Lab 5

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Purpose

The purpose of this experiment is to prepare diphenylmethanol from benzophenone by sodium borohydride reduction. The reagents we will be using are benzophenone, sodium borohydride, 2-propanol, sodium hydroxide, and dichloromethane. The apparatus and techniques we will be employing are a condenser, distillation, melting point, and thin layer chromatography.

Drawing of Structure

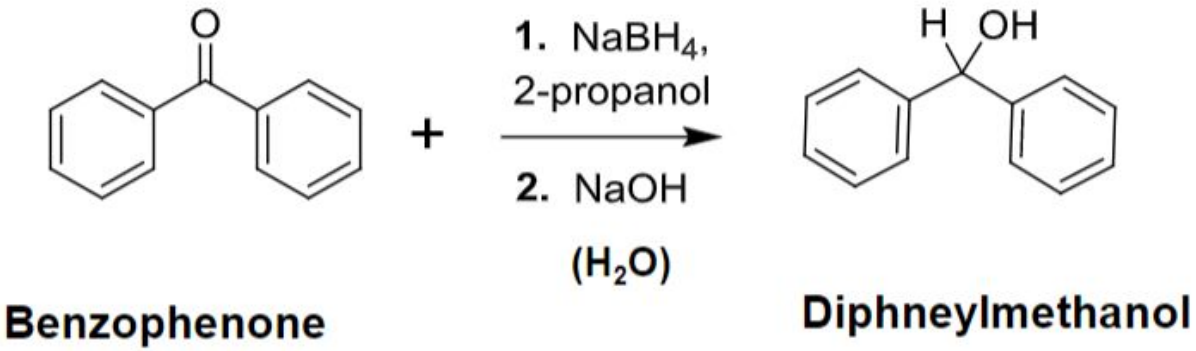


Table of Reagents

Name	MW	Density	Amount	Moles	MSDS	Role
Benzophenone	182.22 g/mol	1.11 g/cm^3	.55 g	.003 moles	May cause cancer	Reactant
Sodium Borohydride	37.83 g/mol	1.07 g/cm^3	.06 g	.0015 moles	Very hazardous	Reactant
2-Propanol	60.1 g/mol	.785 g/cm^3	3 mL	.039 moles	Highly flammable	Solvent
Sodium Hydroxide	39.997 g/mol	2.13 g/cm^3	3 mL	.1597 moles	Corrosive, may cause skin burn and eye damage	Reactant/ Workup
Dichloromethane	84.93 g/mol	1.33 g/cm^3	3 x 5 mL	.078 moles	Corrosive, eye damage, respiratory irritation, organ toxicity, cancer	Solvent/ Workup

Calculations

Limiting Reagent: Benzophenone

$$0.55\text{g benzophenone} * \frac{1\text{ mol benzophenone}}{182.217\text{g}} * \frac{1\text{ mol diphenylmethanol}}{1\text{ mol benzophenone}} * \frac{184.24\text{ g diphenylmethanol}}{1\text{ mol}} = 0.556\text{ g diphenylmethanol}$$

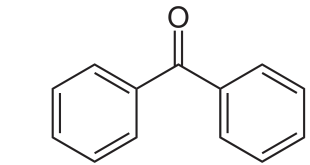
Theoretical yield= 0.56 grams

Procedure

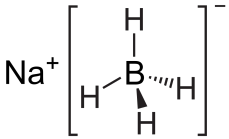
1. In a 25 mL round bottomed flask place 0.55 g (0.003 mole) of diphenyl ketone (benzophenone).
2. Add a slurry of 0.06 g (0.0015 mole) of sodium borohydride in 3 mL of 2-propanol.
3. Add boiling chips and reflux the mixture for 30 min on a heating mantle.
4. Allow solution to cool to room temperature.
5. Decompose borate ester complex: Add 3 mL of 10% aqueous sodium hydroxide solution and swirl the reaction mixture vigorously until the precipitate has dissolved completely.
6. Break up any resistant lumps carefully with the aid of water. Add 5 ml of water and 5 mL of dichloromethane.
7. Extract the diphenylmethanol by shaking it with two successive 5 mL portions of dichloromethane.
8. Combine the extracts, transfer them to a distillation apparatus, and carefully distill off the dichloromethane (use heating mantle).
9. On cooling and standing the residue will crystallize to give product.
10. Measure the weight, melting point and TLC to confirm the product.
11. Only two spots on TLC: Benzophenone and your product from step 9. (use 20% Ethylacetate and 80% hexane for TLC development)

Stepwise Procedure

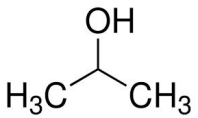
1. In a 25 mL round bottomed flask place 0.55 g (0.003 mole) of diphenyl ketone (benzophenone).
2. Add a slurry of 0.06 g (0.0015 mole) of sodium borohydride in 3 mL of 2-propanol.



0.55g of benzophenone



0.06g of sodium borohydride



3 mL of 2-propanol

Combine

Stepwise Procedure (cont.)

3. Add boiling chips and reflux the mixture for 30 min on a heating mantle.
4. Allow solution to cool to room temperature.

Mixture from step 2

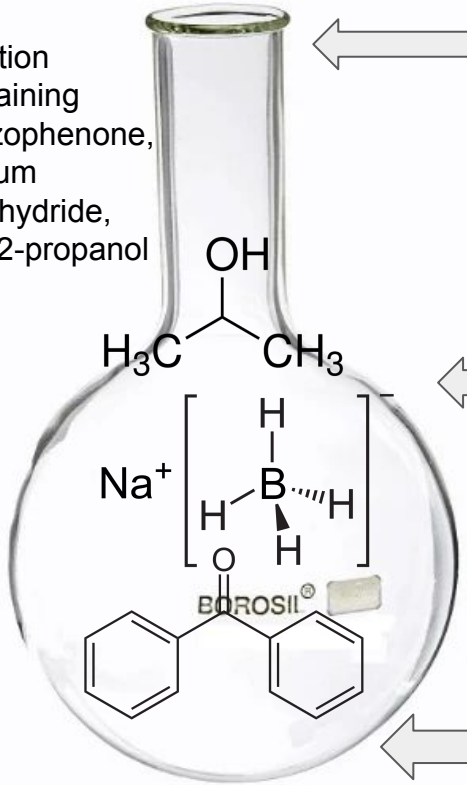
Reflux 30 min

Cool to room temperature

Stepwise Procedure (cont.)

5. Decompose borate ester complex: Add 3 mL of 10% aqueous sodium hydroxide solution and swirl the reaction mixture vigorously until the precipitate has dissolved completely.
6. Break up any resistant lumps carefully with the aid of water. Add 5 ml of water and 5 mL of dichloromethane.

Solution containing benzophenone, sodium borohydride, and 2-propanol



Swirl until all precipitate dissolves

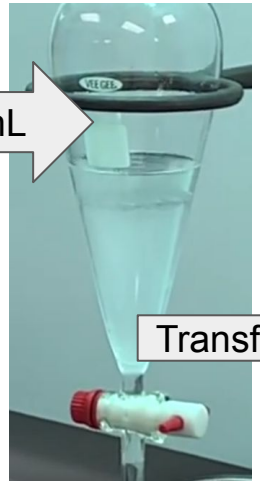
5 mL of water

5 mL of Dichloromethane

Stepwise Procedure (cont.)

7. Extract the diphenylmethanol by shaking it with two successive 5 mL portions of dichloromethane.
8. Combine the extracts, transfer them to a distillation apparatus, and carefully distill off the dichloromethane (use heating mantle).

Extract 2x5 mL

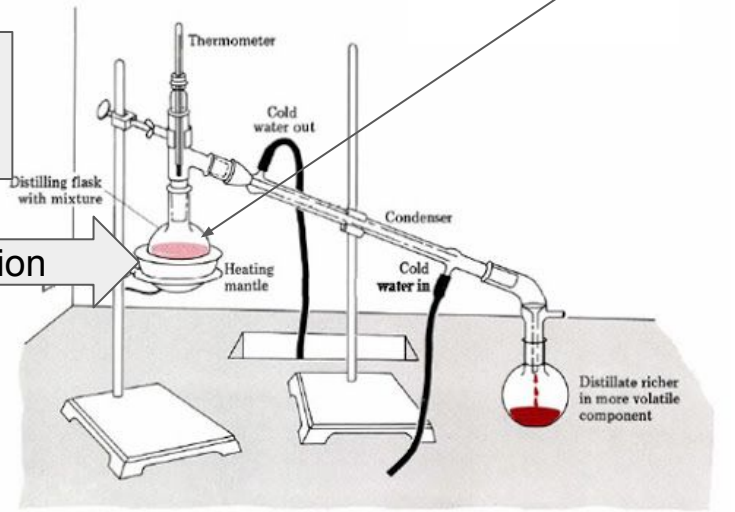


Add reaction mixture

Bottom layer is DCM + product

Transfer this layer to distillation

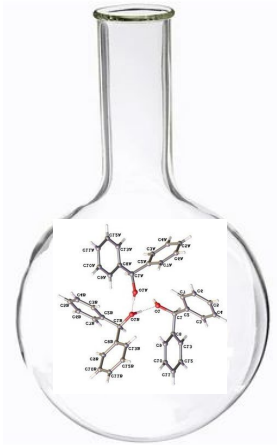
Product will remain in distilling flask



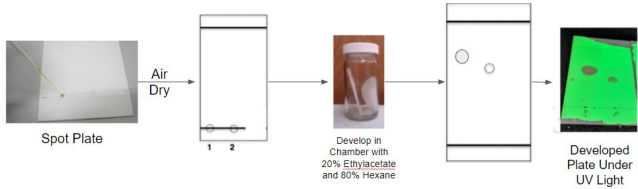
Stepwise Procedure (cont.)

9. On cooling and standing the residue will crystallize to give product.
10. Measure the weight, melting point and TLC to confirm the product.

Allow the reaction mixture to cool



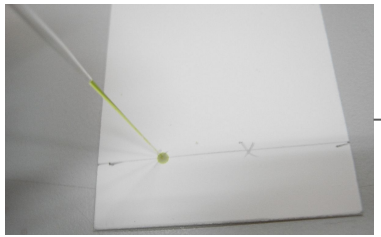
Colorless crystal will form



Measure melting point, weight and TLC to confirm product formation

Stepwise Procedure (cont.)

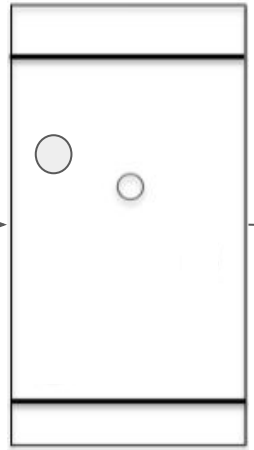
11. Only two spots on TLC: Benzophenone and your product from step 9. (use 20% Ethylacetate and 80% hexane for TLC development)

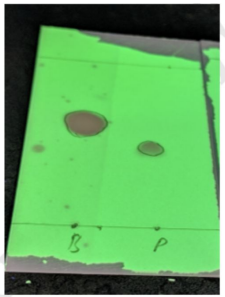


Spot Plate

Air
Dry

Develop in
Chamber with
20% Ethylacetate
and 80% Hexane





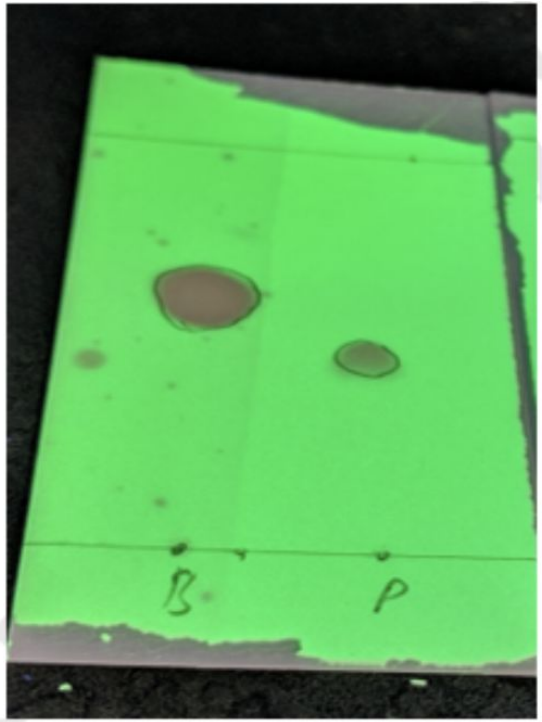
Developed
Plate Under
UV Light

Results

Crude Product: .4 g

Recrystallized Product: .3 g

Solution	Rf Value
Benzophenone	.574
Final Product	.426



TLC: B = Benzophenone
P = Product

Post Lab Questions

1.) If we use 0.002 mole of diphenyl ketone and 0.001 mole of sodium borohydride then which compound will be the limiting reagent?

Diphenyl ketone will be the limiting reagent because each mole of sodium borohydride contributes four hydride ions.

2.) In few cases reaction didn't go to completion. Which of the following could be the possible reason or reasons for incompletion of the reaction?

(a) Sodium borohydride was not enough (may be some of sodium borohydride decomposed).
(b) Refluxing was not done for entire 30 minutes.
(c) Solution was not heated to reflux
(d) All of the above

Conclusion

In this lab we accomplished preparing diphenylmethanol from benzophenone by sodium borohydride reduction. In this lab we practiced the technique of refluxing a mixture and carrying out thin layer chromatography. The R_f values from the TLC shows that the reaction was carried out completely since only one distinct appearance on the paper. As we've learned in previous labs, this proves there are no impurities. The R_f value of benzophenone was 0.574 and the R_f value of diphenylmethanol was 0.426**.** Benzophenone had a higher R_f value and travelled farther because the solvent is nonpolar, and benzophenone is less polar than diphenylmethanol. The practical application of this, as we saw with the thin layer chromatography, is to confirm a reaction has been carried out.