EXPERIMENT 6
SODIUM BOROHYDRIDE REDUCTION OF 4-tert-BUTYLCYCLOHEXANONE TO 4-tert-BUTYLCYCLOHEXANOL:

REACTION: Reduction of an Alcohol
TECHNIQUES: TLC, Extraction

In this experiment, we will explore the reduction of a ketone to a secondary alcohol using a common mild reductant – sodium borohydride. We will use thin layer chromatographic (TLC) technique to monitor the progress of the reduction reaction.

READING ASSIGNMENT:

Background handout
This handout for procedure
Supplementary information in Janice Gorzynski Smith (2^{nd} ed), Chapter 12

PRE-LAB ASSIGNMENT:

In your lab notebook make a table of the chemicals you will use in this experiment. Remember to use an appropriate scale for the reaction based on your yield from last week!
NOTE: you will have to set aside at least 50 mg of your product for IR and NMR experiments for the next two weeks! You need 50 mg of tert-butylcyclohexanone for this experiment, but if you don't have enough left over your TA will provide you with extra.
In your lab notebook rewrite the procedure for the reduction reaction USING YOUR OWN WORDS.
Draw a flow chart for the work up procedure. For every extraction or wash label bottom/top and aqueous/organic layer.

EXPERIMENTAL NOTES:

IMPORTANT SAFETY INFORMATION
4-tert-butylcyclohexanol and 4-tert-butylcyclohexanone are irritants! Wear gloves when working with either of them!
Sodium borohydride is harmful and all contact with skin or eyes should be avoided!
Diethyl ether is extremely volatile and flammable! Use it in the hood! Be certain that a hot plate doesn't ignite the vapors, causing a fire.

Dissolve 150 mg of 4-tert-butylcyclohexanone in 3 mL of ethanol in 25 mL Erlenmeyer flask equipped with a magnetic stir bar on a magnetic stirrer. **Slowly** add the appropriate amount of sodium borohydride (3 molar equivalents) to the flask. After 15 minutes, check the reaction progress using TLC (see instructions for monitoring the reaction progress as described in Exp 5). Once you have determined the reaction is complete, slowly quench the reaction by adding 2 mL of deionized H_2O drop-wise, followed by drop-wise addition of 3M HCl until no more H_2 gas is released.

Cool the reaction mixture to room temperature and add 5 mL of Et_2O. Transfer the solution into a separatory funnel (If there is solid in the bottom of the Erlenmeyer carefully decant the solution into the sep. funnel leaving any solid behind. Rinse out the remaining solids with 2 mL of diethyl ether and add the rinsings to the sep. funnel, again leaving the solid behind in the Erlenmeyer.) Extract the reaction mixture and separate the layers and set the Et_2O layer aside. Take the aqueous layer and extract with 5 mL of Et_2O again. Add the ether layer from the second extraction with the one from the first extraction and pour it back into the sep funnel. Wash the combined organic layers with 5 mL of deionized water. Dry the organic layer with anhydrous sodium sulfate for a few minutes, and transfer it to a tared Erlenmeyer flask. Rinse the anhydrous sodium sulfate with 2 mL of ether and add the rinsed ethers to the ether extract. Evaporate the ether in the fume hood using gentle heating on a hot plate or by blowing air. Cool the residue until it becomes a solid, weigh the pure product, obtain a melting range, and turn it in to your TA in a properly labeled flask.

LAB WRITE-UP:

Results:

- Write a paragraph summarizing the results you obtained week 1 and 2 of this project. It should include appearances, masses, melting points, % yield for both reactions, TLC Rf values for all compounds, etc.