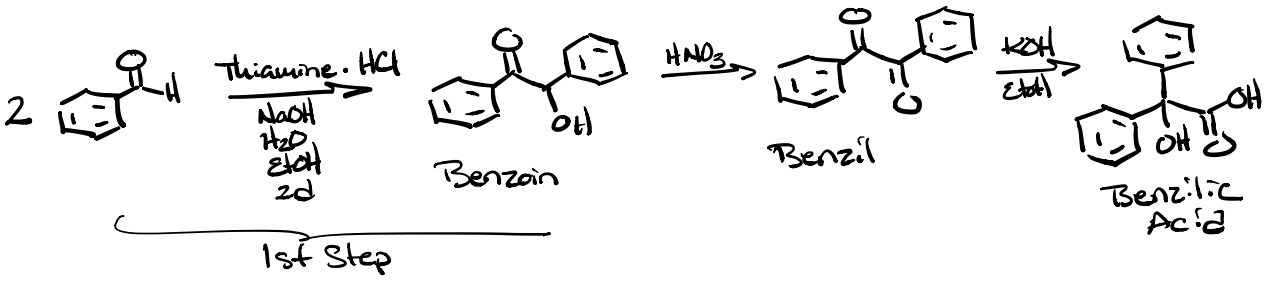
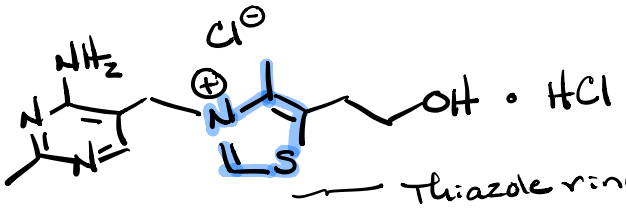
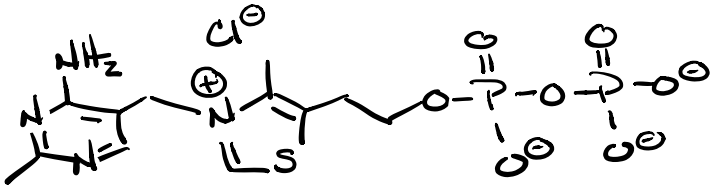
Multistep Synthesis of Benzilic Acid



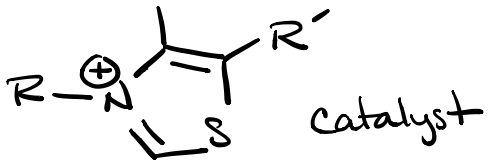
Thiamine Vit B_1



Thiazole ring "active Catalyst"



Thiamine pyrophosphate



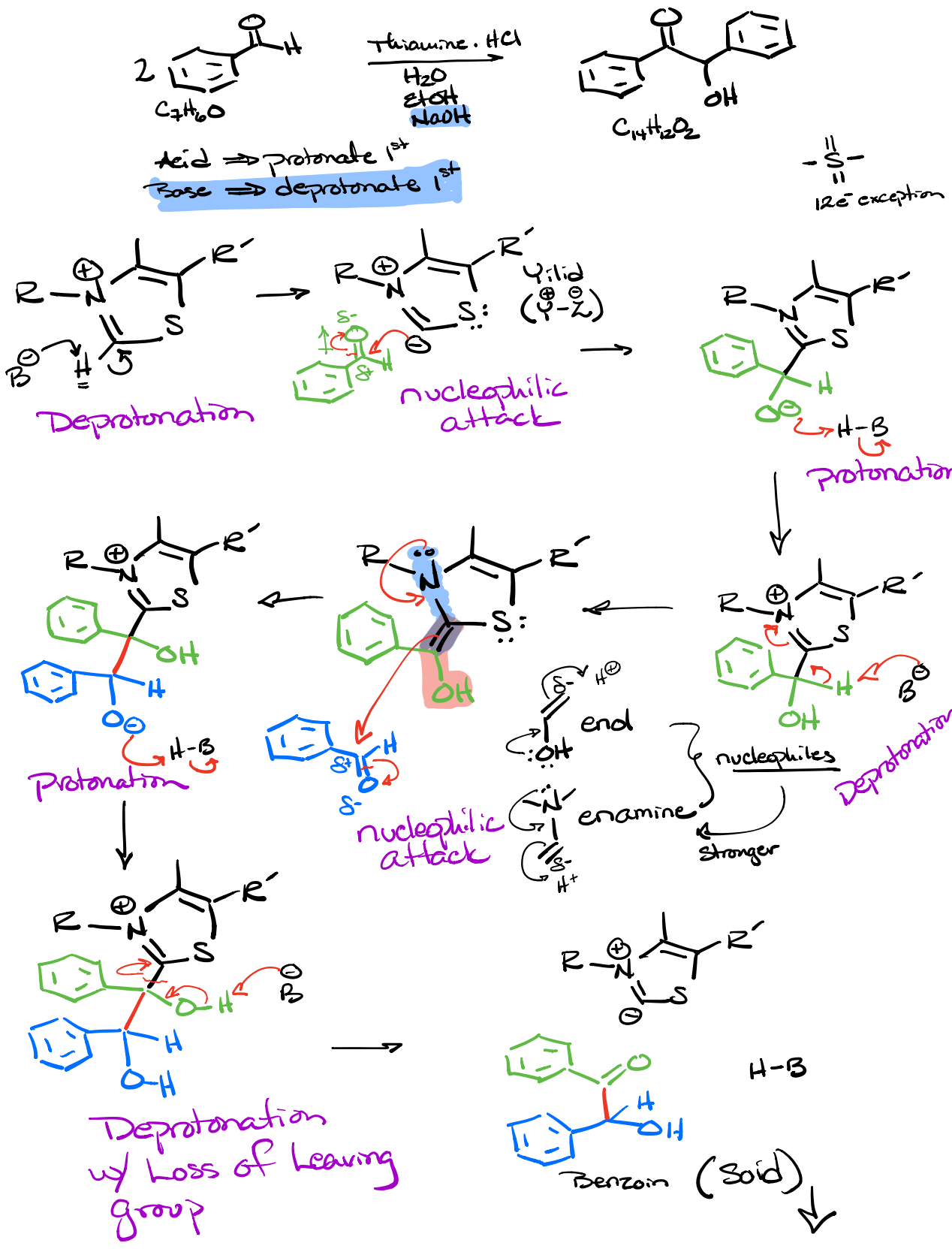
Catalyst

New C-C bond

$R\text{-}Mg\text{-}Br$ $\quad R'\text{-}LG \xrightarrow{S_N2} R\text{-}R'$

$R\equiv^{\ominus}Na^{\oplus}$ $\quad R'\text{-}LG \xrightarrow{S_N2} R\text{-}R'$

not Compatible w/ H_2O


2
C_7H_6O
Thiamine · HCl
H_2O
EtOH
NaOH
$C_{14}H_{12}O_2$
Acid ⇒ protonate 1st
Base ⇒ deprotonate 1st
12e⁻ exception
Deprotonation
Ylid
nucleophilic attack
Protonation
Protonation
Deprotonation
nucleophiles
enamine
Stronger
end
nucleophilic attack
Deprotonation w/ Loss of Leaving group
H-B
Benzoin (solid)

Procedure

- 0.30 g Thiamine·HCl into 25-mL Erlenmeyer
- Add 0.45 mL DI H_2O, Swirl flask
- Add 3.0 mL 95% EtOH, Swirl flask
- Add 0.90 mL 2 M $NaOH_{(aq)}$ Swirl flask
  ⇒ Turns bright yellow & fades to dull yellow over about 3 min.
- Tare the flask
- Add 0.90 mL benzaldehyde
- Re-weigh flask to find Mass Benzaldehyde.
- Allow to stand for 2 days

Workup

- Ice for 10 min
- filter on Hirsch, rinse w/ 1 mL DI H_2O
- Take mass ⇒ % yield
- Recrystallize
- MP
- IR

Grignard Formal — What can we use from the Formal Guidelines online?

procedure in Pavia et. al.[ref #] was followed without modification). Any deviations, additions or omissions to the reference procedure must be clearly stated here. The experiments designed by individual students must be described in a separate paragraph.

Your Mel-Temps are Mel-Temp II by Barnstead/Thermolyne model 1001 with a Fluke 51 K/J digital thermometer. The IR instrument is a Nicolet Avatar 360 FTIR ESP with Omnic 8 software by Thermo Fisher Scientific.

An example of what this section should look like is given below. We do not have all of this instrumentation, this is only for example.

Bromobenzene, magnesium turnings, anhydrous diethyl ether, 6 M $HCl_{(aq)}$, and petroleum ether were obtained from the chemistry stockroom and used without modification. Melting points were determined using a Barnstead/Thermolyne Mel-Temp II model 1001 equipped with a Fluke 51 K/J digital thermometer. Infrared spectra were obtained on a Perkin-Elmer 1600 Series FTIR. ^{1}H-NMR and ^{13}C-NMR were obtained on a Bruker AC250 250 MHz NMR equipped with a quad nucleus probe, and in some cases on a Varian Unity500+ 500 MHz NMR. Chemical shifts are reported relative to tetramethylsilane in δ ppm. ^{11}B-NMR spectra were obtained on a Bruker AC250 250 MHz NMR equipped with a quad nucleus probe. ^{11}B-NMR chemical shifts are reported relative to BF_3-OEt_2 in δ ppm. Purity of the materials synthesized, unless noted otherwise, was assessed solely through ^{1}H-NMR.

different IR

was not used

Fifteen milligrams of magnesium turnings (6.43 mmols) were placed into a 20-mL round bottom flask. The flask was fitted with a magnetic stir bar, a Claisen head with rubber septa, and drying tube. A solution of Bromobenzene (1.01 g, 6.43 mmols) in anhydrous diethyl ether (4.0 mL) was added to the stirred reaction flask via syringe dropwise. Addition of bromobenzene/diethyl ether solution was maintained at a rate to produce a steady reflux of the reaction mixture. After addition of bromobenzene/diethyl ether solution, the reaction was allowed to cool to room temp. A solution of benzophenone (1.09 g, 5.98 mmols) in anhydrous diethyl ether (2 mL) was added rapidly via syringe. The reaction was allowed to stir for 20 min. The reaction mixture was quenched with $HCl_{(aq)}$ (6.0 mL). The resulting biphasic system was separated using a separatory funnel, and the aqueous phase extracted with 10 ml diethyl ether. The ether layers were combined and evaporated to yield a yellow solid/oil mix. The solid was triturated with petroleum ether and filtered to yield the crude triphenylmethanol (1.25 g, 4.80 mmols) as a white powder. The crude material was recrystalized from isopropanol to afford the pure triphenyl-

or

the procedure in Pavia [#ref] was followed without modificaton.

methanol. Yield: 1.04 g, 4.00 mmols (67%). Mp. 160.5 – 161.0 °C. FT-IR (Solid, KBr pellet, cm^{-1}): xxxxx, xxxxx, xxxxx, xxxxx (where xxxxx are the important wavelengths for characterization).

Results

This section includes data tables, graphs and sample calculations. All tables and graphs will have appropriate titles and be a half page in size. The axes of the graphs will be properly labeled including units. Results should be summarized in text as well as tables. I know it's redundant, but that's only because our data sets are rather small.

Discussion/Conclusion - These may be treated as one or two separate sections

For organic papers, the first part of the discussion should be about the reaction mechanism. This is not a hard rule. If the mechanism is very small it can be placed in the introduction section. Likewise if there are topics to discuss that could come before the mechanism it is fine to do so.

The mechanism should be broken down into individual reaction steps. Each step should be proceeded by a figure and caption illustrating the step. The text should then explain what is taking place. The figures can be hand drawn, as can the caption headings. Computer drawn images are really nice but take a long time to master. There are a number of free applications for Macs and PC's that will allow you to draw molecules, however I would rather have you focus on other areas of the report and simply draw the reaction diagrams by hand. Just leave space for the figures in the text and add them afterwards.

In addition to the mechanism the discussion section is generally where the results section is thoroughly integrated to produce sound conclusions. Critical thinking is employed to analyze the results and deduce valid conclusions. In organic chemistry this section should include a discussion of any experimental difficulties, errors, or questions. It may also include answers to lab manual questions. In fact I would recommend looking at the lab manual questions and using them as an outline with which to construct this part of the discussion.

The Conclusion section is very brief, usually a single paragraph. Most often I think of this as a restatement of the abstract. Don't just copy the abstract however. The conclusion should reaffirm what was done and what was found. Sometimes, comments regarding future experiments may be appropriate.

Language and Format

In general, the report is written in the third person, impersonal observer voice. For example, "Ten grams of NaOH was weighed", **not** "I weighed 10 g of NaOH."