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Synthesis of Benzil From Benzoin by Oxidation Reaction

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Abstract: *The synthesis of benzil from benzoin is a classic organic chemistry experiment that involves the oxidation of secondary alcohol to a ketone using an oxidizing agent, such as nitric acid. Benzil is a useful compound that is often used as a starting material for the synthesis of other organic compounds, such as dyes and pharmaceuticals. The reaction involves the conversion of benzoin to benzil via a mechanism that involves the formation of hemiketel intermediate. This reaction is typically carried out in a solvent, such as ethanol or methanol, and the product is isolated by recrystallization. This experiment provides students with an opportunity to learn about oxidation reaction, the chemistry of carbonyl compounds, and importance of purification techniques in organic synthesis.*

Keywords: Benzil, benzoin, conc. Nitric acid

I. INTRODUCTION

The synthesis of benzil from benzoin involves the oxidation of benzoin using nitric acid. Benzoin is a colorless solid that is soluble in alcohol and has melting point of 137°C. Benzil, on the other hand, is a yellow solid that is insoluble in water but soluble in alcohol and has melting point of 95-97°C.

The reaction between benzoin and nitric acid involves the formation of an intermediate compound called nirobenzoin. Nitric acid acts as a strong oxidizing agent, which converts the alcohol groups in benzoin to aldehyde groups in nitrobenzoin. Further oxidation of nitrobenzoin using nitric acid results in the formation of benzil

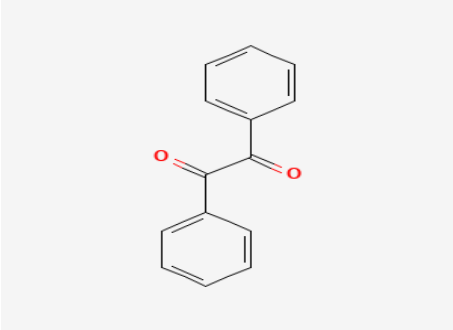

Fig. No.1 Structure of benzil

Parameters	description
Molecular formula	(C6H5CO)2
Molecular weight	210.23g.mol
Colour	Yellow crystalline powder
Odor	Characteristics
Melting point	96°c
Boiling point	345°c
Density	1.23g/cm
Solubility	Soluble in benzene, ethanol Insoluble in water, methanol

Table No.1

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II. EXPERIMENTAL DETAILS

Reagents and Instruments:

Reagents: Benzoin, Conc. Nitric acid, ethanol, sodium hydroxide solution, water

Instruments: Round bottom flask, Reflux condenser, heating mantle, Beaker, dropper, Buchner funnel, filter paper, vacuum pump, spatula, glass rod, thermometer

Synthesis of Benzil:

The process as follows: Dissolve 5g of benzoin in 30ml of concentrated nitric acid. Heat the mixture gently on a water bath for about 15min. until a yellow precipitate of benzil is formed. Cool the mixture in an ice bath to promote crystallization. Filter the precipitate using a Buchner funnel and vacuum filtration. Wash the crystals with distilled water to remove any remaining traces of acid. Wash the crystals with a solution of sodium bicarbonate to neutralize any residual nitric acid. Recrystallize the benzil by dissolving it in hot ethanol and allowing it to cool slowly. Collect the crystals by vacuum filtration, and dry them in a desiccator.

Drug Name	Oxidizing agent	Temperature	Reaction time	Yield %
Benzil	Conc. Nitric acid	200°C	1.5hr	97.7%

Table no. 2 Synthesis of benzil

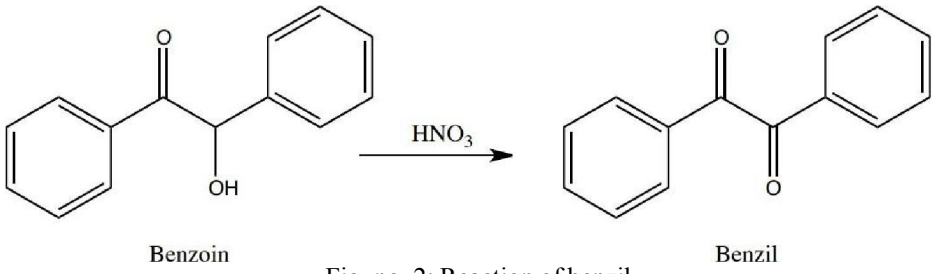
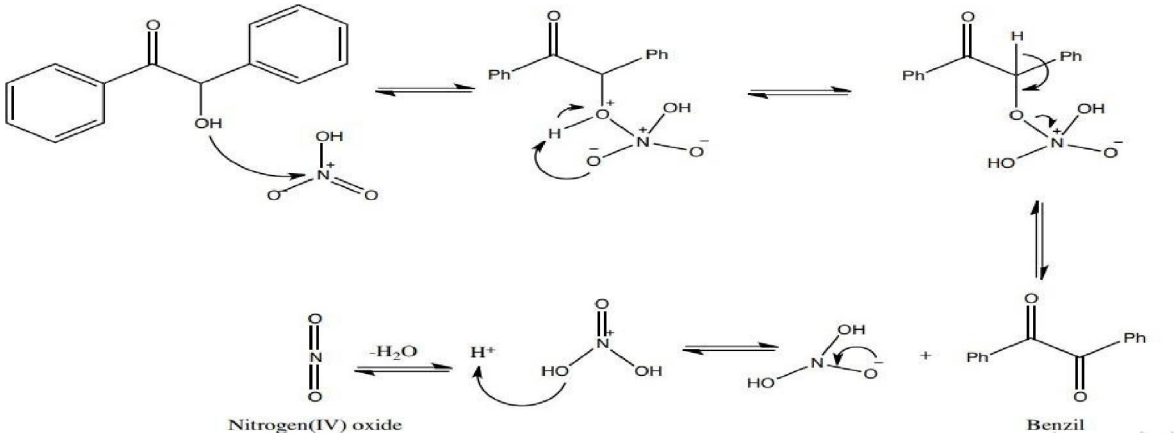


Fig. no. 2: Reaction of benzil

MECHANISM:



IDENTIFICATION TEST:

COLOR:

Benzil is a yellow crystalline solid at a room temperature. When dissolved in certain solvents or subjects to certain reactions, it can exhibit a range of colors including yellow, orange, red and even green. However, its typical color is yellow.

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ODOR:

Benzil is a white crystalline solid with a mild, pleasant odor. However, the exact odor of benzil can vary depending on factors such as the purity of the compound, the concentration of the sample, and the individual's sense of smell.

MELTING POINT:

The melting point of benzil is 94-95°C. It's important to note that melting point of a compound can vary depending on factors such as sample purity and method of determination, so slight variations in reported values may exist in the literature.

BOILING POINT:

The boiling point of benzil, which is diketone compound with the chemical formula C14H10O2, is 344-345°C at standard pressure of atmosphere (101.3kPa).

SOLUBILITY:

The solubility of benzil varies depending on the solvent used. In general, it is sparingly soluble in water, but solution in many organic solvents such as ethanol, acetone, and chloroform.

According to the Merck Index, the solubility of benzil in water at 20°C is approximately 0.2g/100mL. However, the actual solubility of benzil in water may vary depending on factors such as temperature, pressure, and ph.

III. SPECTRAL ANALYSIS

The spectral analysis of the synthesis of benzil from benzoin by nitric acid can be performed using various techniques such as infrared spectroscopy, nuclear magnetic resonance (NMR) spectroscopy and UV spectroscopy.

UV SPECTROSCOPY:

The synthesis of benzil from benzoin by nitric acid can be analyzed using UV spectroscopy. Benzoin, which is a ketone, can be converted to benzil through nitration with nitric acid. This reaction involves the oxidation of the alcohol group to a ketone group, followed by the formation of a cyclic dimeric intermediate, which eventually forms benzil. UV spectroscopy can be used to monitor the reaction progress by measuring the absorbance of the reaction mixture at specific wavelengths. Overall UV spectroscopy is a useful technique for analyzing the synthesis of benzil from benzoin by nitric acid. It can provide information about the reaction progress, the formation of impurities, and the kinetics of the reaction.

INFRARED SPECTROSCOPY:

Infrared spectroscopy can be used to confirm the formation of benzil by detecting the characteristic absorption peaks in the infrared spectrum. The synthesis of benzil from benzoin using nitric acid can be monitored using infrared (IR) spectroscopy. Nitration of benzoin with nitric acid results in the formation of benzil, which has a different IR spectrum compared to benzoin.

NUCLEAR MAGNETIC RESONANCE:

This spectroscopy is based on the measurement of absorption of electromagnetic radiations in the radio frequency region from roughly 4to900MHz. The NMR spectrum of benzil which is characterized by the presence of aromatic and carbonyl signals.

RECRYSTALLIZATION

Recrystallization is a technique used in chemistry to purify a solid compound by dissolving it in a solvent and then allowing it to slowly crystallize out of the solution. This process is based on the principle that impurities are less soluble in a given solvent than the pure compound, so they will remain dissolved while the pure compound crystallizes out.

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The recrystallization of benzil using different solvents, including ethanol, ethyl acetate, acetone and water. The ethanol was the best solvent for recrystallization, producing pure benzil crystals with a high yield. Water was found less effective, producing smaller and less pure crystals.

IV. RESULT AND DISCUSSION:

IDENTIFICATION TESTS:

Parameters	description
Molecular formula	(C6H5CO)2
Molecular weight	210.23g.mol
Colour	Yellow crystalline powder
Odor	Characteristics
Melting point	96°c
Boiling point	345°c
Density	1.23g/cm
Solubility	Soluble in benzene, ethanol Insoluble in water, methanol

Table: No. 3

Parameters of benzil

UV SPECTROCOPY:

The use of UV light in the synthesis of benzil from benzoin allows for a more efficient and selective reaction to occur, as it can promote the reaction under milder condition compared to traditional chemical oxidants. The specific result obtained from the UV wavelength of 252nm indicates that the reaction is likely proceeding through a photochemical mechanism involving the carbonyl group in the starting material.

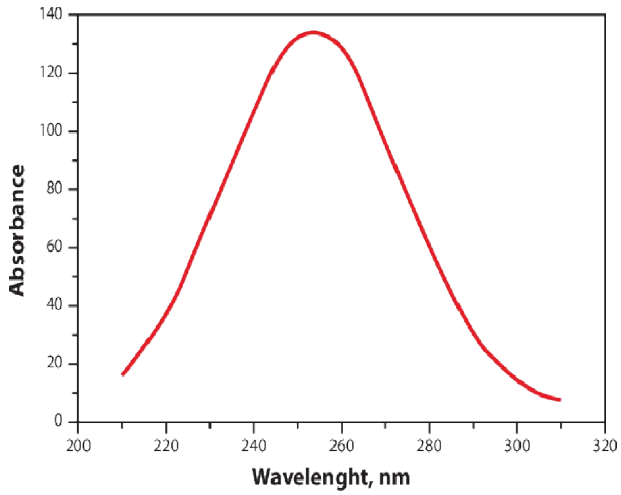

Fig. 2 UV spectroscopy of benzil

IR SPECTROSCOPY:

In the infrared spectrum of benzil, this is similar to the standard spectrum in the infrared spectrometer database. The absorption peak at 3063cm^{-1} corresponds to the C-H stretching vibration of the methylene group, the peak at 1659cm^{-1} corresponds to the C=o stretching vibration of carbonyl group. The carbonyl group which is conjugated with the benzene ring. So the absorption shifts to the low frequency (the normal absorption frequency of C=O is at 1740- 1700cm^{-1}) 1593cm^{-1} the absorption peak at this point corresponds to the vibration of benzil ring skeleton the strong peak at 1211cm^{-1} corresponds to the stretching vibration of C-C and the peak at 718cm^{-1} corresponds to the out-of-plane bending vibration of C-H on the benzene ring.

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Group	Absorption peak(cm^{-1})	Vibration
Methylene Group(C-H)	3063	Stretching Vibration
Carbonyl Group(C=O)	1659	Stretching Vibration

Table 3: The NMR of benzil

Group	Absorption peak(cm^{-1})	Vibration
C-C	1211	Stretching vibration
C-H	718	Out-Of-Plane

Table No.4: The Carbonyl group is conjugated to benzil rings and showing vibration with benzil ring

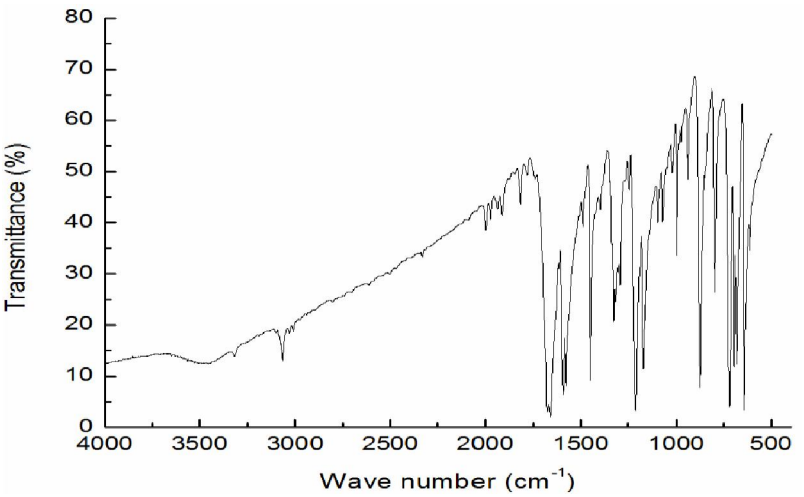

Fig.3. IR Spectrum of benzil

NMR Spectroscopy:

The obtained oxidation product benzil was also characterized by NMR and the result is shown in fig.4. The peak with chemical shift of 7.99-7.50 ppm corresponds to hydrogen on the benzene ring. According to the peak area data , the ration of three kinds of hydrogen is 2.10:1.00:2.19, which is close to 2:1:1 in accordance with the molecular formula of benzil, further confirming that the synthesis product was indeed benzil.

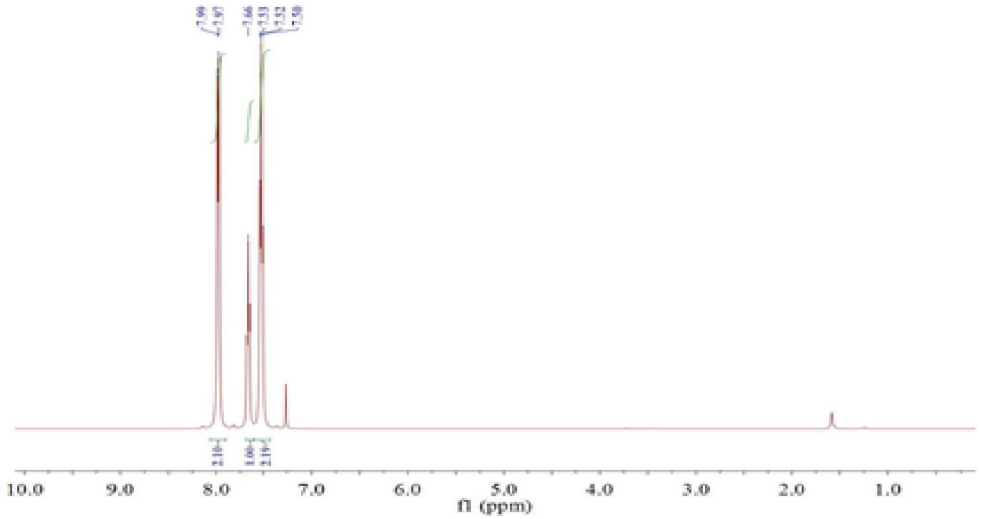

Fig.4. NMR spectrum of benzil

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V. CONCLUSION

The benzil was synthesized in high yield by the procedure. The product was successfully characterized by melting point, IR data, UV spectroscopy and NMR. The reaction proceeds via a series of intermediate steps, ultimately resulting in the formation of benzil. The yield of the reaction is influenced by several factors, including the concentration of the reactants, the temperature and reaction time. The synthesis of benzil from benzoin is a useful and straightforward method for obtaining this valuable organic compound.

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