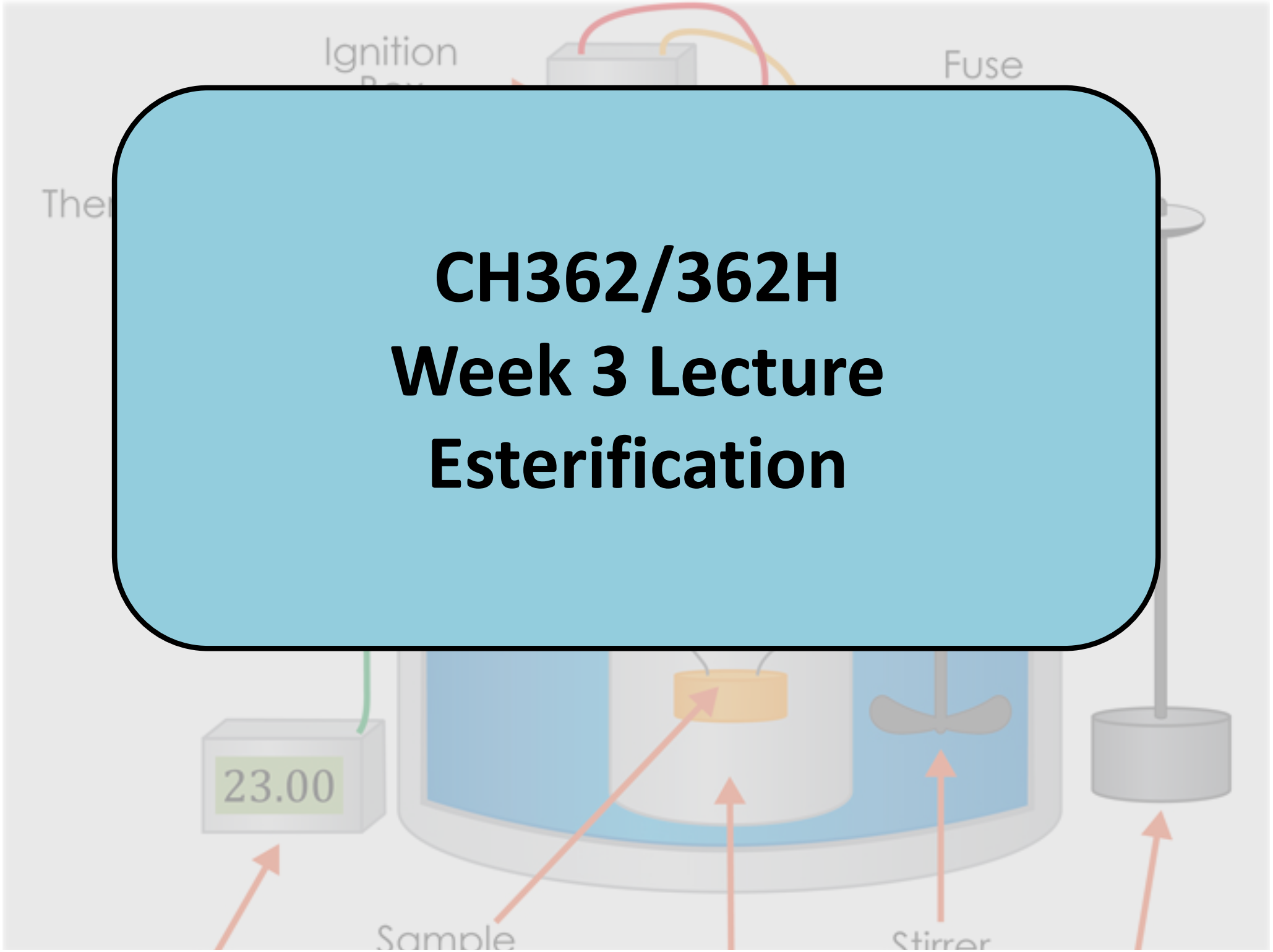
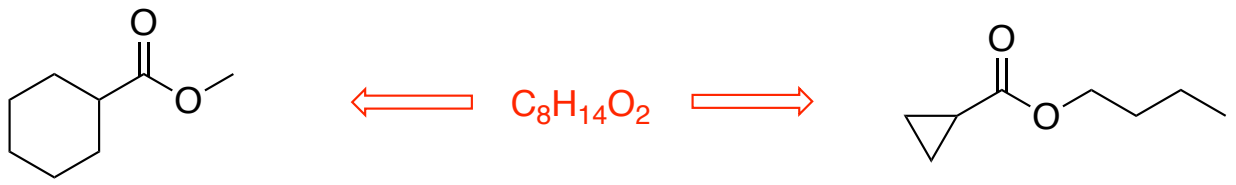


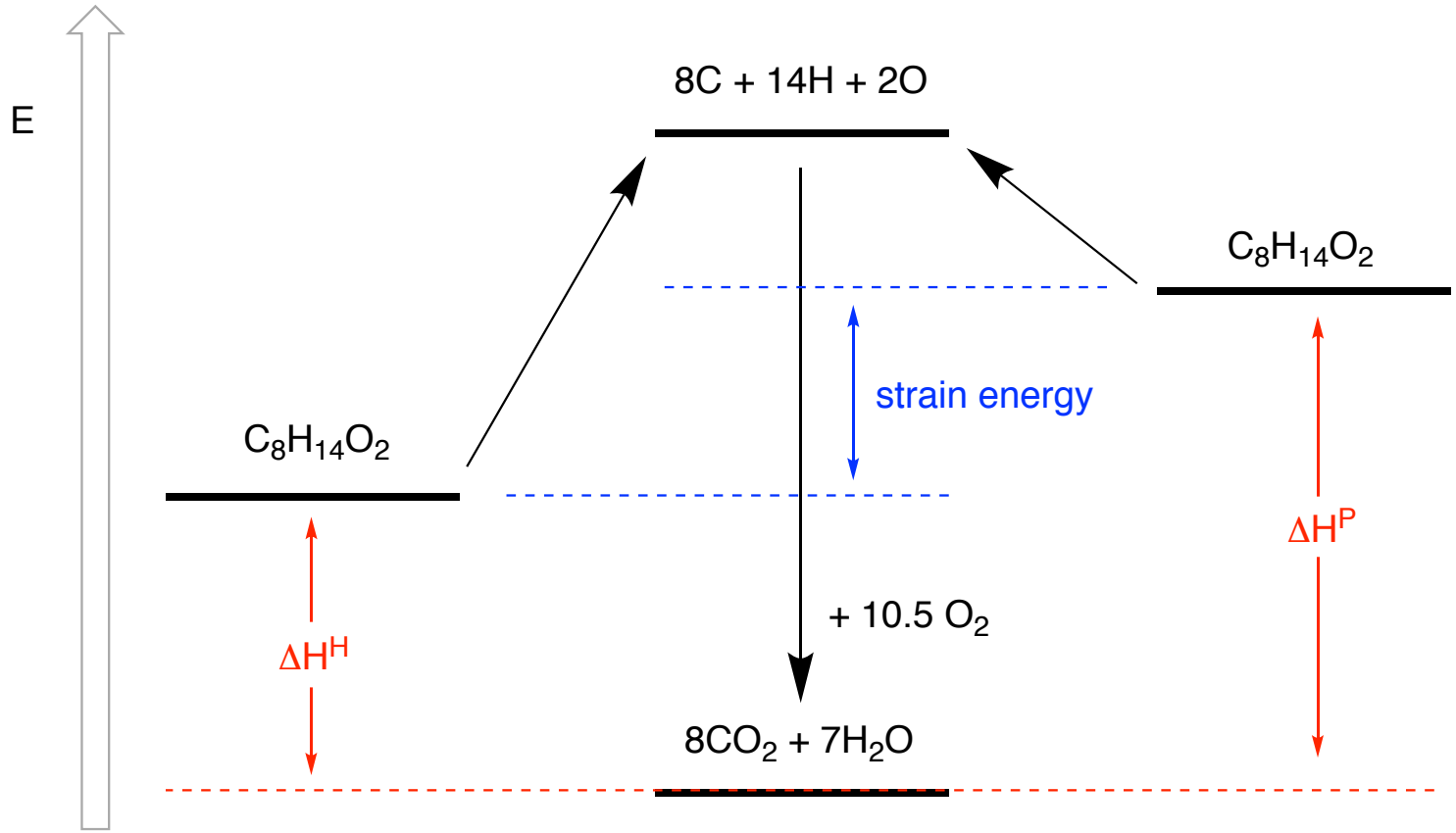
CH362/362H
Week 3 Lecture
Esterification

Project 1 Overview: Strain Energy of the Cyclopropane Ring from Heats of Combustion

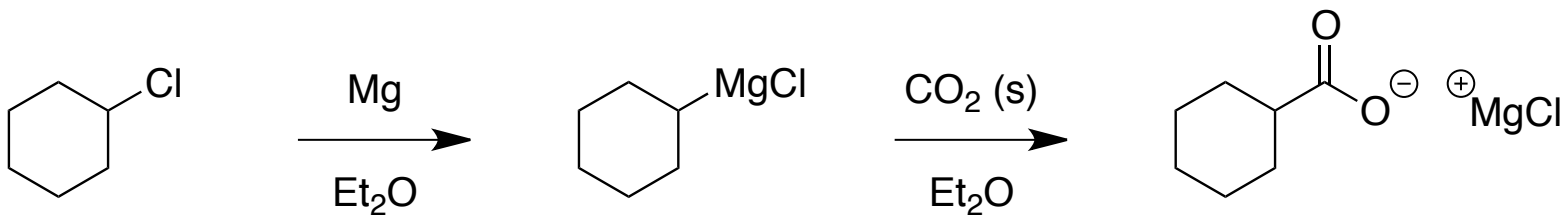


methyl cyclohexanecarboxylate
"H-ester"

butyl cyclopropanecarboxylate
"P-ester"



Synthesis of Methyl Cyclohexanecarboxylate (the "H-ester")

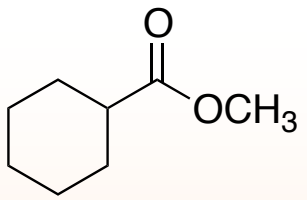


chlorocyclohexane

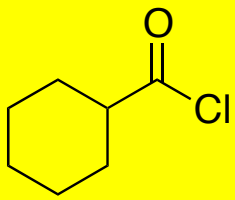
cyclohexylmagnesium chloride (Grignard reagent)

magnesium carboxylate salt

H_3O^+

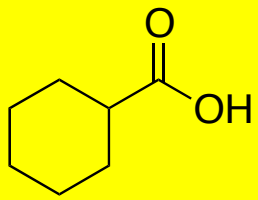


CH_3OH



$SOCl_2$

DMF



- **use at most 11.5 g (90 mmol)**
- ***save 0.50 g for analysis***

methyl cyclohexane-carboxylate "H-ester"

acid chloride

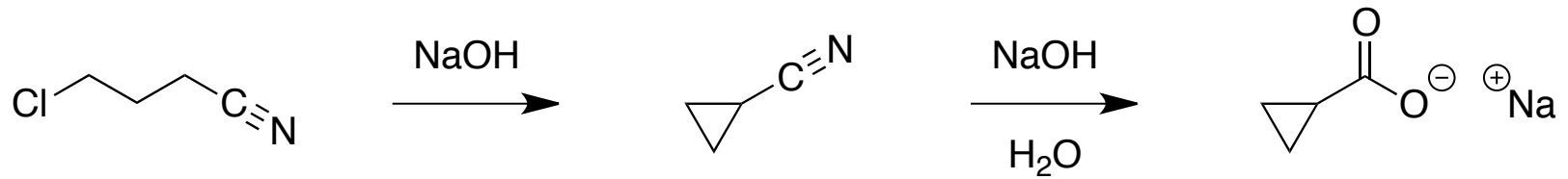
cyclohexanecarboxylic acid "H-acid"

purify by vacuum distillation characterize by IR spectroscopy, bp/pressure range, GC, and refractometry

synthetic activities for wk3

purify by vacuum distillation characterize by IR spectroscopy and bp/pressure range

Synthesis of Butyl Cyclopropanecarboxylate (the "P-ester")

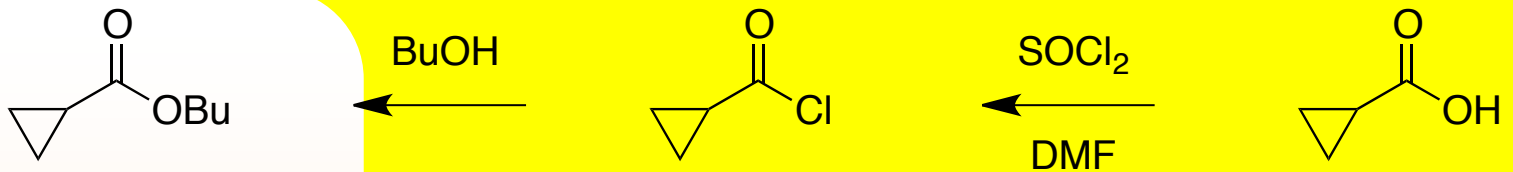


4-chlorobutyronitrile

cyanocyclopropane

sodium carboxylate salt

H_3O^+



- **use at most 14.2 g (200 mmol)**
- ***save 0.50 g for analysis***

butyl cyclopropane-carboxylate "P-ester"

acid chloride

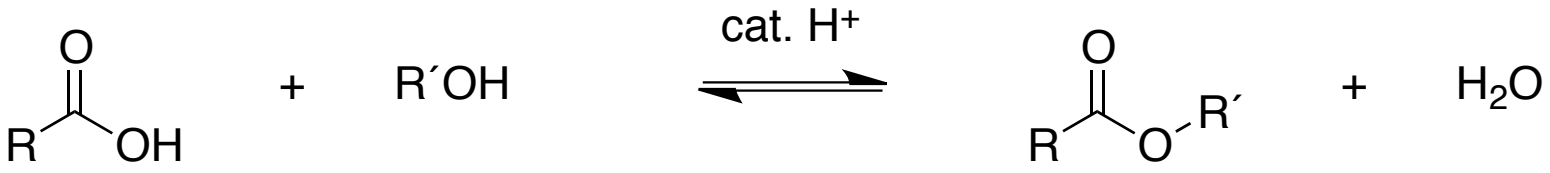
cyclopropanecarboxylic acid "P-acid"

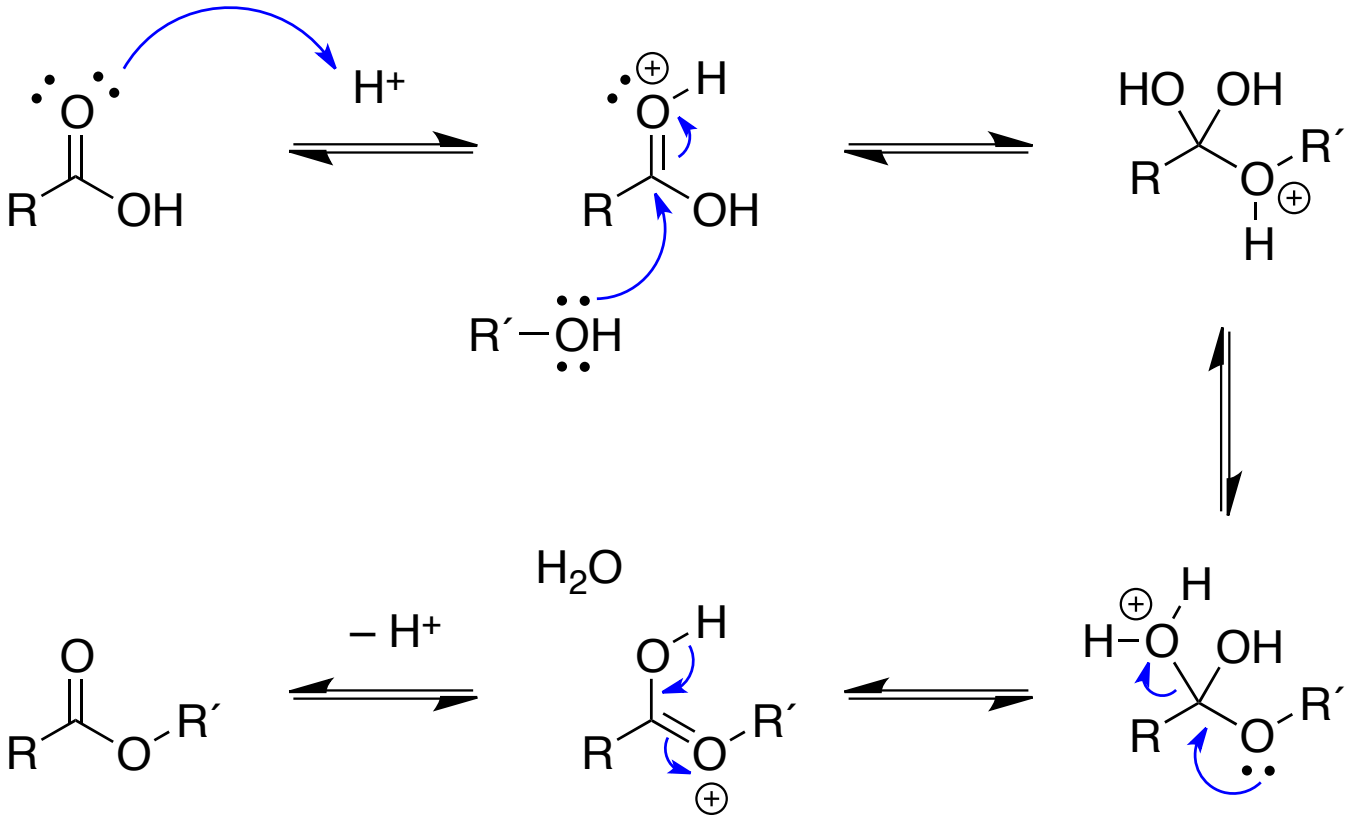
purify by vacuum distillation characterize by IR spectroscopy, bp/pressure range, GC, and refractometry

synthetic activities for wk3

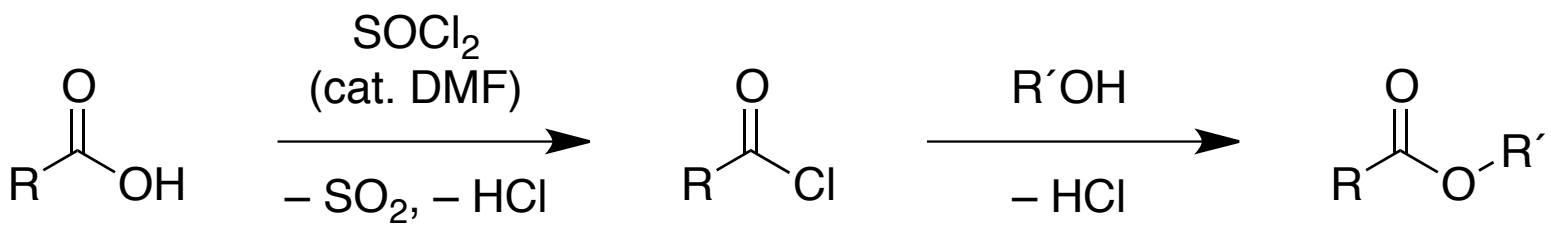
purify by vacuum distillation characterize by IR spectroscopy and bp/pressure range

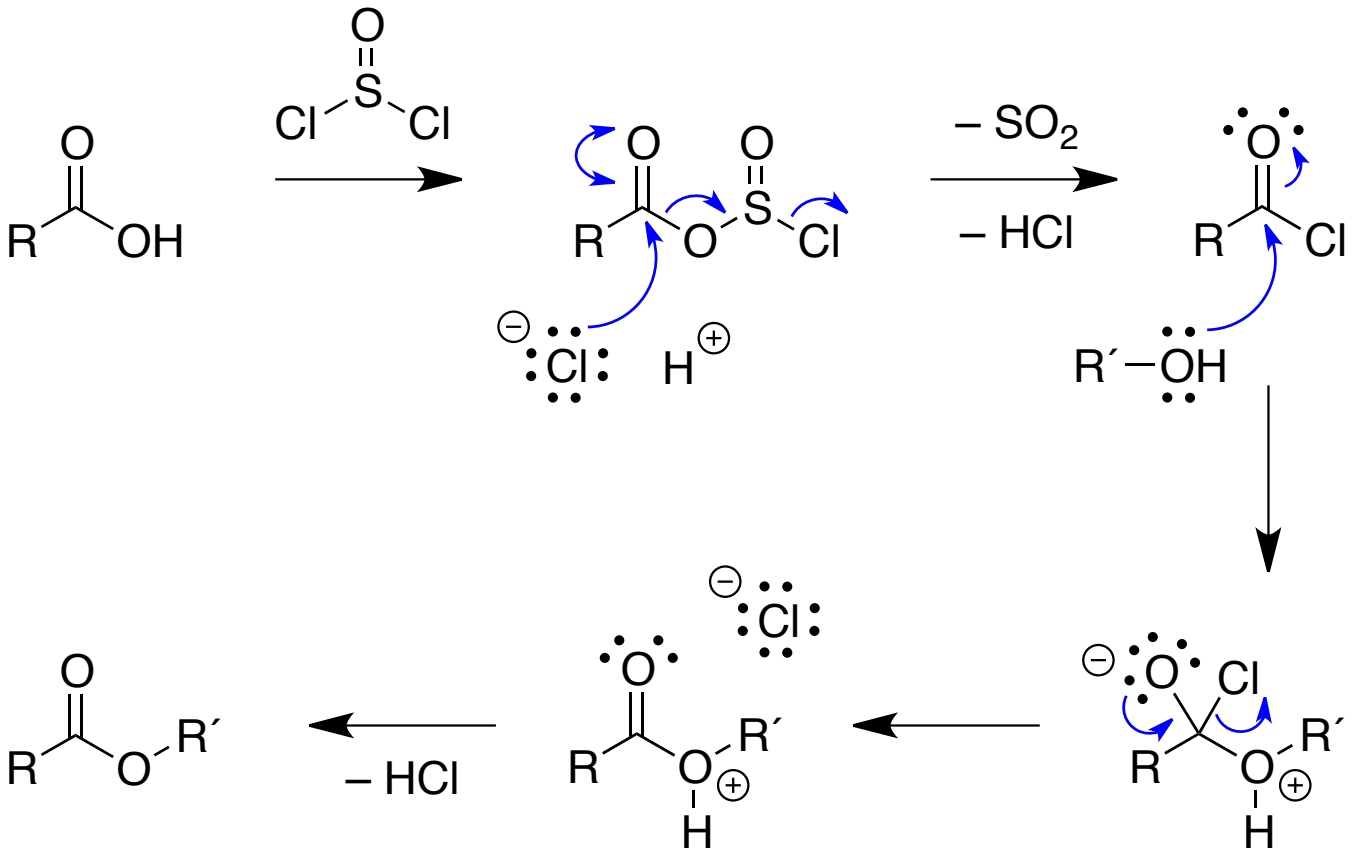
Esterification of Carboxylic Acids: (1) The Fisher Esterification (Reversible)





Esterification of Carboxylic Acids: (2) via Acid Chloride (Irreversible)



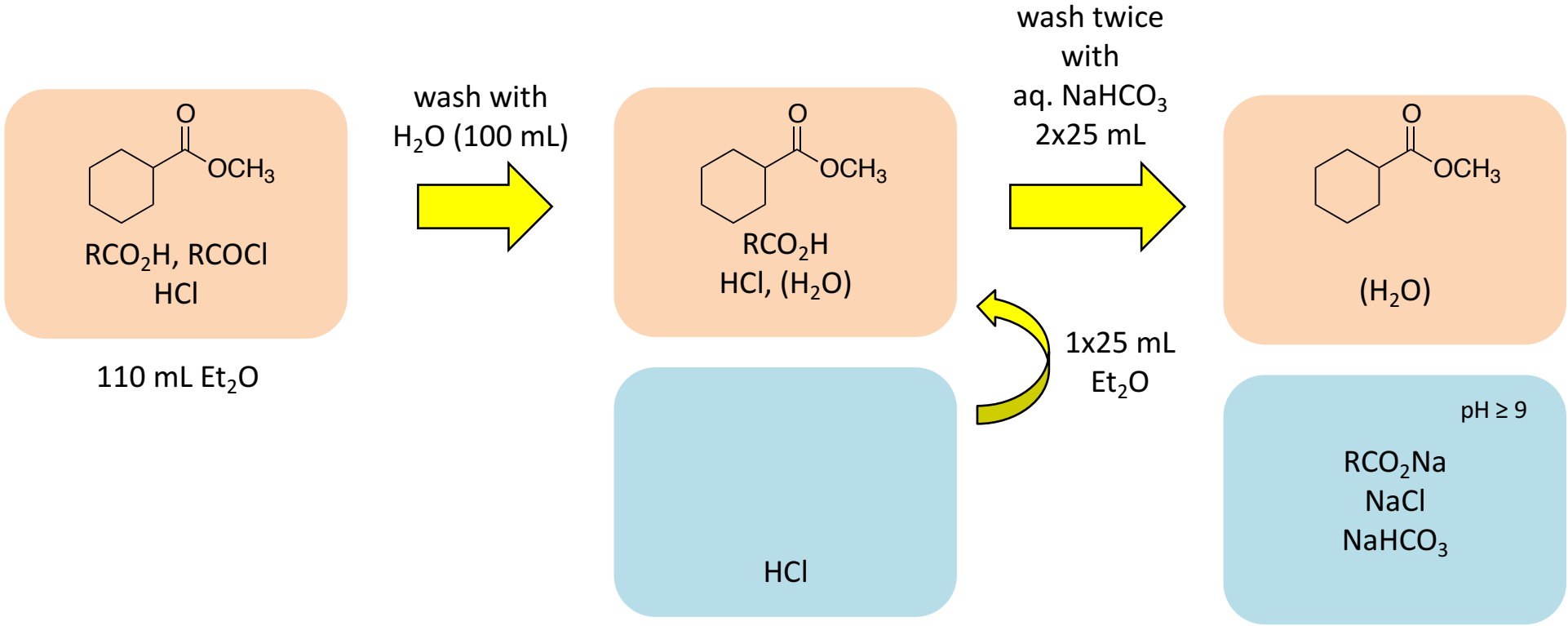


Summary of Work-up for H-Ester and P-Ester

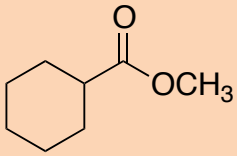
( ) = low solubility

= ethereal layer

= aqueous layer



Summary of Work-up for H-Ester and P-Ester



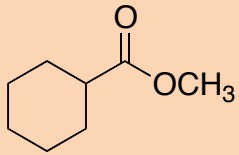
(H_2O)

pH ≥ 9

RCO_2Na
NaCl
$NaHCO_3$

wash with
sat. aq. NaCl
1x50 mL

stopping point for today (Wed.)



- dry over $MgSO_4$

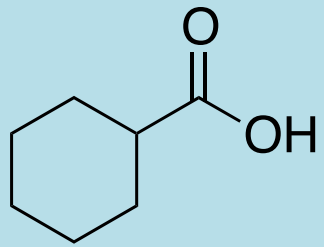
NaCl

- remove Et_2O with rotary evaporator
- vacuum distillation

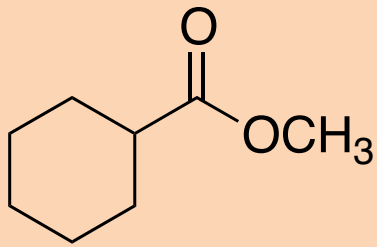
Friday's activity

Considerations for Vacuum Distillation (see CH362 website)

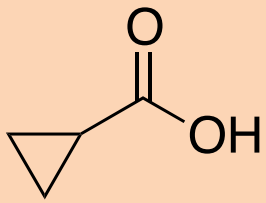
- **ALL (H & P groups) in GBAD 409**



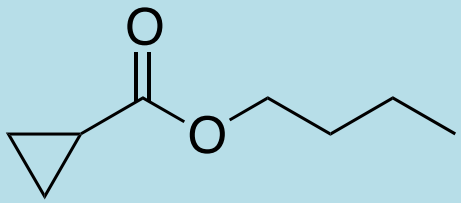
- bp (760 mmHg) = 232-233 °C



- bp (760 mmHg) = 119 °C



- bp (760 mmHg) = 182-184 °C

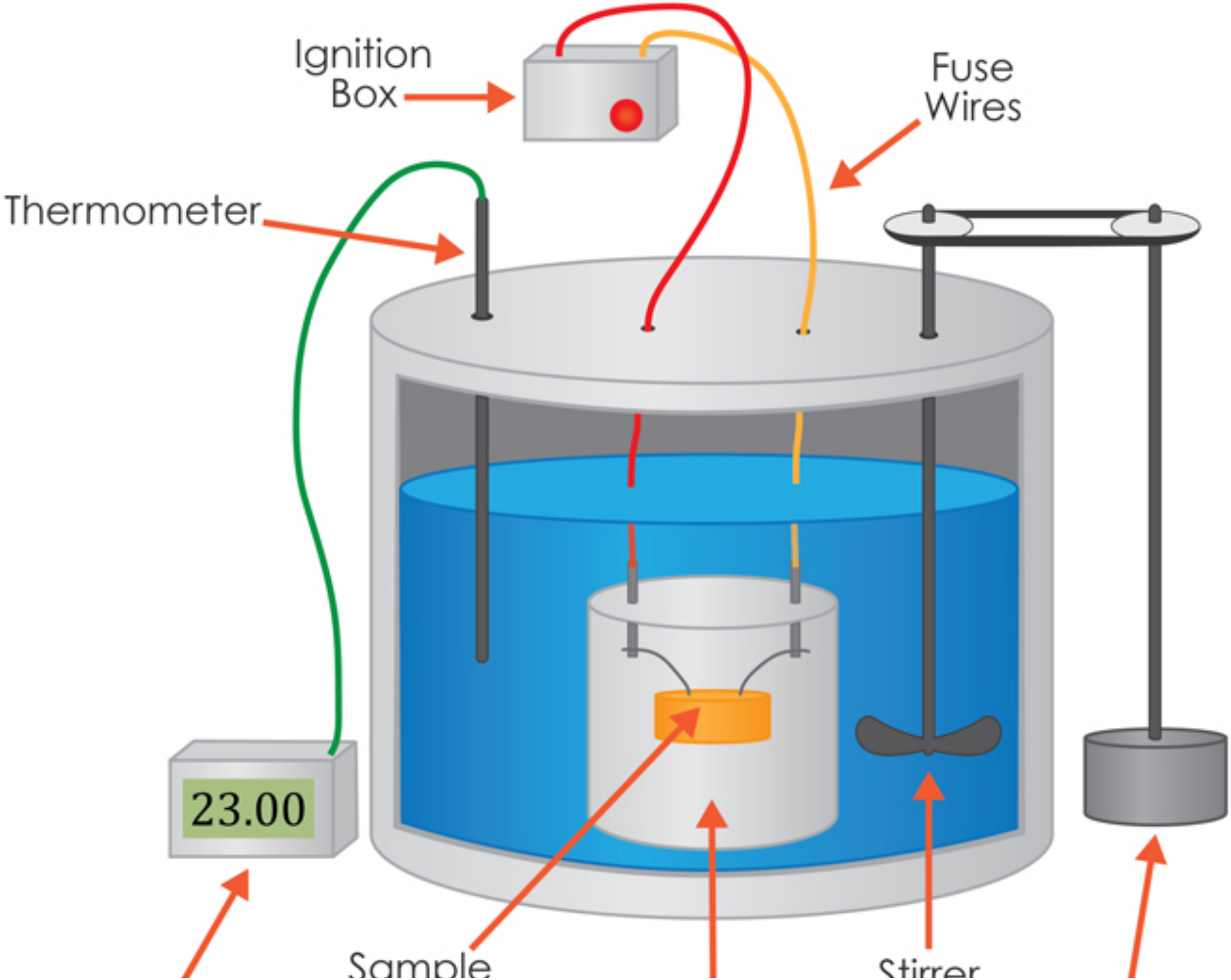


- bp (760 mmHg) = 177 °C

BuOH
- bp (760 mmHg) = 117 °C

= distill with rotary vane mechanical oil pump

= distill with water aspirator pump


Ignition
Box
Fuse
Wires
Thermometer
23.00
Sample
Stirrer