 x 250 mL). Combine the organic phases and wash them with water (150 mL). Dry the organic phase with magnesium sulfate and filter it through filter paper. Evaporate the solvent on a rotary evaporator. The product, 1-hydroxy-1-vinyl-6-methoxy tetralin, is somewhat unstable, particularly toward acids, and will be used without purification in the next step. Be sure to store your product in an appropriate container in the freezer until you a re ready to perform step 2. A small portion of the product should be purified by column chromatography (85:15 hexane-ethyl acetate) to be used for spectroscopic analysis.

2. Notes

1. All glassware for this reaction MUST be oven dried for at least three (3) hours, and preferably overnight, before beginning this experiment.

2. All Grignard reagents react violently with water. Extreme care must be exercised in handling these compounds.

3. The addition must be performed slowly enough so that the reaction does not become hot, although slight warming is to be expected. It is a good idea to keep an ice-water bath on hand should the reaction mixture exotherm excessively.

4. **Never plug a heating mantle directly into a wall socket.** It must be connected to a Variac to regulate the voltage. Never heat an empty heating mantle.

5. This addition should be performed very slowly, especially at first. Do not allow the mixture to boil.

3. Characterization and Report

Determine the crude yield of the product.

Characterize the starting material and purified product by IR and ^{1}H and ^{13}C NMR. Crude product should be characterized by ^{1}H NMR and IR.

Assess the purity of the crude product based on the spectral data.

Tabulate and assign the spectral data.

List the TLC conditions used and the R_f values of starting material and product.

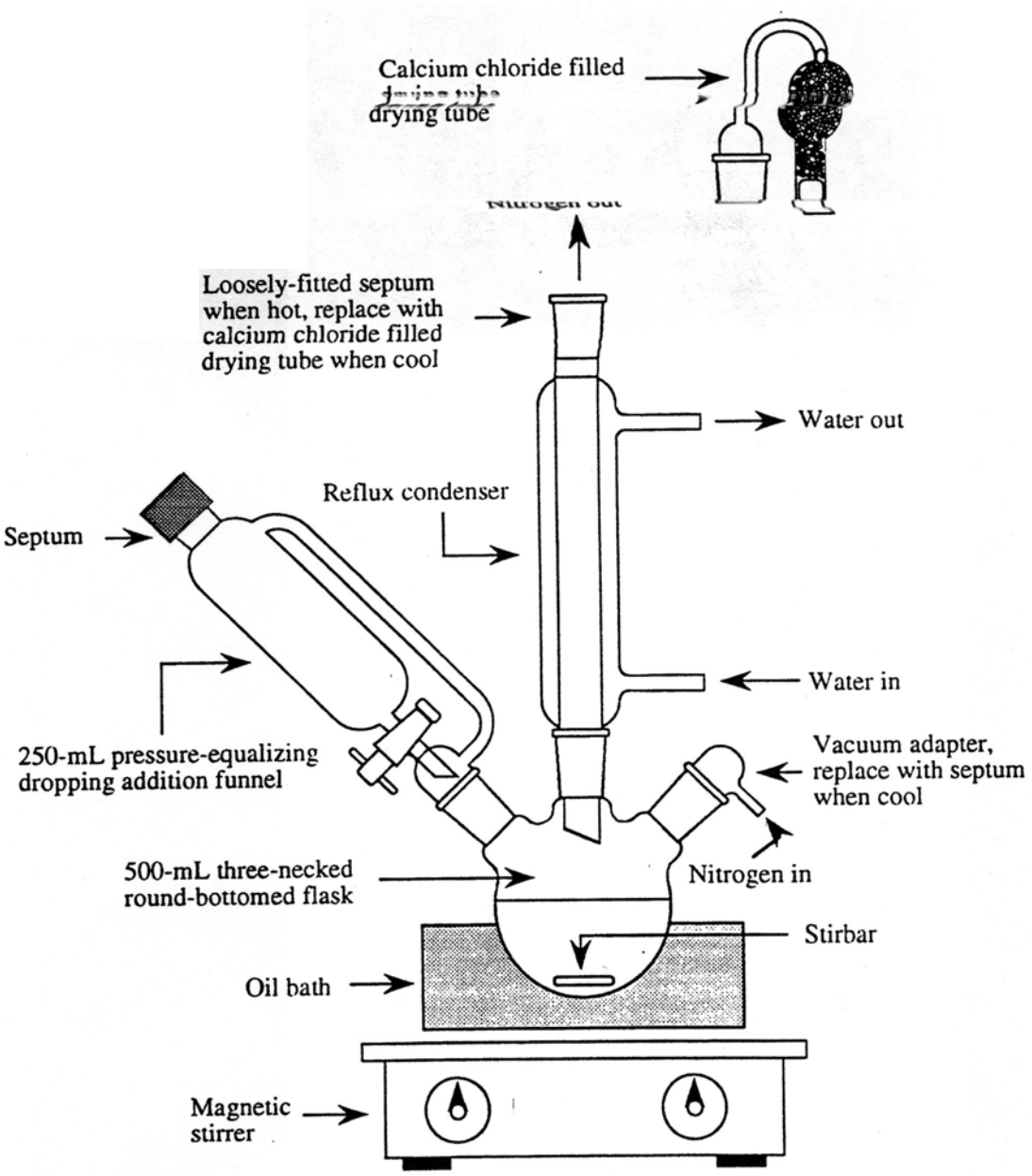

Calcium chloride filled drying tube
Loosely-fitted septum when hot, replace with calcium chloride filled drying tube when cool
Water out
Reflux condenser
Septum
Water in
250-mL pressure-equalizing dropping addition funnel
Vacuum adapter, replace with septum when cool
500-mL three-necked round-bottomed flask
Nitrogen in
Stirbar
Oil bath
Magnetic stirrer

Figure 1