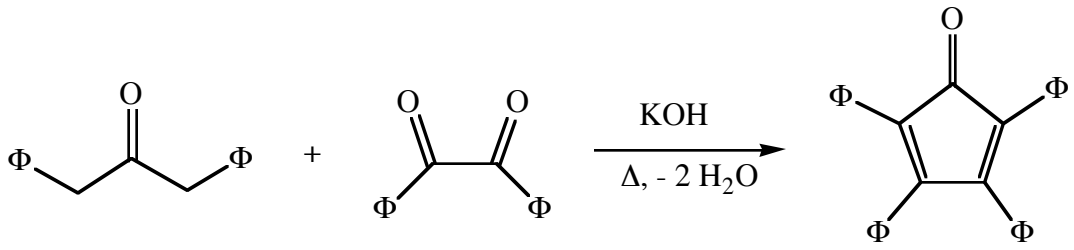
Synthesis of tetraphenylcyclopentadienone (TPCP)



1,3-diphenylacetone Benzil

tetraphenyl cyclopentadienone
(deep, dark purple color)

mp = 218-220 ˚C

Here is another reaction that is an example of two aldol reactions: one intermolecular reaction followed by an intramolecular reaction. However, the carbonyl that is being attacked is a ketone and not an aldehyde. In lecture, we learned that this intermolecular reaction is endothermic and will not produce much product. This is because ketones are more stable than aldehydes. Therefore, the fact that this reaction does produce the TPCP product in good yield tells us that the ketone being attacked (benzil) must be less stable than a normal ketone. Examine the structure of benzil and try to determine why it is less stable and more reactive than a normal ketone.

Experimental Procedure

Place 0.300 g of benzil, 0.300 g of 1,3-diphenylacetone, and 2.4 mL of ethanol in a 5 mL round bottomed flask. Place a magnetic stir bar in the flask, and attach a condenser to your flask. Heat this mixture with a sand bath* at approximately 80 ˚C until the solids dissolve. After the solids have dissolved, carefully add 0.50 mL of ethanolic potassium hydroxide (ethanol with 1 g of KOH dissolved in each mL) by using a 9-inch disposable pipet (the long disposable pipets). You can add this ethanolic potassium hydroxide without removing the condenser. This will be an exothermic reaction, so the solution will begin to foam and boil. It will also immediately turn deep purple. After adding the potassium hydroxide solution, raise the temperature of your sand bath to heat the mixture at reflux. Reflux your mixture for 15 minutes.

* In order to be able to stir your reaction using the magnetic stirrer, we will use a different set-up for our sand bath. Use a tuna fish can filled part way with sand as your sand bath. Place this on top of the hot plate/magnetic stirrer. You can then heat the sand and stir the solution at the same time. Once the period of reflux is complete, remove the flask from your sand bath and immediately transfer the hot solution (pour it quickly) to a small beaker or erlenmeyer flask. You may have to rinse the reaction flask with a **small amount** of hot ethanol to remove all the product. Allow the solution to slowly cool to room temperature, and then place your beaker in an ice bath. This will complete the recrystallization of your product. Collect your crystals on a Hirsch Funnel, and remember to wash your crystals with a small amount of **cold** ethanol.

QUESTIONS

1. Why is benzil more reactive than a normal ketone?
2. Why is this reaction so exothermic?