

benzoin من الـ Benzoin $\leftarrow$ راح نحضر مركب الـ benzoin
من الـ Benzoin $\leftarrow$ صنعناه

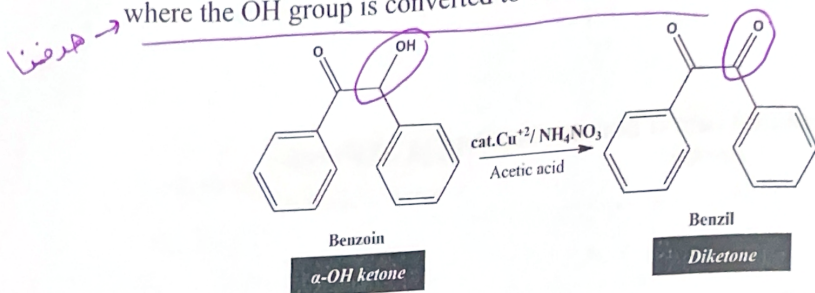
Experiment 3

Multistep synthesis of Phenytoin

Part 2: Oxidation of Benzoin to Benzil

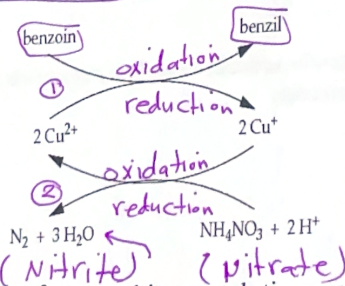
Dimerization
of Benzoin

Preparation of benzil can be done by Mild oxidation of benzoin using catalytic oxidation reaction / Copper acetate / nitric acid or any other mild oxidizing agent, where the OH group is converted to a ketone group.



- Benzoin can be oxidized to the diketone benzil using a Cu^{2+} salt and ammonium nitrate. Only catalytic amounts of copper(II) acetate are necessary because the Cu^{2+} is continuously recycled.

من الـ Benzoin $\leftarrow$ Benzil
Redox reaction
oxidation بالـ Cu^{2+}
(reduction بالـ Cu^{+})



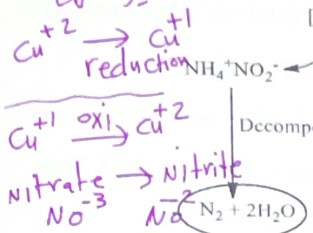
- Cupric acetate (Cu^{+2}) is used in a catalytic amount (less than 1% of stoichiometric requirement) and is continuously re-oxidized from the reduced (cuprous state Cu^{+}) by ammonium nitrate (NO_3^-) which is present in excess and is reduced to ammonium nitrite (NO_2^-) which decompose in the reaction mixture into nitrogen gas.

Refer to appendix IX: Oxidation of Benzoin to Benzil using nitric acid.

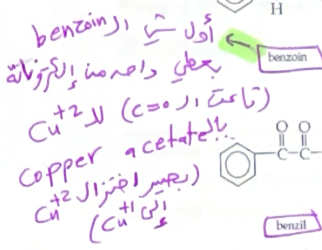
reoxidation بالـ Cu^{+2} $\leftarrow$ Cu^{+}
again and again from cycle to cycle
ammonium nitrate $\leftarrow$ $\text{NO}_3^- \rightarrow \text{NO}_2^-$
nitrate $\leftarrow$ nitrite

The reaction is

Ben Zoin $\xrightarrow{\text{Oxi}}$ Benzil $\text{NH}_4^+ \text{NO}_3^-$



- (Conjugate base of acetic acid) acetate ion بيجزي الـ



- resonance
stabilized
radical
11.5
benzoic
(more
stable)



Find other Mild oxidizing agents.

benzoin cation is benzyl acetate ion
other cu single بنزين

بنزين البروتون من benzoin
و بنزين benzil

Procedure

1. In a round bottomed flask place 1.75 g of unrecrystallized benzoin, 5 ml of glacial acetic acid, 0.8 g of pulverized (reduced to fine particles) ammonium nitrate, and 1 ml of a 2% solution of cupric acetate.
2. Add 1-2 boiling chips, attach a reflux condenser and bring the solution to a gentle boil. As the reactants dissolve, evolution of nitrogen begins.
3. Boil the blue solution for 1.5 hr to complete the reaction.
4. Cool the solution to 50-60°C and pour it into 10 ml of ice-water mixture in a beaker [to ppt product, since benzil insoluble in water], while stirring it. Then Benzil separates out as yellow oil, which immediately solidifies.
5. After crystallization of benzil is complete, collect the crystals on suction filtration and wash them thoroughly with water.
6. Press the product as dry as possible on the filter.
7. If desired, it may be purified by re-crystallize from methanol or 75% ethanol. (After dissolving the product in hot ethanol, add water dropwise to reach the cloud point and allow it to crystallize).

	Weight (gm)	M.P
Benzil		

4 Brain storming Question

- 4.1 Discuss the chirality of the product, does it optically active? Explain your answer.
- 4.2 What structural features make benzil yellow and benzoin colorless (white)?
- 4.3 Would you have obtained the same results for the oxidation of benzoin if the label on the copper acetate bottle had read "cuprous acetate"?