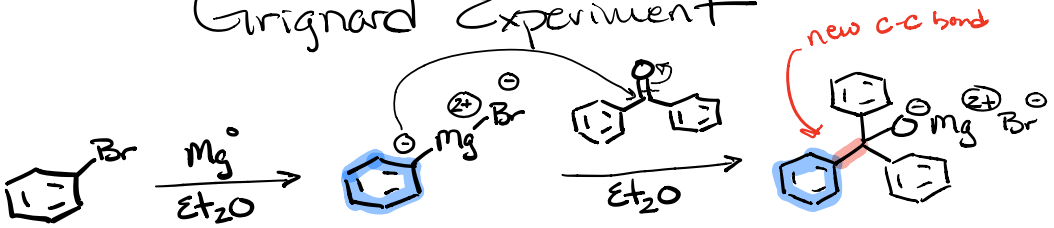
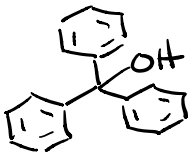
Grignard Experiment

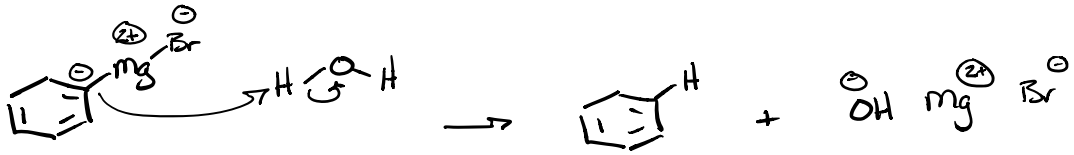
Experiment 33A



Et_2O = diethyl ether



triphenylmethanol
or
triphenylcarbinol

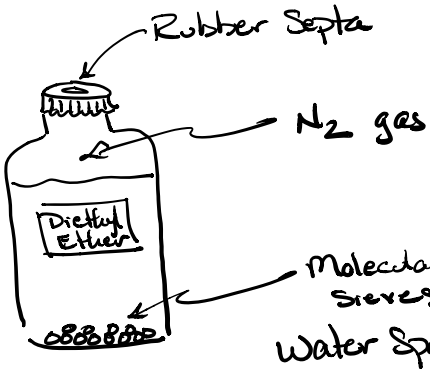


Strong Base

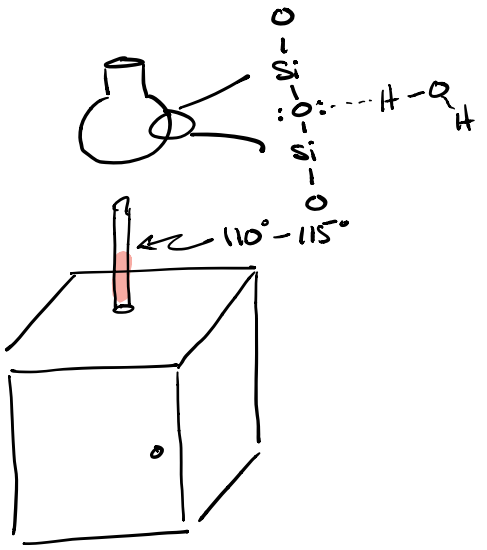
Diethyl ether

hygroscopic

Anhydrous diethyl ether

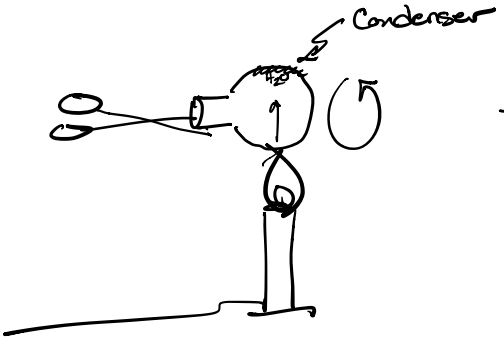


Water Sponges
Clay material
Slow at absorbing water

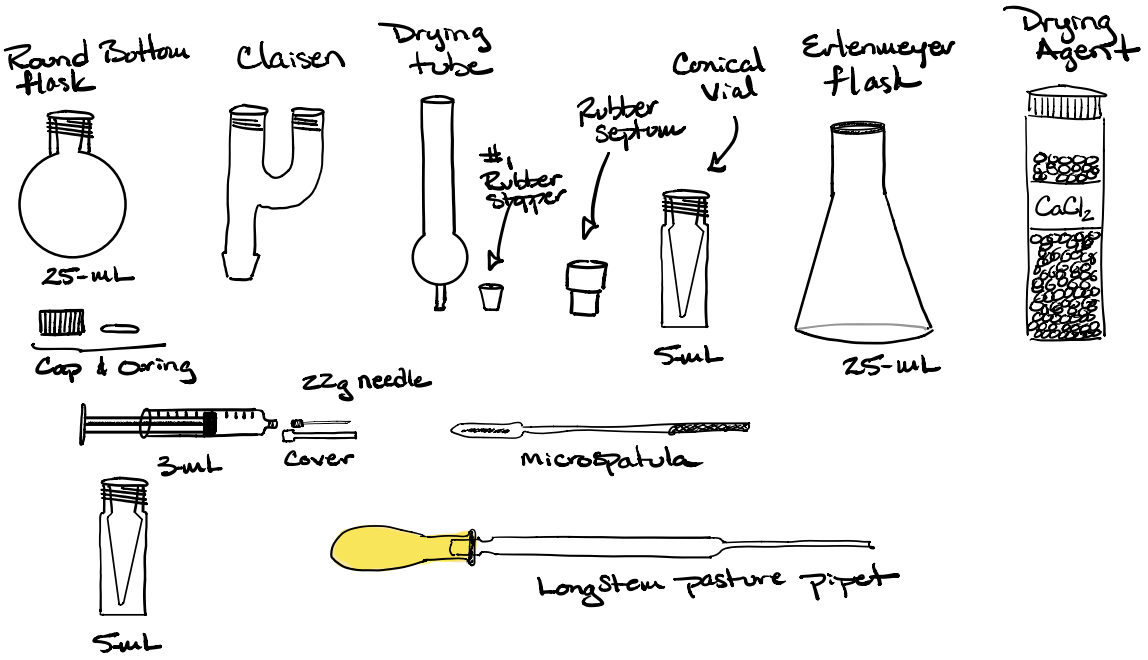


hydrophilic

- Reagents are dried chemically
  (solvents & starting materials)
- Glassware dried in oven for 24 hr

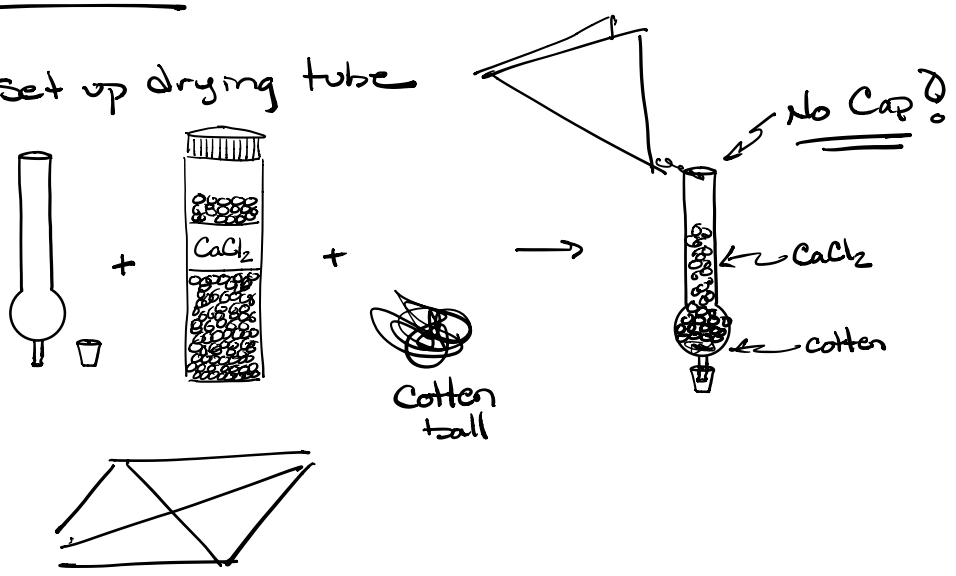


flame drying

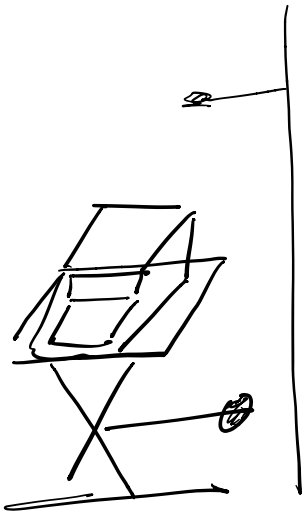


Procedure

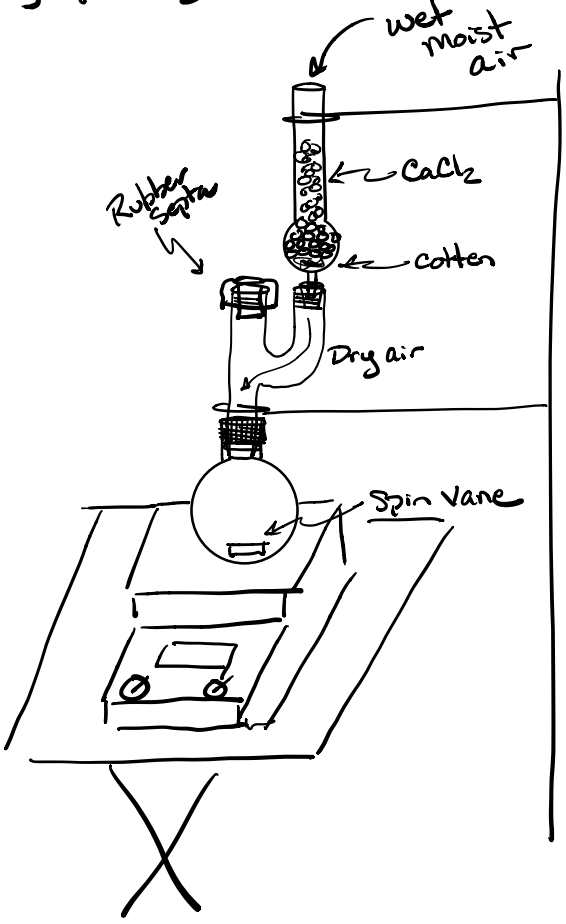
1. Set up drying tube



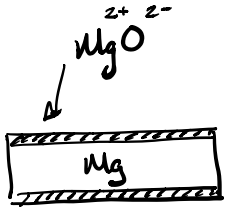
② Assemble Jackstand, Clamps, Hot-plate



③ Bring glassware out of oven and assemble fairly quickly while still hot.



④ weigh out ~0.15 g Mg turning



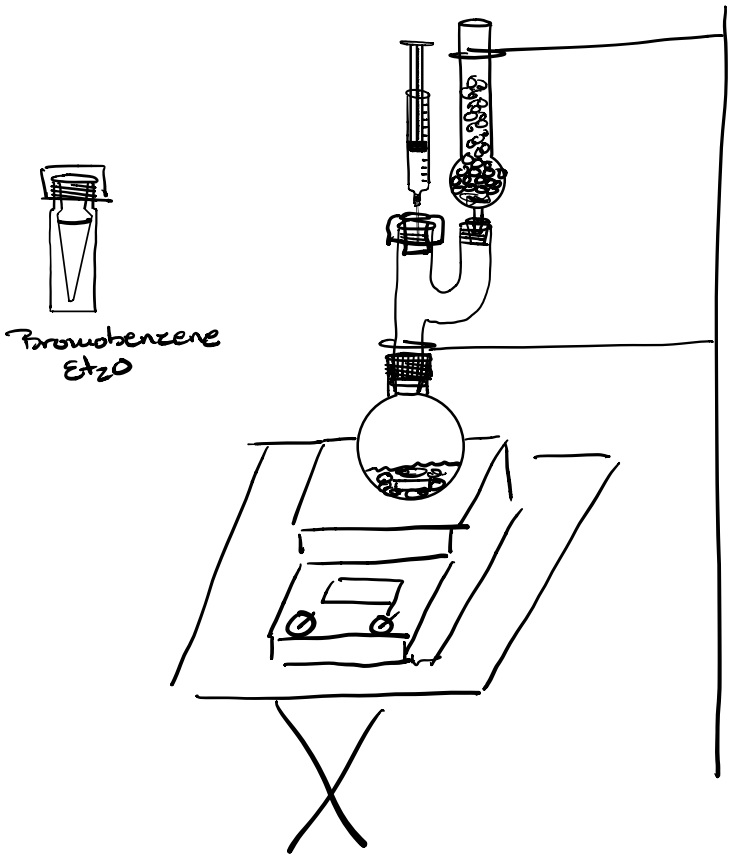
⑤ Add Mg turnings to RB flask

⑥ - Use 5-mL Conical vial
- tare vial
- Add ~0.70 mL bromobenzene using calibrate pasture pipet
- Reweigh the vial to obtain mass of bromobenzene

Volume is temperature dependent
mass is constant

- Add 4.0 mL of anhydrous diethyl ether to make a solution w/ bromobenzene

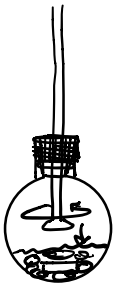
⑦ Use the syringe to add ~ 1 mL of the bromobenzene/diethyl ether solution to the round bottom flask through rubber septa on the Claisen.



ways to deal w/ oxide

- ~~Add~~ small amount of Solid I_2
- Sonicator
- *- Crush Mg

⑧ Call me over to crush the Mg



Reaction becomes turbid & grey as Reaction Starts.

⑨ Add remaing bromobenzene solution to flask using Syring over 15 min.

* Exothermic & Bp of Et_2O 32°C

Add at a rate below boiling of the reaction

⑩ Rinse the Conical vial w/ 2.0mL of Et_2O and add that rinse to the reaction via Syringe.

⑪ Use Same Conical vial

tare vial

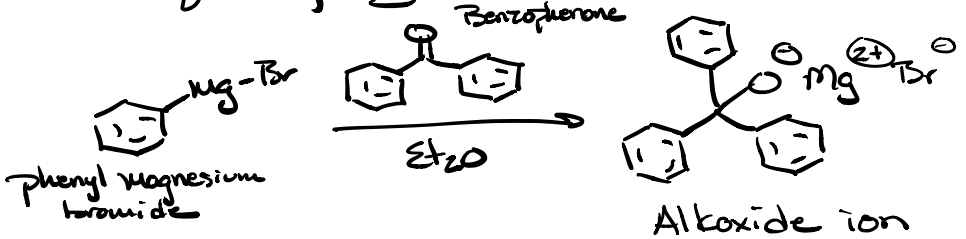
Add ~ 1.09 g solid benzophenone

Add ~ 2.0 mL Et_2O and stir/mix to make homogeneous Solution

⑫ Use Syringe to transfer benzophenone Solution to RB flask

* very Exothermic

- Add quickly but at a rate below boiling



phenyl magnesium bromide

Alkoxide ion

Ionic

"Adduct"

Insoluble in Et_2O

(13) Rinse the conical vial w/ 1mL Et_2O & add to RB flask

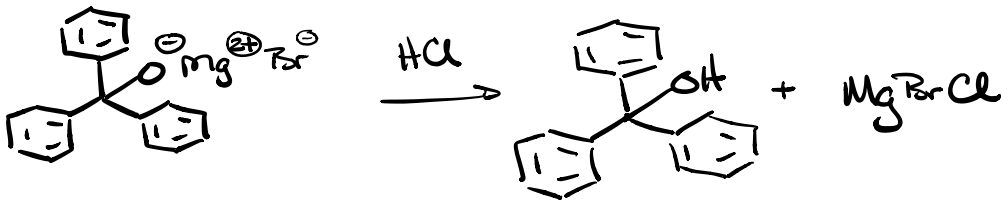
(14) When stirring fails as the solid adduct forms, open the RB flask and stir by hand until the reaction becomes homogeneous grey color.

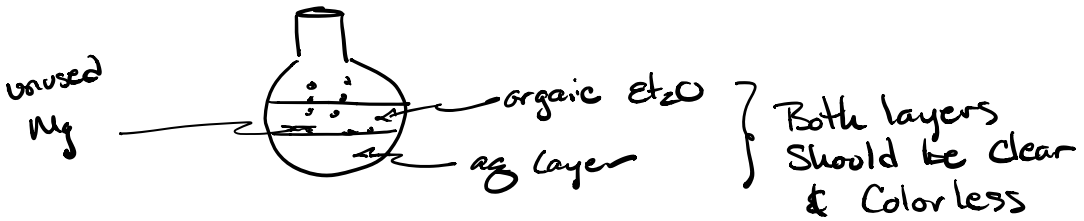
$\Rightarrow$ No longer H_2O Sensitive

Work-Up

(1) Add 6mL 6M HCl to the roundbottom flask *~~Dropwise~~

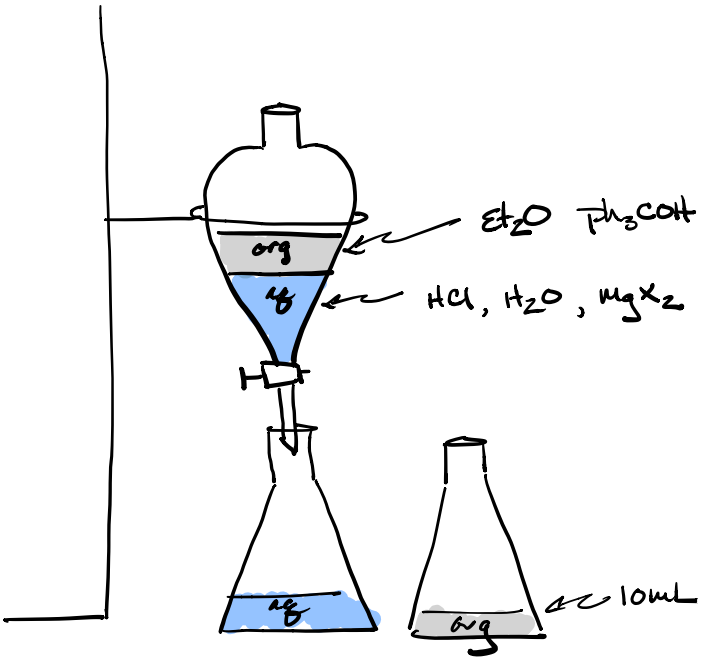
Extremely Exothermic



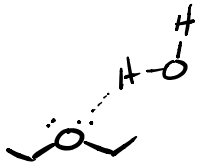


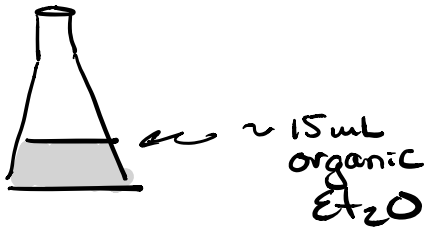
$$Mg + 2HCl \longrightarrow MgCl_2 + H_{2(g)}$$

② Transfer entire reaction contents to a Seperatory funnel & extract the organic layer

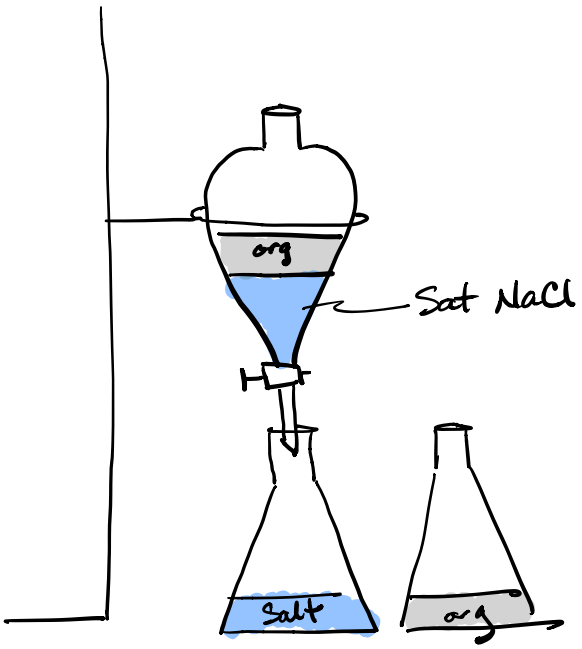


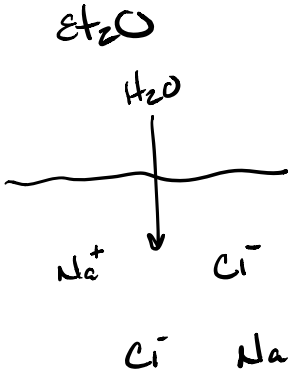
③ Add aq layer back into Seperatory funnel & Re-extract with 5 mL Et_2O
Combine the two organic layers





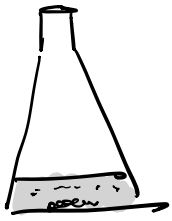
④ Predrying step. Add Et_2O layers back into Sep funnel & Wash w/ 5 mL Brine Solution
Brine is Sat $NaCl_{(aq)}$





⑤ Dry Et_2O w/ $MgSO_{4(s)}$

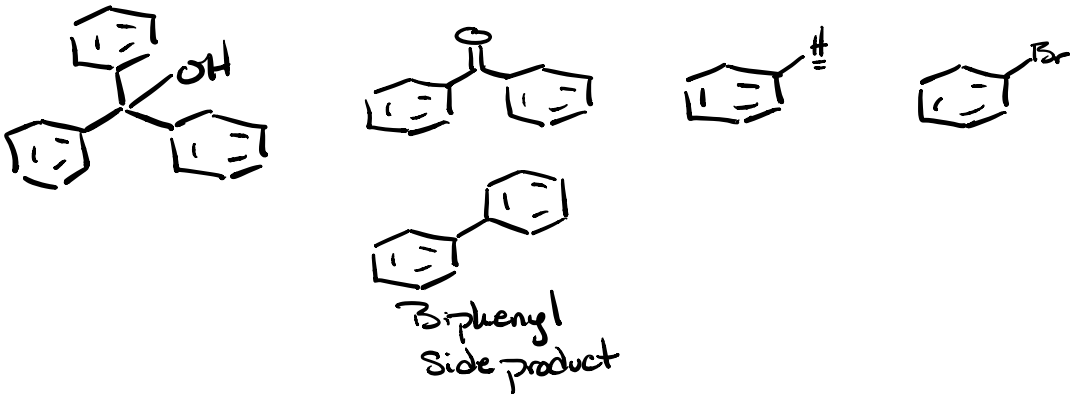
$$MgSO_4 \text{ (powder)} + H_2O \longrightarrow MgSO_4 \cdot 7H_2O_{(s)} \text{ (sticky solid)}$$



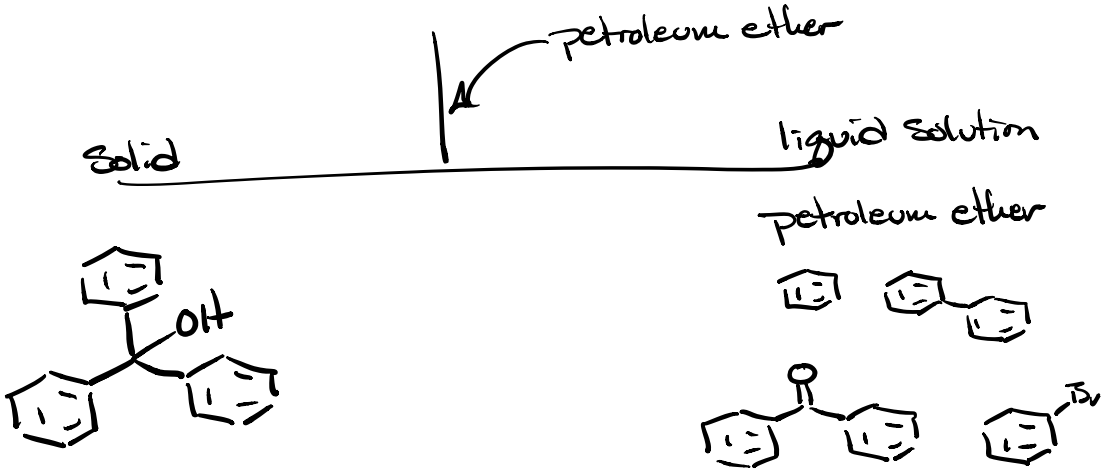
⑥ Decant into clean dry 25-mL RB flask & Rotovap

⑦ Triturate solid residue with 3 mL petroleum ether

Solid Contains:



Petroleum ether mixture of C_4-C_6 hydrocarbons
Completely non-polar



⑧ Filter on hirsch funnel

⑨ weigh Solid

⑩ Recrystallize from isopropanol

⑪ MP, Solid FTIR, final Mass
Calc % yield