Lab 7 - Synthesis of Benzopinacolone

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1 Introduction

This experiment uses a reflux process whereby a solution is heated to vaporization and then condensed the vapor back into the original solution repetitiously. The purpose of this experiment was to rearrange the benzopinacol molecule by means of dehydration into the ketone benzopinacolone. As the dehydration proceeds, a carbocation forms and a phynol group shifts to form a bond with the carbocation center creating a more stable arrangement.

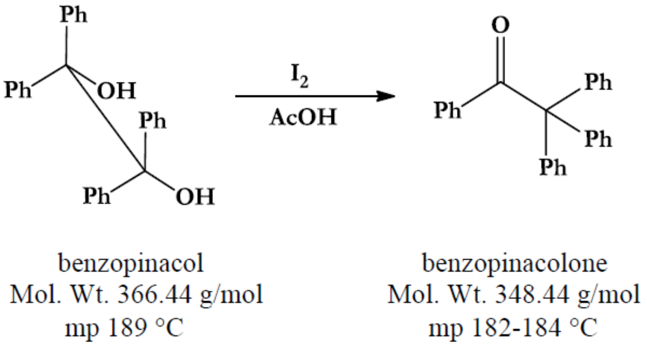


Figure 1: Summary of the conversion of a 1,2-diol, benzopinacol to benzopinacolone by dehydration

2 Materials

2.60g benzopinaco
15mL glacial acetic acid
20mL ethanol (ice cold)
1 grain iodine (I_2)
2 ($50mL$) round bottom flasks
1 heating mantle
1 variac
heat plate
magnetic stir bar
2 rubber tubes
west condensor
50mL graduated cylinder
1 ring stand & clamps
watch glass
keck clip
filter paper
ice bath
cold water source
filter funnel
capillary tube
IR spectrometer
boiling point apparatus

3 Procedure

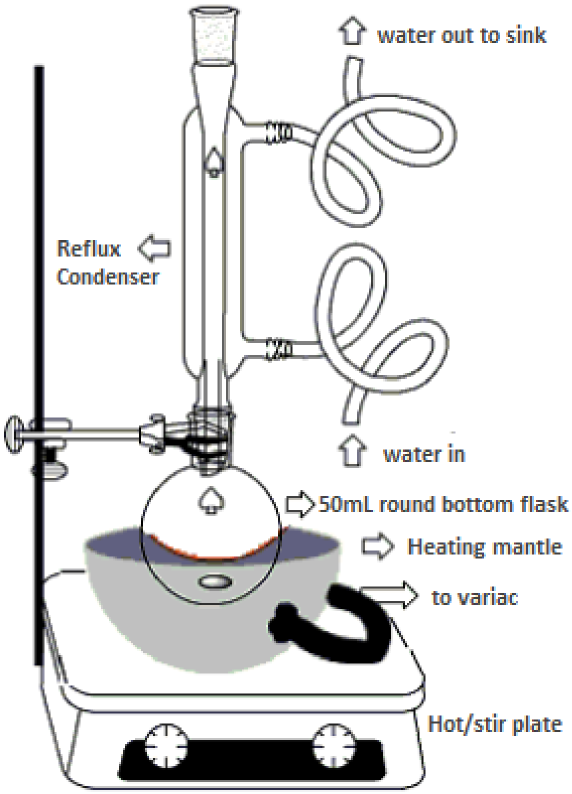


Figure 2: Example of reflux condenser setup

The reflux apparatus was constructed using the materials listed above. 2.60g benzopinacol was added to a 50mL round bottom flask. 15mL glacial acetic acid, 1 grain I_2, and a magnetic stir stick were also added to the 50mL round bottom flask. The flask was then attached to the reflux condenser and secured with a keck clip. The variac was set to 120v, the knob turned to 70%, and water flow was turned on. The knob on the heat plate was turned to begin spinning the magnetic stir stick. The solution was refluxed for 30 minutes. When the reflux was complete the solution was left to cool to room temperature, 15mL ethanol was added, swirled, and the flask was set into an ice bath for 5 minutes to decrease solubility. After the ice bath 5ml ethanol was added, swirled, and the flask was iced again. The solution was then vacuum filtered. The benzopinacolone residue was scraped onto a pre-weighed watch glass and left to dry. Once dried the benzopinaclone was weighed, the melting point was recorded, and the IR spectrum was obtained.

4 Calculations/Results

Experimental Data

Mass benzopinacol:

$2.60000g$

Mass watch glass:

Initial (empty) weight $= 532047g$
Final weight (with benzopinacolone) $= 55.4823g$
Actual yield benzopinacolone $= M_f - M_i = 2.2776g$

Overall reaction:

$$C_{26}H_{22}O_{2(s)} \overset{I_2}{\underset{C_2H_4O_2}{\Longrightarrow}} C_{26}H_{20}O_{(s)} + H_2O_{(l)}$$

Theoretical yield:

$$\text{2.60g benzopinacol} * \frac{\text{1mol benzopinacol}}{\text{366.45g benzopinacol}} * \frac{\text{1mol benzopinacolone}}{\text{1mol benzopinacol}} * \frac{\text{348.44g benzopinacolone}}{\text{1mol benzopinacolone}} = 2.47g$$

Theoretical yield benzopinacolone $= 2.47g$

Percent yield:

$$\text{Percent yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} * 100 = 92\%$$

Boiling Point:

Initial melt= 181.2 $°C$
Final melt= 182.9 $°C$

$$\text{Midrange} = \frac{B.P._{initial} + B.P._{final}}{2} = 182.5\ °C$$

IR Spectrum

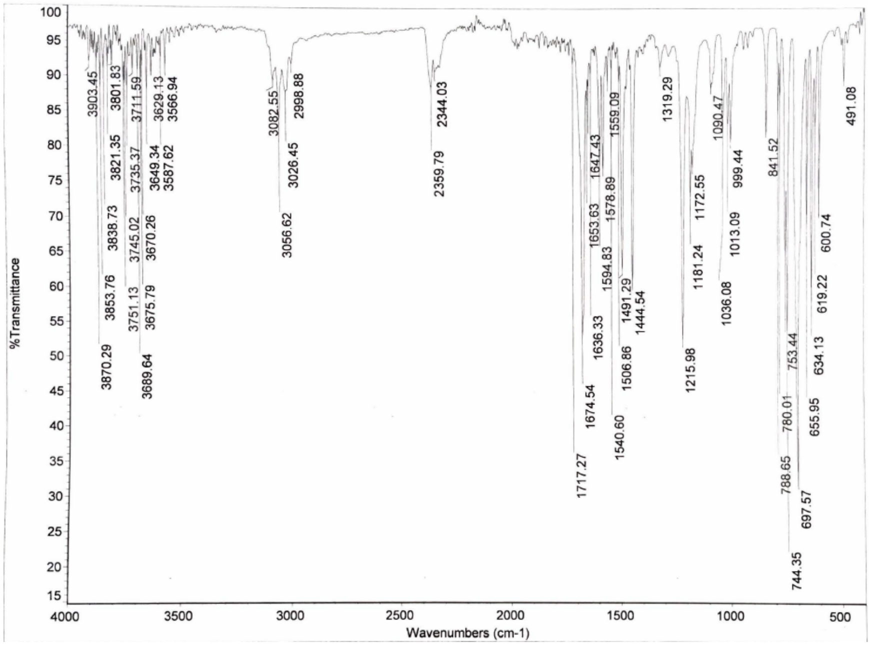


Figure 3: IR spectrum of benzapinacolone, taken in lab.

$Figure$3 displays the IR spectra of the benzapinacolone product that was taken in lab. The molecule is characterized by a strong carbonyl (C=O) stretch at $1674.54cm^{-1}$, an (Sp^2 C-H) stretch at $3056.62cm^{-1}$, 2 (C=C) medium aromatic stretches at both $1444.54cm^1$ and $1491.29cm^1$, and a strong monosubstituted aromatic (Sp^2 C-H) bend at $697.57cm^{-1}$.

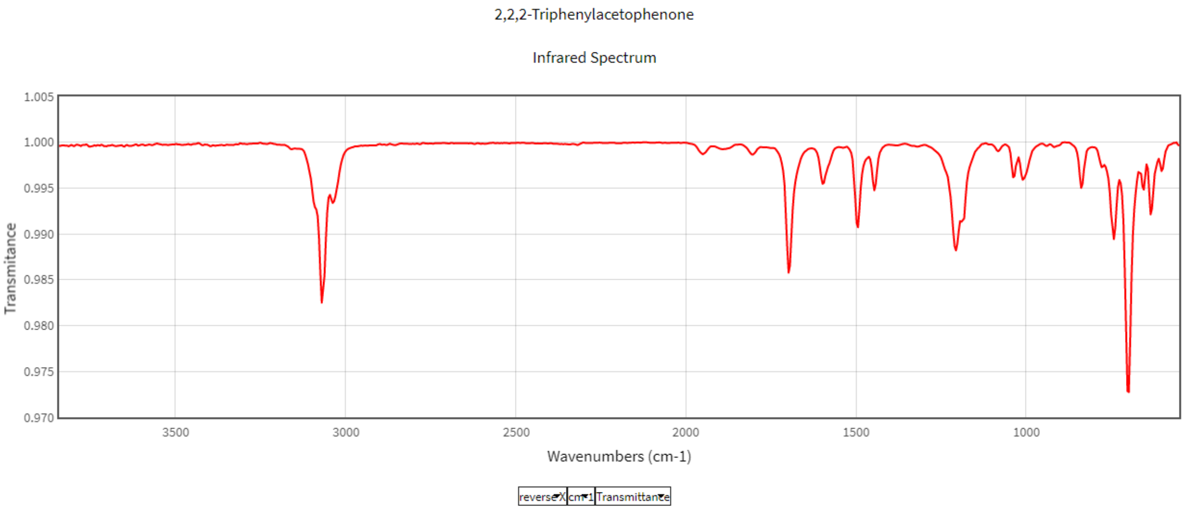


Figure 4: Standard IR spectrum of pure benzopinacolone from *NIST Chemistry WebBook*, *SRD* 69
http://webbook.nist.gov/cgi/cbook.cgi?ID=C466375&Mask=80#IR-Spec

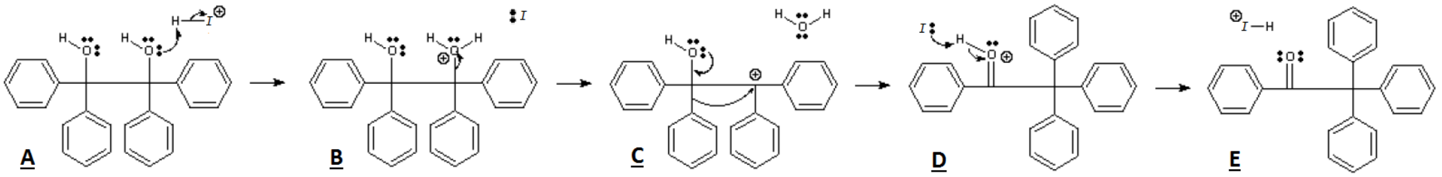
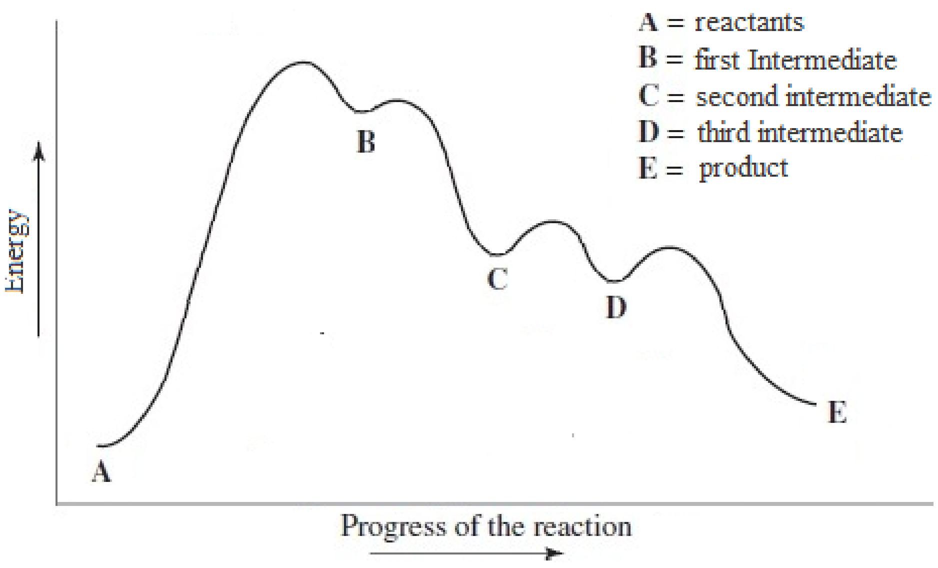


Figure 5: Mechinism of the conversion of benzapinacol to benzapinacolone

5 Conclusion

The sythesis of benzopinacolone from benzopinacol was a success. Sample purity was verified by comparison to the standard IR spectrum of pure benzopinacolone from the literature. The major peaks of both the IR spectrum of the product formed in lab and the Standard IR spectrum of pure benzopinacolone agreed. The comparison can be observed between $figure$ 3 and $figure$ 4 on page 5. The benzopinacolone molecule was characterized by the carbonyl functionality at $1674.54\ cm^{-1}$ and the lack of a hydroxyl group which was present on the origional benzopinacol molecule. The sample purity was further confirmed by the melting point of the product which had a midrange of $182.5\ ^{\circ}C$ and was in agreement with the melting point of $182\ ^{\circ}C$ pure benzopinacolone noted in the litterature. The conversion produced a high yield, 92% of the calculated theoretical yield. The actual yield was $2.2776g$ benzopinacolone.

- Energy diagram for the mechanism in $figure$ 5 following the transition of benzopinacol to benzopinacolone.



- The driving force behind the carbocation rearrangement is to maximize stability of the carbocation by shifting to the carbon it will be most stable. The rearangements can be acomplished by hydride shifts and alkyl shifts, whereby the resulting configuration is the most stable arrangement for the carbocation. In the case of this experiment all octets were ultimately filled.
- Because of the hydroxyl groups on the benzopinacol molecule, it is more polar and will be more soluble than benzopinacolone in ethanol.