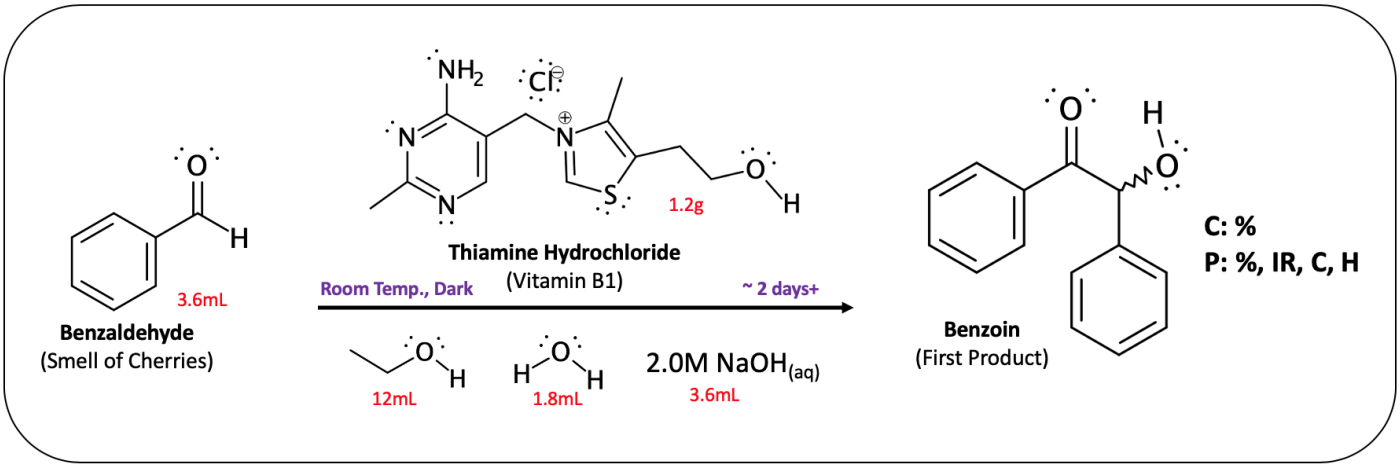
4B. Procedure for Benzoin Synthesis



Note on Data Collection

In the procedure below, approximate amounts of reactants are given. If you are lucky enough to be in the laboratory to record these values, do so to the most decimal places given by the instrument. If this experiment is being done virtually online, watch the video of the experiment and use the values from the video to fill in the Reaction Table.

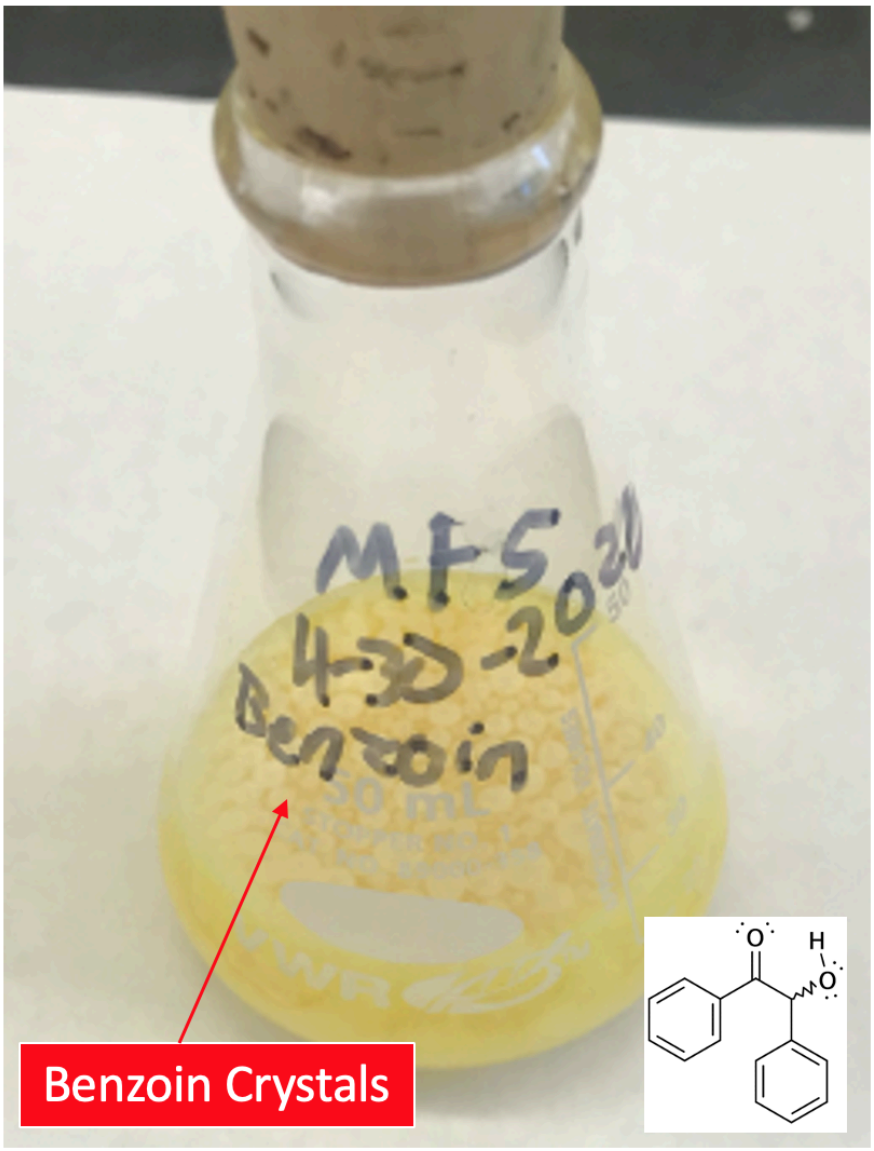
Reaction Setup

1. Weigh out ~1.2g Thiamine HCl into a 50mL Erlenmeyer Flask labeled with your name, date and Benzoin Synthesis. Record your actual amount into the Reaction Table, in the Google slide linked below...
   Report Worksheet for Benzoin
   (See note above if doing this experiment virtually)
2. Add ~1.8mL Water to the same Flask, gently swirl for a few seconds. Record into the Reaction Table.
3. Add ~12mL Ethanol to the same Flask. Record into the Reaction Table. Gently swirl for about 30 seconds.
4. Add ~3.6mL 2.0M NaOH(aq) to the same Flask. Record into the Reaction Table. Gently swirl and the solution will become bright yellow at first then fade to a lighter yellow after about 30 seconds. Record into the Reaction Table.
5. Purge the Erlenmeyer Flask with Nitrogen gas and cork. Record the mass of this Flask, the cork and all the reactants so far. You can write this on the side of the Reaction Table.
6. Let me help you cannulate ~3.6mL of Benzaldehyde into the Fask (as is described in the Introduction), purge with Nitrogen gas, cork and record the mass. Calculate and write the mass of Benzaldehyde, into the Reaction Table.
7. Place this Flask in a slightly larger Beaker and store in the dark, in your locker for two or more days.
8. Crystals should form after a couple days, if not try to start crystallization with the following methods…
   a. Use a microspatula or glass rod to scratch into the solution then up the inside wall and out of the solution.
   b. Cool in an ice water bath (Beaker just large enough to fit it).
   c. Ask a lab partner for a seed crystal and try seeding the flask.
      i. If these methods do not work ask for help.

Crude Product Isolation

After Benzoin has precipitated it will be isolated via vacuum filtration with Ice cold water.

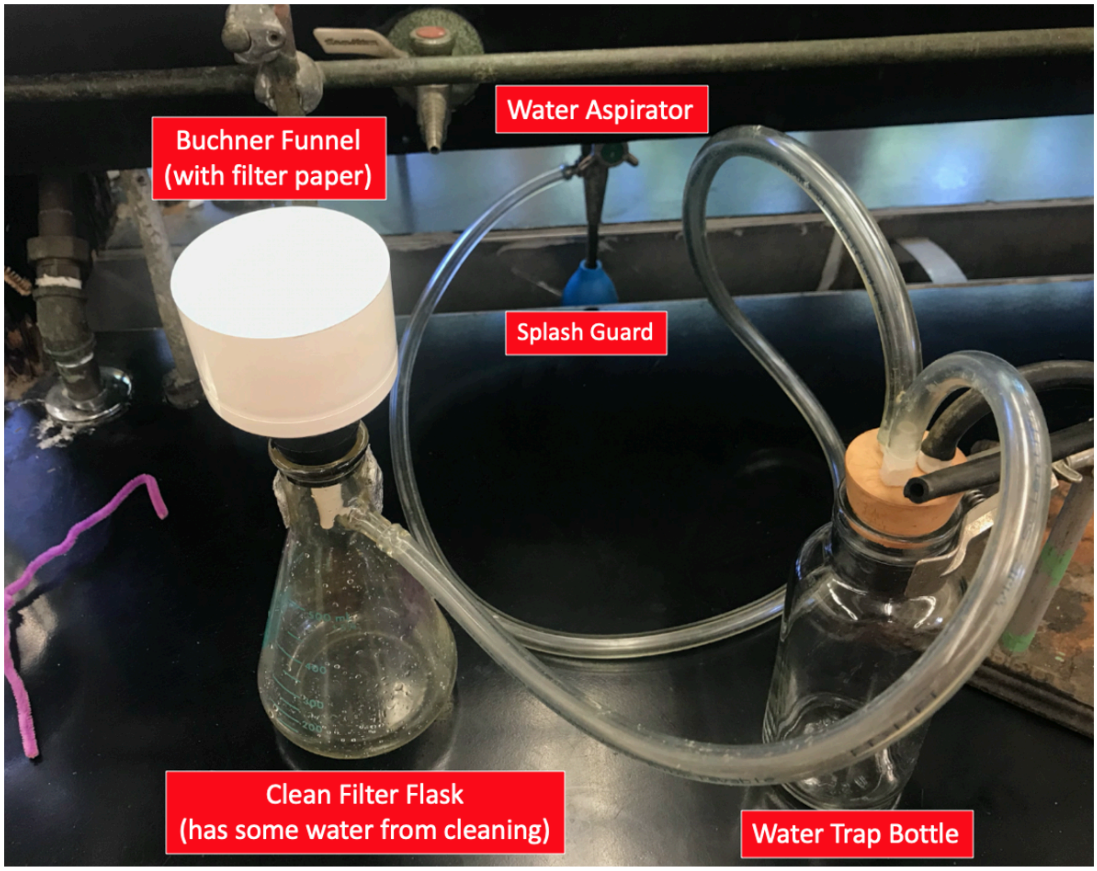
Crude Benzoin Crystals



1. First cool the Reaction Flask in a Beaker of ice water for 5-10min. Choose a Beaker that is only slightly larger than the Flask.

2. Using the sink's water Aspirator, Water Trap Bottle, Filter Flask and Buchner Funnel you will filter away the other reactants from the Crude Benzoin Crystals.

Vacuum Filtration



3. Start the vacuum filtration, as you've been done before by…
    a. Turning on the water Aspirator to draw a vacuum
    b. Wet the filter paper with ice cold water (no solid ice)
    c. Break up the crude crystals of benzoin with a micro spatula
    d. Swirl the Flask and quickly dump the crystals onto the center of the Filter Paper.
    e. Use the ice cold water to help transfer your crystal out of the Flask and to wash the crystals on the filter paper. The washes will help remove some of the yellow impurities but will also be lowering your yield so don't wash too much.
    f. Let the crude crystals dry on the filter paper under vacuum for a few minutes.
    g. Carefully remove the filter paper and crystals from the Buchner funnel, using tweezers and transfer to a watch glass to air dry. The crystals can be left to air dry on the Filter paper / watch glass for 10mins to days.

h. After the crystals are dry (or mostly dry) on the Filter paper, gently scrape the crystals from the Filter paper onto a different ***pre-weighed*** watch glass. If the crystals are dry determine the crude mass of Benzoin and record into the Reaction Table.

Purification via Ethanol Crystallization and Characterization

1. Using the standard crystallization procedure you will use hot ethanol and vacuum filtration to purify the Benoin.
2. Record the mass of the crystallized Benzoin into your Reaction Table.
3. Take an IR spectrum of your Crystallized Benzoin (not required for the Crude benzoin).
4. Take a ^{1}H-NMR spectrum of your Crystallized Benzoin (not required for the Crude benzoin). Use CDCl3 with TMS unless you are lucky enough to have Deuterated DMSO with TMS.
5. Take a ^{13}C-NMR spectrum of your Crystallized Benzoin (not required for the Crude benzoin).