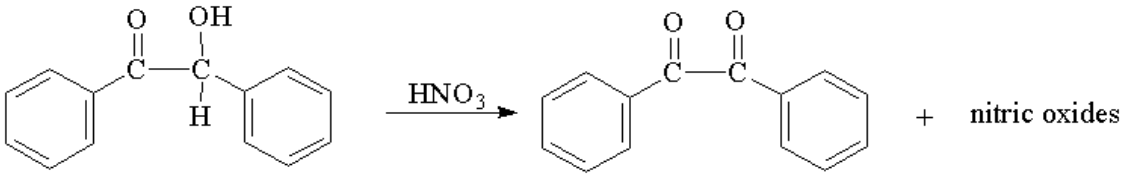
Oxidation of Benzoin to Benzil.



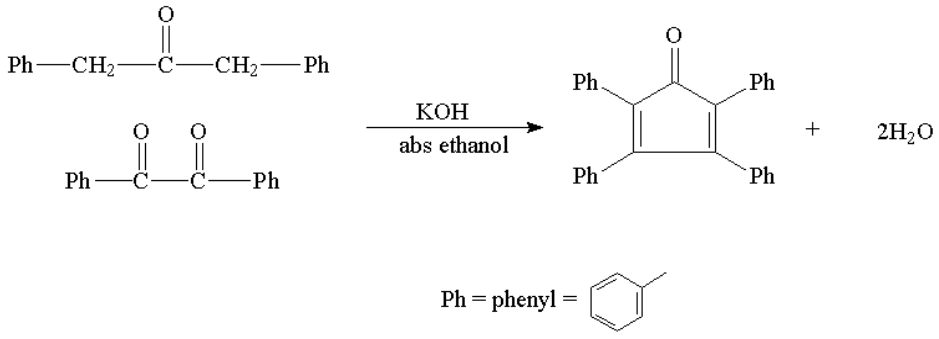
CAUTION: Concentrated Nitric Acid is extremely caustic and will burn exposed skin.

Work in a hood! Into a 125 mL Erlenmeyer flask, place 2.0 g of benzoin (weighed to the nearest tenth of a g) and carefully add 7 mL of concentrated nitric acid. Heat the mixture on a hot plate with occasional slow swirling for 30 minutes or until the brown-red nitric oxide gases are no longer evolved. The fumes are toxic and noxious so be certain that the fume hood sash is pulled down to a level that prevents the gases from escaping..

Carefully cool the flask and contents using a slushy ice bath. Once your reaction reaches about room temperature, then pour into 35 mL of ice water and swirl to coagulate the precipitated product. Collect the yellow solid using suction filtration and wash twice with 5 mL of cold water to remove some of the nitric acid present. Press the crystals to remove more water by placing another piece of filter paper over the crystals and pushing with a beaker or cork; the suction flask MUST be supported and sitting flat on the desktop. This crude product can be recrystallized from 95% ethanol while it is still slightly wet (4 mL/g). Dissolve it in hot ethanol, add water dropwise to reach the cloud point (it looks and stays cloudy), and allow it to slowly crystallize. Cool on ice, filter, dry, record the yield, and take the mp. Drying will take a while so store in an open container (it won't dry if you seal up the container) covered with a chemwipe. Once dry, weigh, record the yield and the percentage yield. Record the mp; literature mp 94-96 ºC).

Dispose of waste as instructed. Typically, one of the hoods in the lab will contain waste jars for the lab activity.

Preparation of Tetraphenylcyclopentadienone.



Cyclopentadienone is a relatively unstable compound which will dimerize even at low temperature. However, the corresponding tetraphenyl compound is quite stable.

Procedure: Into a 50 mL round bottom flask, place 0.7 g of benzil, 0.7 g of dibenzyl ketone, and 5 mL of absolute ethanol. Attach a reflux condenser and affix a drying tube that is charged with calcium chloride. Heat the mixture until the solids dissolve. It is critical to prevent water (moisture) from coming into contact with the reactants, hence the drying tube. Raise the temperature to provide a slow reflux and add a solution of 0.1 g of potassium hydroxide (CAUSTIC) in 1 mL of absolute ethanol (this solution will be already prepared) dropwise through the top of the condenser by briefly removing the drying tube as you add the hydroxide. The reaction is very fast and a purple color will appear.

After addition of the base, allow the mixture to reflux for 15 minutes while periodically shaking the flask. Cool the reaction flask to room temperature, then in an ice bath. Filter using vacuum, wash twice with 5-mL portions of cold 95% ethanol, and air dry. When the crystals are dry (which may take until the next lab period), weigh, record the yield and the percentage yield. If needed you may recrystallize a portion of the purple product using a 1:1 mixture of 95% ethanol and toluene (12 mL/0.5 g). Record the mp; literature mp 219-220 ºC).

Dispose of waste as instructed.