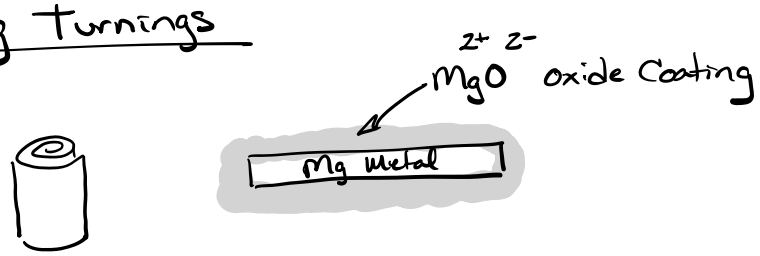
Grignard Experiment
Synthesis of Triphenylmethanol 2nd Day

*2a) Weigh out 0.15g Mg turnings

3) Add mg turnings to Round bottom flask while assymboling the classware.

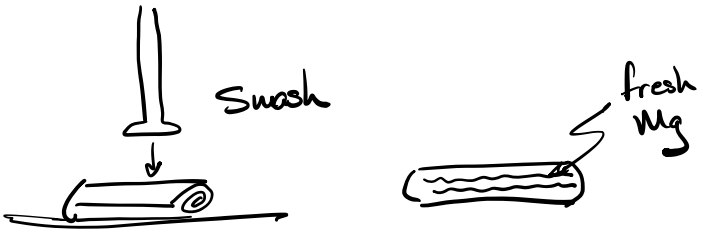
Mg Turnings



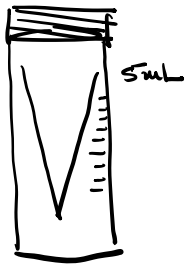
$R\text{-}Br + Mg^{\circ} \rightarrow R^{-}\text{-}Mg^{2+}\text{-}Br^{-}$

Mg must have $2e^{-}$ to give away to make Rxn work

Must Remove oxide Coating for Rxn to work.



④ Use 5 mL Conical Vial



*- Tare Vial

- Add ~ 0.70 mL bromobenzene

*- Reweigh Conical vial to get mass bromobenzene

- Add ~ 4 mL of anhydrous diethyl ether to Conical vial & mix

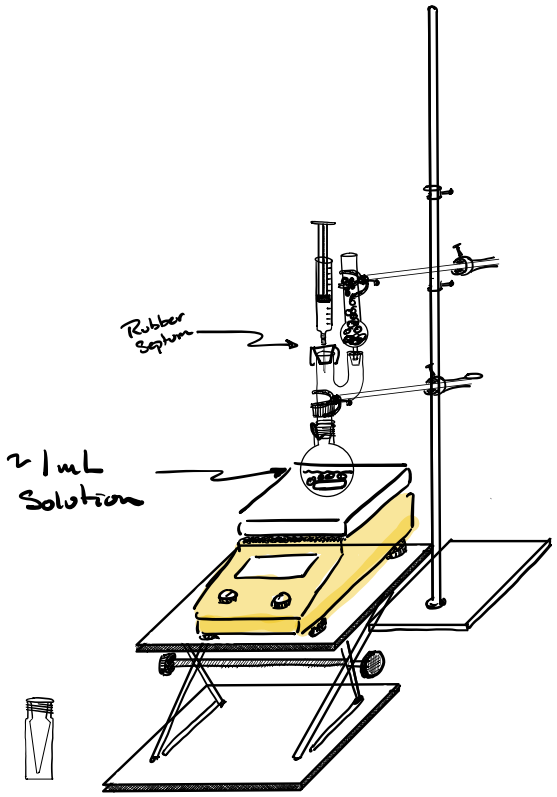
⇒ Solution of bromobenzene in diethyl ether

Density ∝ temp

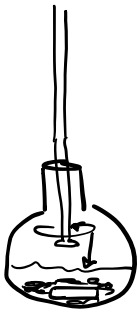
↑ Volume ∝ temp ↑

mass independent of temp

⑤ Use a 3 mL Syringe to add ~ 1 mL of the bromobenzene solution into Round bottom flask through the Septa.



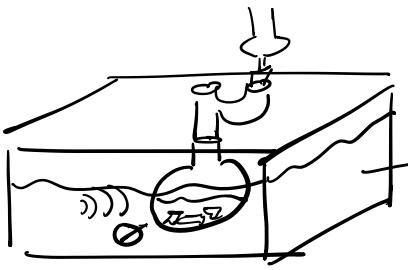
⑥ Call me over to crush Mg turnings



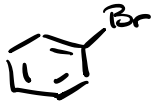
- Grind Mg turnings until solution turns turbid
  ⟹ Rxn started

⑦ Add remaining bromobenzene solution via syring over 15 min.

* Add at a rate that keeps Rxn below boiling



Sonicator set temp & cleans Mg

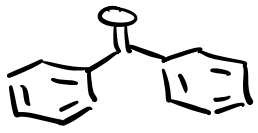


Expensive & Difficult

⑧ Rinse Conical vial w/ 2mL fresh diethyl ether and add to the reaction.

⑨ Use same 5mL Conical vial

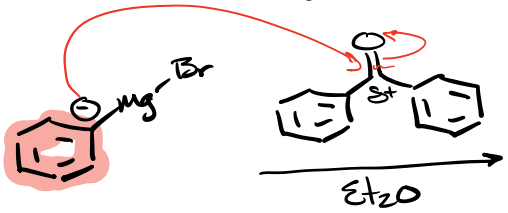
- Tare
- Add ~ 1.09 g solid benzophenone
- Reweigh Conical & benzophenone
- Add ~ 2.0 mL diethyl ether & Mix to make homogeneous

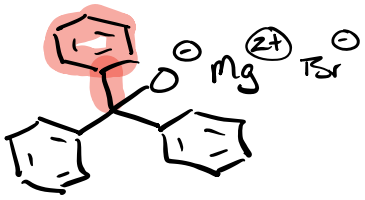


benzophenone

(10) Use same Syringe to transfer benzophenone Solution to RB flask

* very exothermic
* Quickly, but try to keep addition below boiling





Alkoxide anion

Ionic

not soluble in Et_2O

Crashes out as a Solid.

⇒ End of day 1

Safe store as the alkoxide ion

Rxn is over

Start workup & isolation phase

Work-up Day 2 on Rxn

① Add 6mL of 6 M HCl * Dropwise

Extremely Exothermic

6 M 6 M

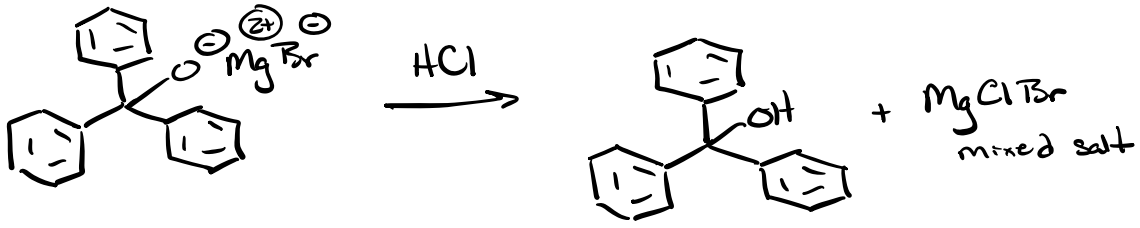
italic

Single underscore _ italic

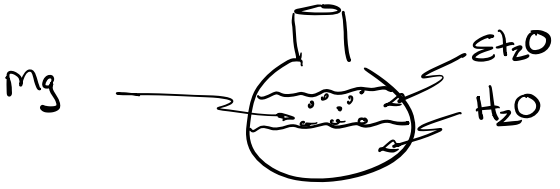
Double underscore = Bold

year page #

JACS 1943, 1701



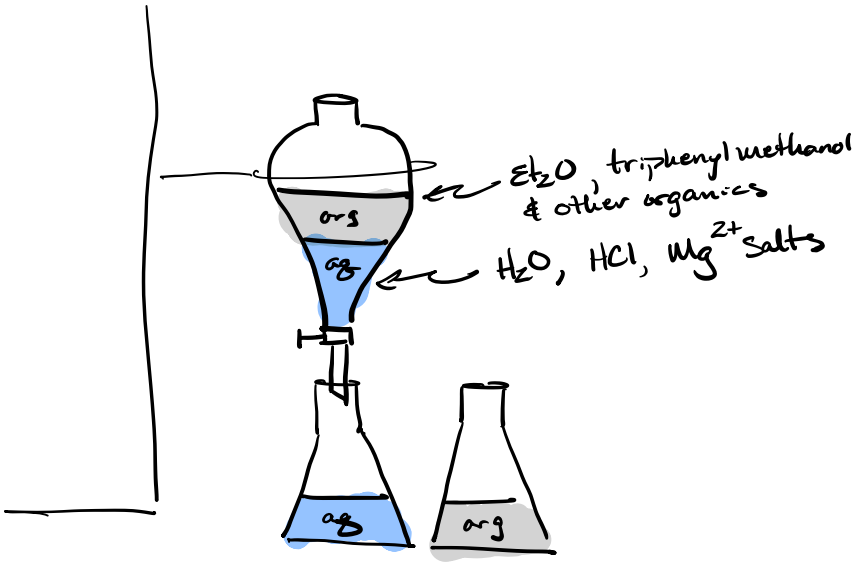
Biphasic System



* Must Continue Stirring until all bubbling Stops & both layer Clear & Colorless

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

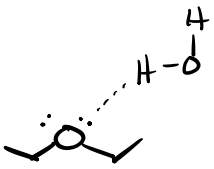
② Transfer the contents to a Seperatory funnel & extract the organic layer.



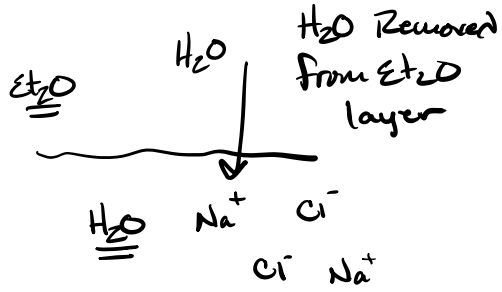
③ Add aq layer back into Seperatory funnel & Re-extract with 5 mL fresh diethyl ether
Combine the organic layers

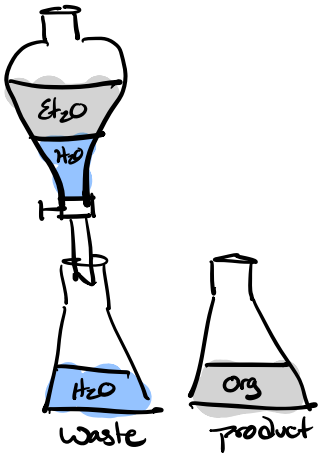
$\Rightarrow$ aq layer is waste.

④ Pre drying Step. Add org layer back into Sep funnel & Add 5 mL brine solution (Sat. $NaCl_{(aq)}$)

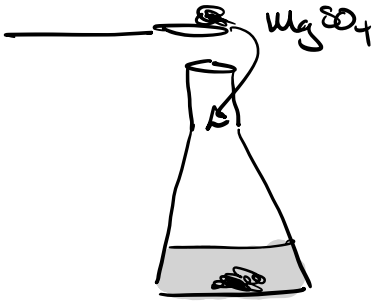


Holds H_2O tightly





⑤ Chemical drying with $MgSO_{4(s)}$



Clumps wet
free flowing dry

$$MgSO_{4(s)} + 7H_2O_{(l)} \rightarrow MgSO_4 \cdot 7H_2O_{(s)}$$

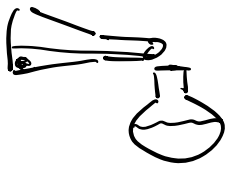
free flowing Solid

Sticky Solid

⑥ Decant into 25mL RB flask

Decant ⇒ pour slowly to leave behind solid.

& Then Rotovap

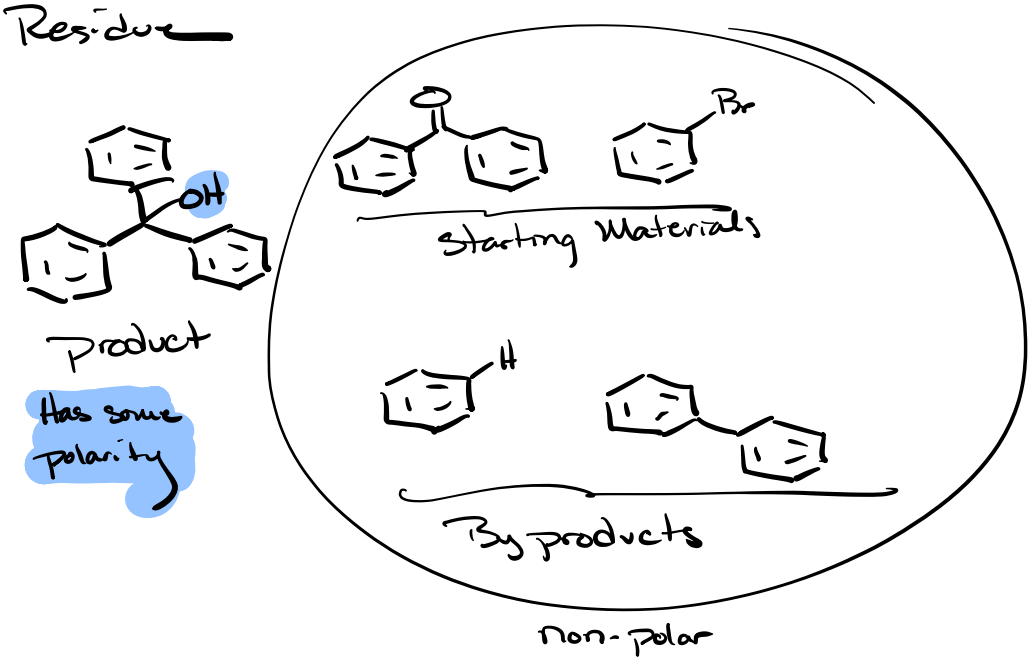


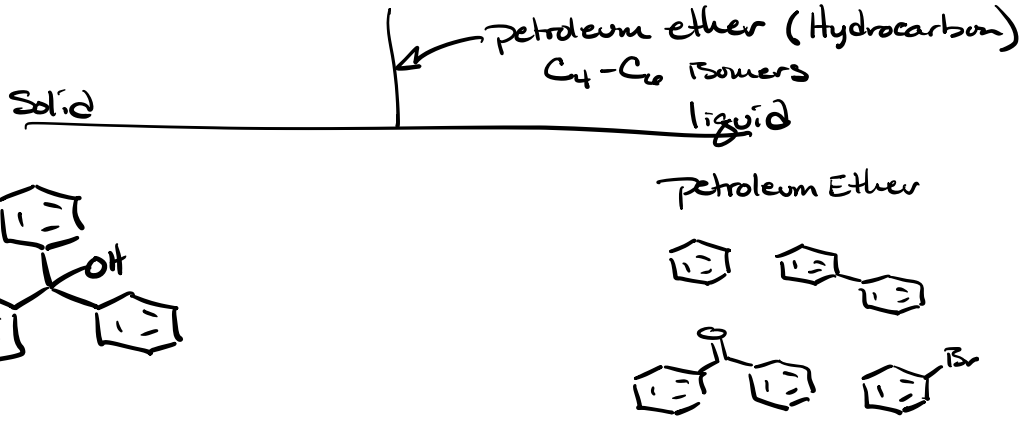
Rotovap

- Heats
- Rotates
- Reduces Pressure

} Increase Evaporation Rate

⑦ After Rotovaping we get a stick solid Residue





⑧ Triturate with petroleum ether
↖ "wash"
warm & mix for a few minutes

⑨ Filter on hirsh funnel

⑩ Weigh the recovered solid

(11) Recrystallize from 2-propanol (isopropanol)

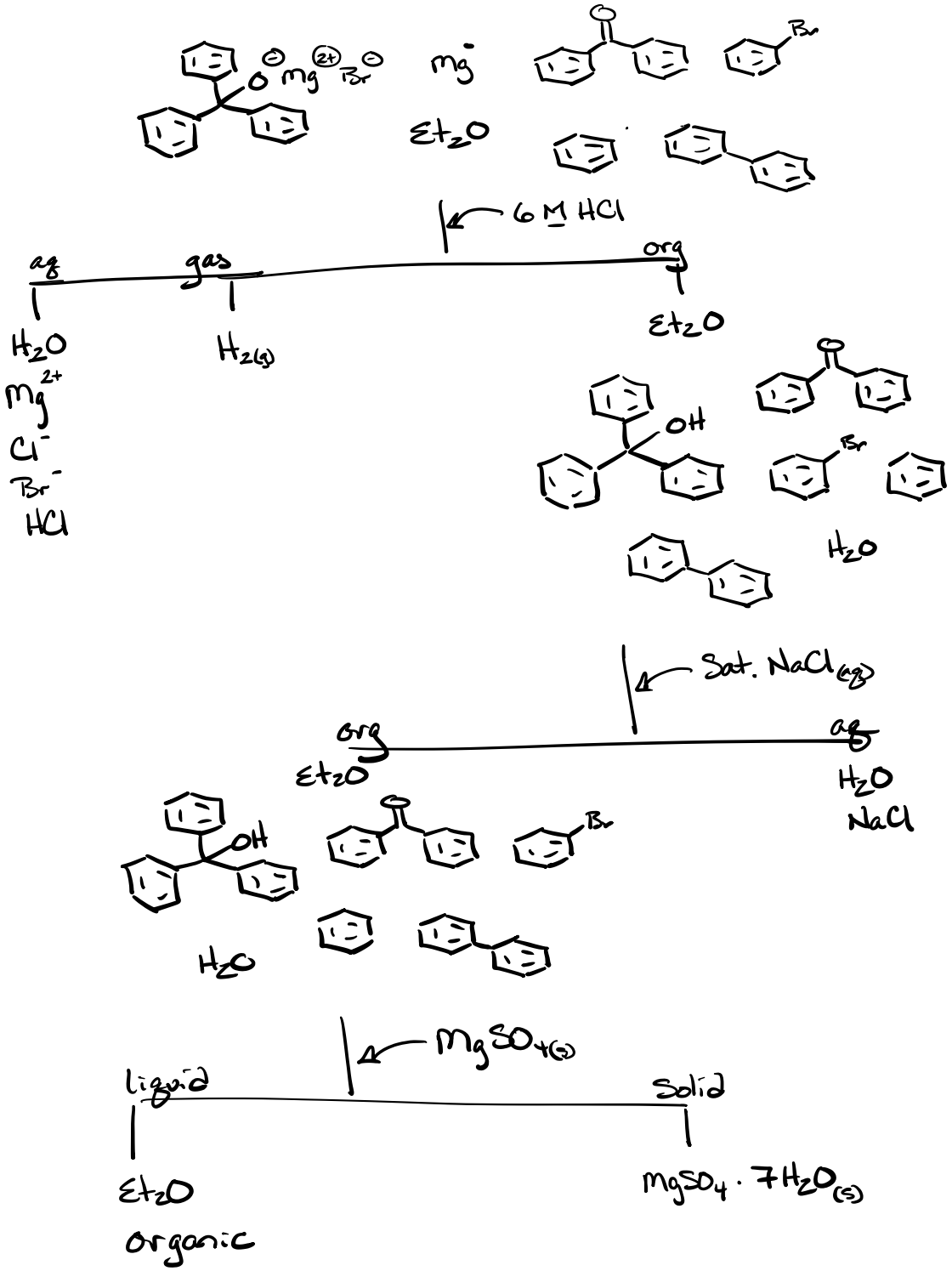
(12) Final mass

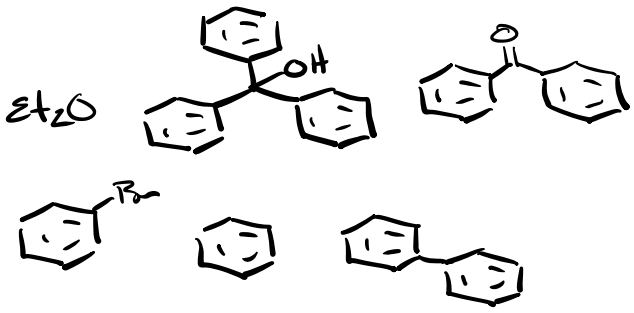
Melting point ← } video

Solid IR ← }

Calc % Yield

Separation Scheme



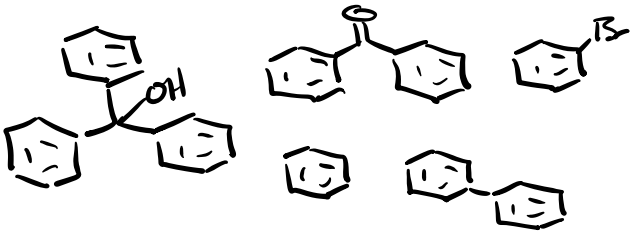


Rotovap (Heat)

Solid Residue

gas (liquid)

Et_2O

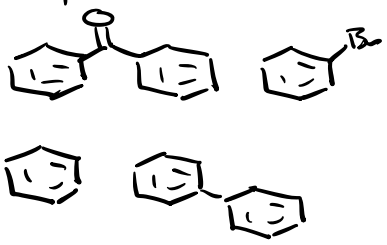


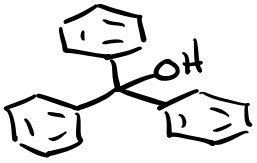
Petroleum Ether (trituration)

liquid

Petroleum Ether

Solid





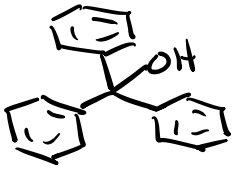
Impurities

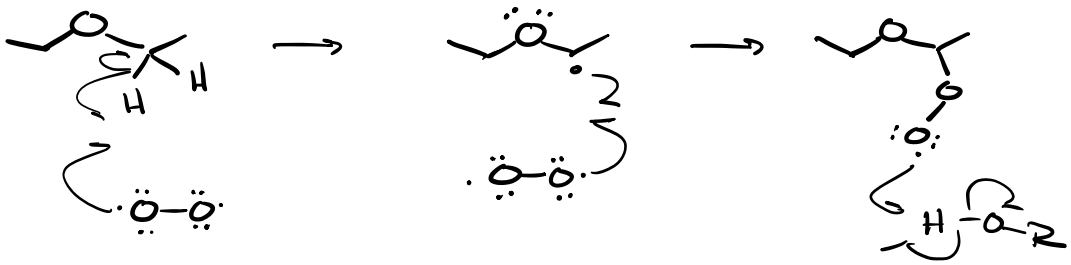
2-propanol (Recrystallization)

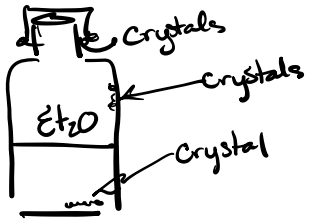
liquid

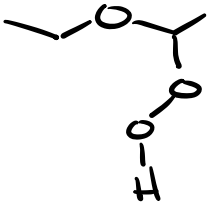
Solid

2-propanol impurities









peroxide explosive