56A:

Observations and Measurements:

After reacting the Thiamine HCl and water, ethanol was added and the mixture was cooled which made the mixture a cloudy white color. After a certain point in adding the NaOH, the mixture became yellow and pH was tested after all NaOH was added. Based on the pH paper, the pH was around 9.6 to 10. Adding the benzaldehyde and leaving it over the week after the reaction yielded a yellow precipitate, but after washing with water and methanol in vacuum filtration, the color was lost and the precipitate became a white solid.

Table 1: Melting Points

Benzoin Type	Start Range	End
Pure	121	123
Experimental	120	123

Table 2: Collected Data

4.999g Thiamine HCl used	15.924g Benzaldehyde used
Weighing Paper Mass: .421g	Total Mass: 14.782g

Data and Calculations:

Actual mass: 14.782g - .421g = 14.361g

Theoretical Yield: 15.924g * 1 mol / 106.1g * 1 mol / 2 mol * 212.2 g / mol = 15.924g

Percent Yield: 14.361g / 15.924g * 100 = 90.18%

Discussions and Conclusions:

The recorded melting point of Benzoin is 137 C, but when collecting the melting points of pure vs experimental, both ranges start around 120 and end at 123 C. This shows that the experimental product was close to the pure product having the thermometer being the error. From the IR, there are peaks at the 3350-3450cm $^{-1}$ range where it corresponds to the alcohol group in the carboxylic acid. The ketone group for the carboxylic acid is in the 1650-1700 range which lines up with a documented IR of Benzoin.

56B:

Observations and Measurements:

After adding the Copper(II) Sulfate to the reaction flask, the reaction mixture turned blue at first, then turned brown, and finally green after the reaction mixture got to the reflux point.

After refluxing, adding the mixture to crushed ice created a foamy yellow mixture which was vacuum filtrated and yellow crystals and impurities remained. After recrystallization from ethanol, the product that was obtained was yellow.

Table 1: Melting Points

Benzil Type	Start Range	End
Pure	90	93
Experimental	90	94

Table 2: Collected Data

Benzoin used: 13.783g	Ammonium Nitrate used: 6.889g
Copper Acetate used: .148g	Ethanol used: 57mL
Crude Product Weight: 18.157g	Final Benzoin Mass: 9.064g

Data and Calculations:
Theoretical Yield: 13.783g * 1mol / 212.2g * 1mol/1mol * 210.2g/1mol = 13.653g
Percent Yield: 9.064g / 13.653g * 100 = 66.39% yield

Discussions and Conclusions:

The recorded melting point of benzil is 95-96 C, but based on the melting point collection of the pure and experimental samples, both ranges start at 90 with the pure ending at 93 and experimental ending at 94. This means that the experimental sample is close to the pure sample, but impurities could be within it and there could have been an error with the thermometer. For IR spectra, there are peaks around the 3300-3400 range, but those peaks correspond with the peaks of benzoin which means that some unreacted product is in the final product. To tell that this product is benzil, there is a peak at the 1680 cm-1 that corresponds to a ketone peak, a peak at 1590 cm-1 that corresponds to C=C stretch, and a peak around 3050cm-1 that corresponds to a C-H sp2 stretch.

56C:

Observations and Measurements:

After starting reflux, the mixture was first turning brown, then purple, then black. Adding the mixture to HCl and crushed ice created a slurry mixture and filtering the slurry mixture yielded a white solid. A lot of water was required to recrystallize the white solid and during

recrystallization, there was a lot of yellow-brown impurities within. The final product was a white solid.

Table 1: Melting Points

Benzilic Acid	Start Range	End
Experimental	140	143

Table 2: Data Collected

Benzil used: 8.473g	6M KOH used: 21.1825 mL
EtOH used: 25.419 mL	Benzilic Acid mass: 1.855g

Data and Calculations:
Theoretical Yield: 8.473g * 1mol / 210.2g * 1 mol/1 mol * 228.2g / 1 mol = 9.199g
Percent Yield: 1.855g / 9.199g * 100 = 20.16%

Discussions and Conclusions:

For melting points, the documented melting point of benzilic acid is 153 C, while the experimental is 140-143 C. Since there isn't a pure sample this time to compare with, there could be impurities within the sample or there was an error with the thermometer used. For IR, there should be two distinct peaks for both alcohol groups in the spectra. The spectra shows one peak at around 3390 cm-1 and the second peak around 2950cm-1. These two peak differences display the different environments both alcohol groups are in. The 3390cm-1 peak shows that the group is near a symmetrical environment but the 2590 cm-1 peak shows that the environment is completely different and has a presence in acid. There is also a ketone peak around 1720 cm-1 that corresponds with the carboxylic acid in the compound.

Exercise Questions:
3 a. This white solid means that the benzaldehyde was not pure, which would lower the yield in the multistep synthesis.
B. Using cuprous acetate instead of Copper(II) Acetate means that the oxidation process that would be catalyzed by Copper (I) would not yield the desired product.
C. This reaction required a pH that was less than or equal to 2 in order for the rearrangement to occur. And since the pKa of a carboxylic acid is 4, having the pH under 2 allows for the targeting of a specific location.
7 Same reactions, but start with Furfural.
8

Click Chemistry:

Observations and Measurements:

For phenol propargylation, the mixture that was created and heated at 60°C yielded a yellow mixture, but after rotovapping, the solid is a tint of yellow. The collected solid was a tint yellow that resembled a light coffee powder. Using the product from the propargylation, adding the 2-bromoacetophenone, 4-acetylphenyl propargyl ether, and sodium azide created a yellowish mixture. Adding the ascorbate and letting it dissolve created a clear and murky mixture and with the addition of Copper(II) Sulfate changed the mixture color to brownish, showing a reduction of the copper. Adding the mixture into ice cold ammonium hydroxide and water created a slurry solid which after filtering yielded a dirty yellow solid product.

Table 1: First TLC Data of 4-hydroxyacetophenone in 40% Hexanes/60% Ethyl Acetate

Distance Traveled: 4.0cm	Solvent Front: 10.5cm

Table 2: TLC Data of propargylation product and final product in 40% Hexanes/60% Ethyl Acetate

Propargylation	Distance Traveled: 6cm	Solvent Front: 10.5cm
Final	Distance Traveled: 6.5cm	Solvent Front: 10.5cm

Table 3: Data Collected

4-hydroxyacetophenone used: 1.365g	DMF used: 7.3mL
Propargyl Bromide used: 1.5mL	Potassium Carbonate used: 1.946g
2-bromoacetophenone used: .397g	Sodium Azide used: .139g
4-acetylphenyl propargyl ether used: .352g	Sodium Ascorbate used: .041g
250mL RBF and Holder mass: 137.262g	Total mass: 138.694g
Final Product Wet Mass:	Final Product Mass:

Data and Calculations:

Rf: 4.0cm / 10.5cm = .38

Propargyl Product Yield: 138.694g - 137.262g = 1.432g

Theoretical Yield of phenol propargylation: 1.365g * 1mol/136.15g * 1mol/1mol * 174.196g = 1.746g

Percent Yield: 1.432g / 1.746g * 100 = 82.00%

Rf: 6cm / 10.5cm = .571
Rf: 6.5cm/ 10.5cm = .619
Theoretical Yield of final product: .397g * 1mol/174.196g * 1mol/1mol * 335.357g = .764g
Percent Yield:

Discussions and Conclusions:

The percent yield of the propargylation was 82.00% and the Rf of the 4-hydroxyacetophenone was .38. This ratio of hexanes to ethyl acetate was the one used for the next TLC determination. The