ATISHAY
JAIN

EXPERIMENT 7: SODIUM BOROHYDRIDE REDUCTION OF BENZOIN

PRE-LAB REPORT

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PURPOSE :

The purpose of this experiment is to perform reduction of aldehyde/ ketone to form alcohol using metal hydride. For this experiment we will be using sodium borohydride (NaBH4) for reduction of BENZOIN.

REAGENT TABLE:

COMPOUND	MW (g/mol)	AMOUNT NEEDED	MILLI-MOLES	BP	MP	DENSITY (g/ml)	HAZARDS
BENZOIN		0.5GM				-	
95% ETHANOL	-	4ML	-	-	-		
SODIUM BOROHYDRIDE		0.1GM	-			-	
ACETONE	-	JUST ENOUGH TO DISSOLVE	-		-	-	

HCl	-	0.3ml	-	-	-	-
ETHYL ACETATE	-	UPTO BASELINE (1CM)	-	-	-	-

EXPERIMENTAL PROCEDURE

Part I: Procedure for the Reduction of Benzoin

- Add benzoin (0.5 g) and ethanol (4 mL) to a 25-mL Erlenmeyer flask and swirl gently at room temperature for several minutes.
- Add sodium borohydride (0.1 g) using a micro-spatula in several small portions over 5 minutes.
- Gently swirl the mixture at room temperature for 20 additional minutes.
- Cool the mixture in an ice-water bath, then add water (5 mL) and 6M HCl (0.3 mL).
- Quenching: After 15 minutes, add an additional portion of water (2.5 mL).
- Collect the product via vacuum
- Allow the product to dry on the filter for 15 minutes, and then determine the crude yield and mp. Reserve a small portion (1-2 mg) of the crude material for TLC analysis.
- Recrystallize the crude solid using acetone.
- Determine the mp and yield (% and mass) of purified 1,2-diphenylethane-1,2-diol. Obtain an IR of the product with assistance from our TA.

Part II: (TLC)

- You should dissolve a small amount of benzoin (the starting material), your recrystallized product and your reserved crude product (1-2 mg) in ethyl acetate in each of three vials.
- Spot 2 TLC plates, as shown below.
- Run the TLC plates in 9:1 CH2Cl2: ethanol
    a. Add your eluant to a TLC developing chamber and carefully place the TLC plate in the developing chamber.
    b. Remove the TLC plate, when solvent reaches about 1cm from the top, mark the solvent front and allow it to dry in your fume hood.
    c. Once the plate is dry, visualize the spots under UV light and circle them with a pencil.