

## SYNTHESIS AND ANALYSIS OF BENZOPINACOL FROM BENZOPHENONE BY PHOTOREDUCTION IN GREEN CHEMISTRY.

<sup>1</sup>Lata.C.Potey, <sup>2\*</sup> Dr. Satish B. Kosalge, <sup>3</sup> Rajeshwari S. Sarode

<sup>1</sup>Assistant Professor, Hi-Tech College of Pharmacy, Chandrapur.

<sup>2</sup>Principal, Hi-Tech College of Pharmacy, Chandrapur

<sup>3</sup>Assistant Professor, Hi-Tech College of Pharmacy, Chandrapur.

Date Received:

11<sup>TH</sup> Jan 2014

Date of Accepted:

16<sup>th</sup> Jan 2014

Date Published:

18<sup>th</sup> Jan 2014

### Abstract:

This is free radical reaction and reduction of benzophenone occurred by UV irradiation. Free radical on benzophenone due to photo excitation abstract hydrogen from isopropanol and gives benzopinacol and acetone. Chemically benzophenone is 1,1,2,2, tetraphenyl 1, 2 ethylenediol. Benzopinacol can easily isolate from reaction mixture by suction pump and characterized by using common analytical techniques like UV, IR etc.

**Keywords:** Photochemistry, benzophenone, benzopinacol, free radical.

### Introduction

Photochemistry is the study of the chemical interactions which occur when molecules interact with light. Organic chemists are interested in photochemical preparations of molecules since light can act as a catalyst (or reactant), causing interesting reactions to occur. For example, we have previously discussed the use of ultraviolet light as a catalyst for the formation of radicals in the halogenation of alkanes.

### MATERIAL AND METHODS

#### Apparatus:

The melting points were determined by open capillary method and are uncorrected. Infrared spectra were recorded on FTIR 8400s Shimadzu using KBr. To confirm the completion of reaction TLC were taken by using silica gel G and isopropanol: water(7:3) as mobile phase. UV absorbance was taken by UV-Visible double beam spectrophotometer.

#### Chemicals and Reagents:

Isopropanol, benzophenone, glacial acetic acid, liquid paraffin, dimethyl sulfoxide, methanol is from Loba Chem Pvt. Limited, Mumbai. All solvents were distilled before use and dried whenever required.

#### Mechanism and Reaction

**Step 1:** In this laboratory, we will use ultraviolet light from sunshine to promote a radical reaction. This particular reaction is not catalyzed by light; rather, it is driven by light. In other words, we can think of photons of light as being a needed reactant in this process.

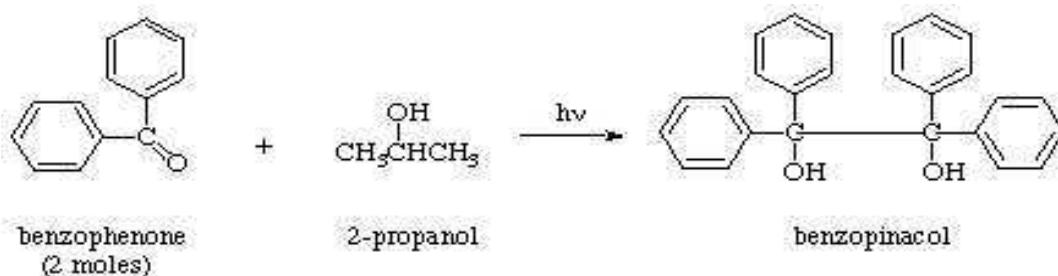
**Step 2:** The light is needed in this reaction to break the  $\pi$  bond between the carbon and oxygen in the carbonyl group of benzophenone forming a diradical. Of the bonds in the two reactants, this

bond is weakest making it most susceptible to the energy provided by the ultraviolet light.

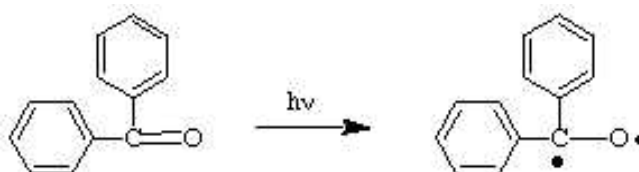
**Step 3:** After the diradical forms it immediately becomes a scavenger, its oxygen atom quickly finding relatively easily removed hydrogen in the 2° position in the plentiful solvent molecules. In this step the diradical becomes a benzhydrol radical, while the solvent molecule becomes a new radical.

**Step 4:** This newly formed radical is now highly reactive, enabling it to offer a hydrogen atom to the oxygen of another benzophenone diradical, thus resulting in the formation of a stable molecule, acetone, as well as another benzhydrol radical. Eventually one benzhydrol radical will collide with another leading to the formation of the final stable product.

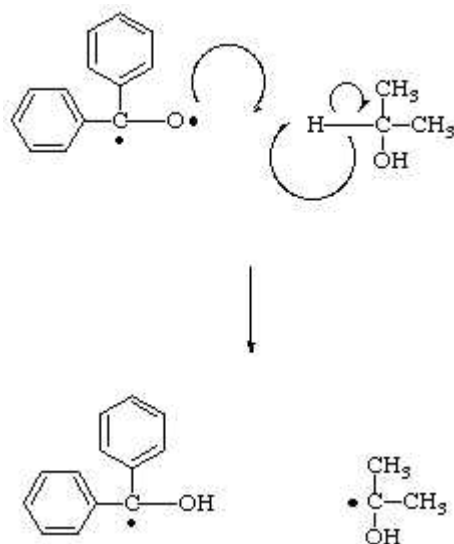
**Step 1:**



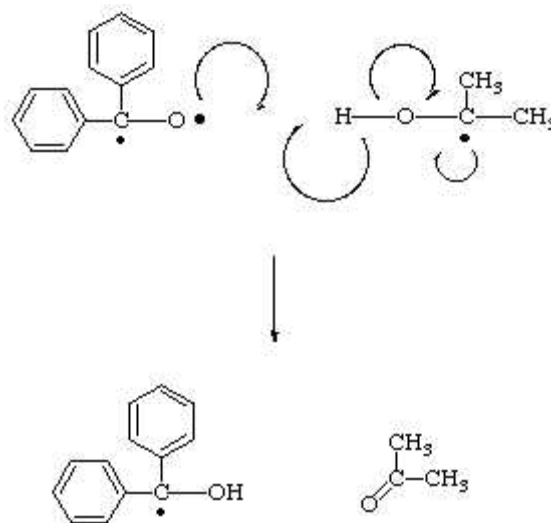
**Step 2:**



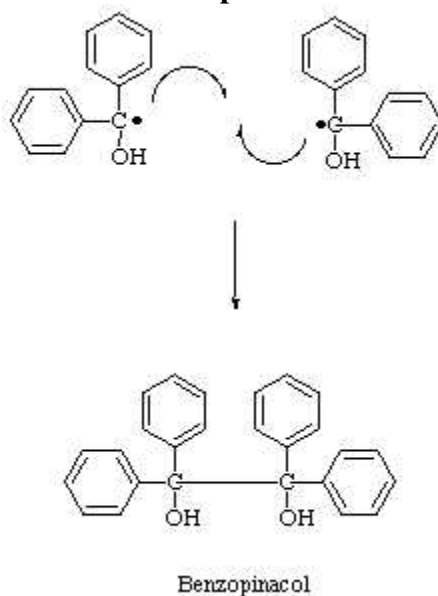
**Step 3:**



**Step 4:**



### Step 5:



### Procedure:-

Place 2 g benzophenone into a test tube and add about 10 ml 2-propanol. Dissolve the solid with gentle warming in a warm water bath. After the solid is dissolved, add 1 drop glacial (100%) acetic acid, then fill the test tube with 2-propanol until it is a little more than half.

Then close the mouth of test tube with rubber cork but precaution should be taken t6hat it should be covered by aluminium foil, otherwise reaction will be occur between reaction mixture and rubber. Keep that test tube in sunlight for two to three days. After precipitation of shiny crystals of benzopinacol along the side wall of test tube scratch it by spatula and dry in oven and report melting point.

### Data Analysis

#### Qualitative Analysis:

**Action of Sodium:** Benzopinacol is having alcohol functional group and it can be confirmed by sodium metal test in which some amount of compound have to dissolve in benzene solution and then in that solution add small piece of sodium metal if effervescence occurs it indicate presence of alcohol.

**Action of Acetyl Chloride:** add 1 ml of acetyl chloride and some amount of bezopinacol, vigorous reaction occurs HCl gas get evolve, bring a glass rod dipped in ammonium hydroxide in contact with HCl gas , white fumes of ammonium chloride are produced, when the gas evolution get stop add 1 ml of water to dissolve HCl gas , separation of an oily layer with ester like odour confirms presence of alcohol.

Table 1: Absorbance of benzophenone and Benzopinacol.

Wavelength/cm <sup>-1</sup>	Absorbance of benzophenone	Absorbance of benzopinacol
310	0.912	0.858
330	0.273	0.210
350	0.117	0.065
360	0.081	0.053

**Quantitative Analysis:** Melting point is 185<sup>0c</sup> which calculate by open tube capillary method. Rf value calculate by taking TLC by using isopropanol: water(7:3) as mobile phase. Practical yield is 3.6 g. For UV detection prepare 0.5 M solution of both benzophenone and Benzopinacol in isopropanol and take absorbance at different wavelength 310, 330, 350, 360.

## RESULT AND DISCUSSION

In this experiment it is aimed to synthesize benzopinacol through photochemical reaction of benzophenone and, benzopinacolone via acid-catalyzed rearrangement of benzopinacol. In this experiment, mixture of benzophenone, isopropyl alcohol and a drop of glacial acetic acid was exposed to sunlight which in turn, undergone photochemical reaction. In this reaction, molecules of benzophenone was brought to n(<sup>\*</sup> triplet state where it possibly abstracted hydrogen from isopropyl alcohol and through subsequent reactions of radicals it formed two diphenyl ketyl radical which dimerized into benzopinacol. Subsequently,

mixture of synthesized benzopinacol, glacial acetic acid and iodine crystal reacted in an acid-catalyzed rearrangement wherein dehydration of benzopinacol resulted into the formation of carbocation. In this state, an aromatic shifted and then, forming delocalized carbocation. Afterward, regenerating the catalyst stabilized the molecule into benzopinacolone. After qualitative and quantitative analysis of the products, it was found out that benzopinacol was successfully synthesized as attested by the IR spectrum which contained OH broad stretch at 3417.86 cm<sup>-1</sup>-3460.30 cm<sup>-1</sup> and overtones at 1800 cm<sup>-1</sup>-1950 cm<sup>-1</sup> region. However, inconsistency was observed in the determined melting point of 185, and percentage practical yield was found to be 40%. UV absorbance occurs at wavelength 310, 330, 350, 360 were found to be 0.858, 0.210, 0.065, and 0.053. Alcohol functional group detected by qualitative analysis by sodium metal test and acetyl chloride test.

**Table 2: FTIR (cm-1) of Benzopinacol.**

Functional group	FTIR ( cm-1)
OH broad stretch	3417.86 cm-1-3460.30 cm-1
overtones	1800 cm-1-1950 cm-1

## REFERENCES

1. Luzchem Research, Inc "Preparation of Benzopinacol via Photoreduction of Benzophenone in 2-Propanol"
2. Durst, T. "CHM 3126: Laboratory Manual," The University of Ottawa, 2001.
3. Turro, N. J. *Modern Molecular Photochemistry*; Benjamin / Cummings Publishing Co.: Menlo Park, 1978.
4. Pitts, J. N. J.; Letsinger, R. L.; Taylor, R. P.; Patterson, J. M.; Recktenwald, G.; Martin, R. B. *J. Am. Chem. Soc.* 1959, 81, 1068-1077.
5. Agrawal, O.P.; *Advanced practical Organic Chemistry*, 22<sup>nd</sup> Edn, Goyal Publishing House, Meerat, 37, 2010.