Lab-11 Benzopinacol Synthesis

December 11, 2017

1 Introduction

This experiment uses a photochemical reaction to synthesize benzopinacol from benzophenone.

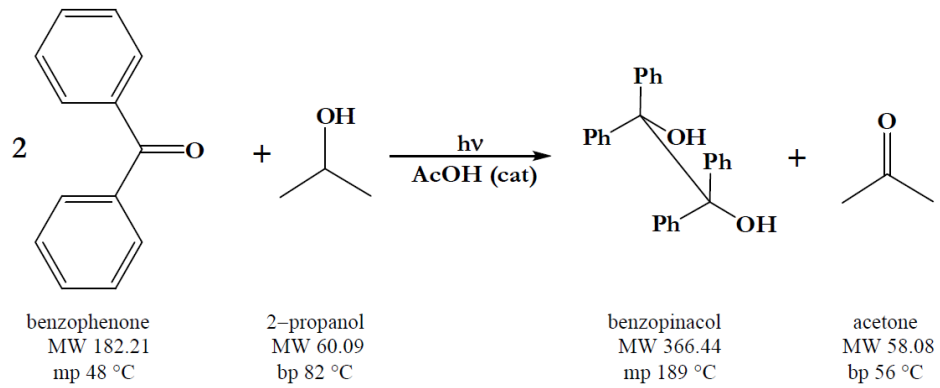

Figure 1: Summary of the photoreduction benzophenone to benzopinacol

2 Materials

6.00g benzophenone	1 ring stand & clamp	parafilm	thermometer
glacial acetic acid	glass stir rod	watch glass	capillary tube
35mL isopropyl alcohol	cork	filter funnel	
1 Large test tube	hot water bath	250ml Erlenmeyer flask	ice bath

3 Procedure

6.00g benzophenone, 35mL isopropyl alcohol, and 1 drop glacial acetic acid from a transfer pipette was added to a large test tube. The test tube was swirled and warmed to 45°C in a warm water bath. The test tube was corked and sealed with parafilm. The test tube was clamped to a ring stand and left in the sunlight. The following lab period the test tube was placed in an ice bath, vacuum filtrated while washing with a small quantity of ice-cold isopropyl alcohol, and the collected crystals were placed on a pre-weighed watch glass and left to dry. Once dried the product was weighed, the melting point was determined, and an IR spectrum of the product was obtained.

4 Calculations/Results

Experimental Data

Mass initial sample:

6.00g benzophenone

Mass watch glass:

Initial (empty) weight = 53.2065g
Final weight (with bezopinacol) = 58.5349g
Actual yield bezopinacol = $M_f - M_i = 5.3284g$

Overall reaction:

$$2C_{13}H_{10}O + 2C_3H_8O \xrightleftharpoons[AcOH(cat)]{hv} C_{26}H_{22}0_2 + C_3H_60$$

Theoretical yield:

limiting reagent: benzophenone ($C_{13}H_{10}O$)

$$6.00g\ C_{13}H_{10}O * \frac{1\text{mol } C_{13}H_{10}O}{182.21g\ C_{13}H_{10}O} * \frac{1\text{mol } C_{26}H_{22}0_2}{2\text{mol } C_{13}H_{10}O} * \frac{366.45g\text{g } C_{26}H_{22}0_2}{1\text{mol } C_{26}H_{22}0_2} = 6.03\text{g } C_{26}H_{22}0_2$$

Percent yield:

$$\text{Percent yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} * 100 = 88.4\%$$

Melting point

Initial melt= 186.0 $°C$
Final melt= 186.8 $°C$

$$\text{Midrange=} \frac{B.P._{initial} + B.P._{final}}{2} = 186.4\ °C$$

IR Spectrum

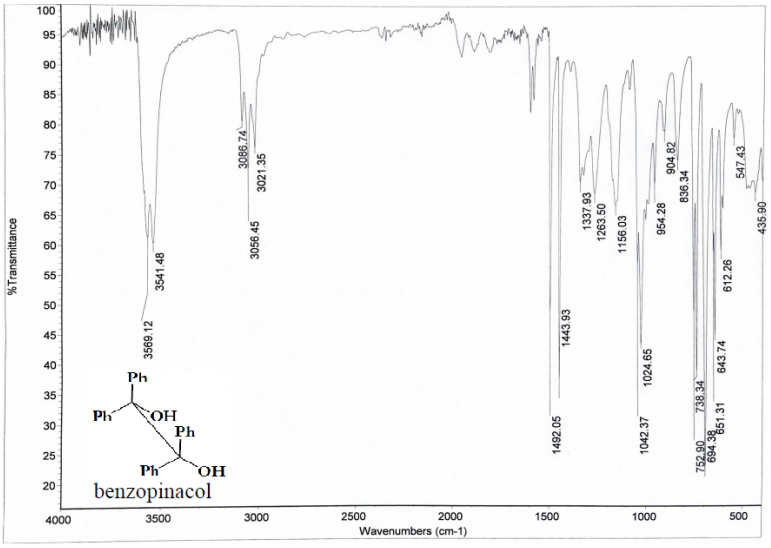

Figure 2: IR spectrum of 2-methoxynaphthalene (nerolin), taken in lab.

Figure 3 displays the IR spectra of the benzopinacol product taken in lab. The molecule exhibited strong (C-O) stretches at $1024.65cm^{-1}$ and $1042.37cm^{-1}$. There were also strong (C-H) oop bends at $694.38cm^{-1}$ and $738.34cm^{-1}$. There were medium-weak aromatic (C=C) stretches between $1400 - 1600cm^{-1}$ most evident at $1443.93cm^{-1}$,$1492.05cm^{-1}$. There were also medium aromatic (Sp^2 C-H) stretches at $3021.35cm^{-1}$, $3056.45cm^{-1}$,and $3086.74cm^{-1}$.

5 Conclusion

The photochemical reduction of benzophenone to benzopinacol was a success. Sample purity was verified by IR spectrum. The reaction had gone to completion as evident by the lack of a carbonyl stretch on the IR Spectrum. The Benzopinacol product was characterized by strong (C-O) stretches at $1024.65cm^{-1}$ and $1042.37cm^{-1}$. The molecule also exhibited medium strength stretches at $3541.74cm^{-1}$ and $3569.12cm^{-1}$ associated with phenol(O-H) groups. The melting range of the sample was between 186.0 $°C$-186.8 $°C$ which gave a midrange of 186.4 $°C$ and was slightly lower than the expected $\approx$ 189.0 $°C$ m.p. of pure benzopinacol. Overall The reduction produced 86.4% of the expected yield of the product. The actual yield was $5.3284g$ benzopinacol.

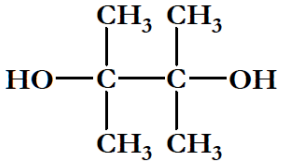

Figure 3: A potential product in the reaction mixture

- There must be two diphenyl ketyl radicals present in the reaction mixture for dimerization to take place producing benzopinacol.
- The air we are breathing is oxygen in its triplet ground state, which is as a stable diradical.