

Week 7-9
Carboxylic Acids

Preparation of Methyl 3-Nitrobenzoate

Preparation of 3-Nitrobenzoic Acid

Preparation of 3-Nitrobenzamide

Week 9

Preparation of 3-Nitrobenzamide

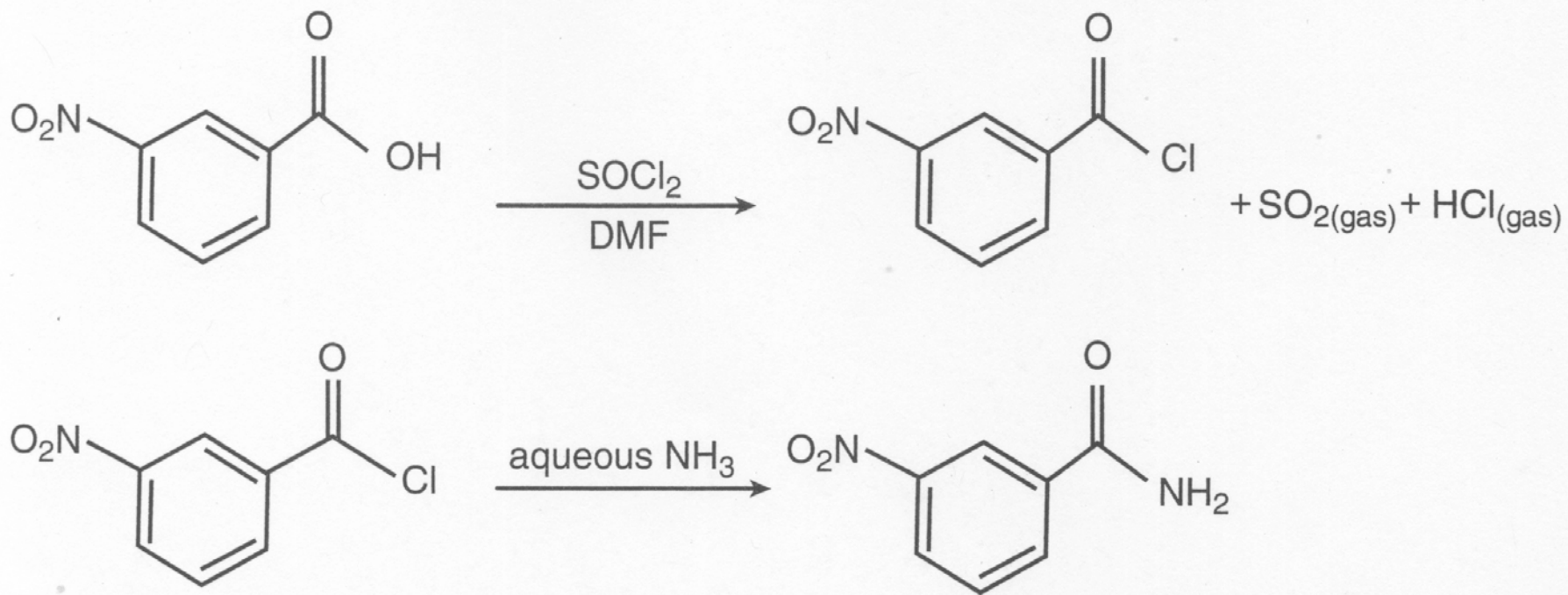
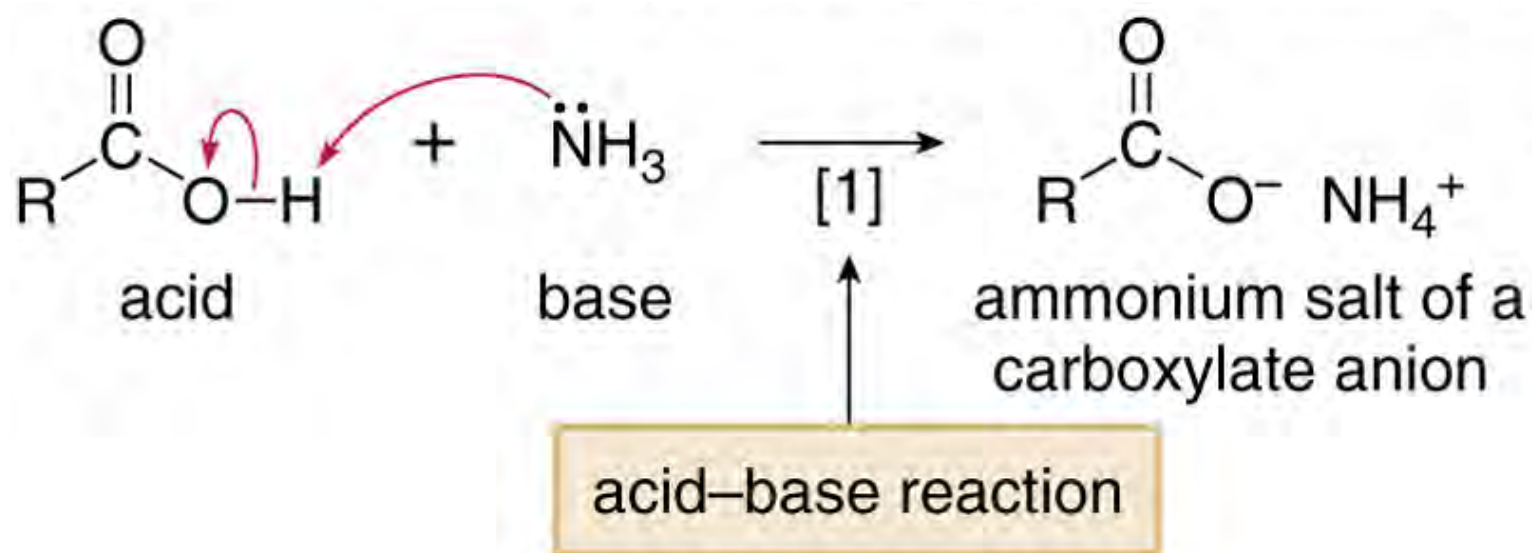


Figure 8.5. Preparation of an amide from a carboxylic acid.

Why Going Through Acyl Chloride?

Acid - Base Chemistry

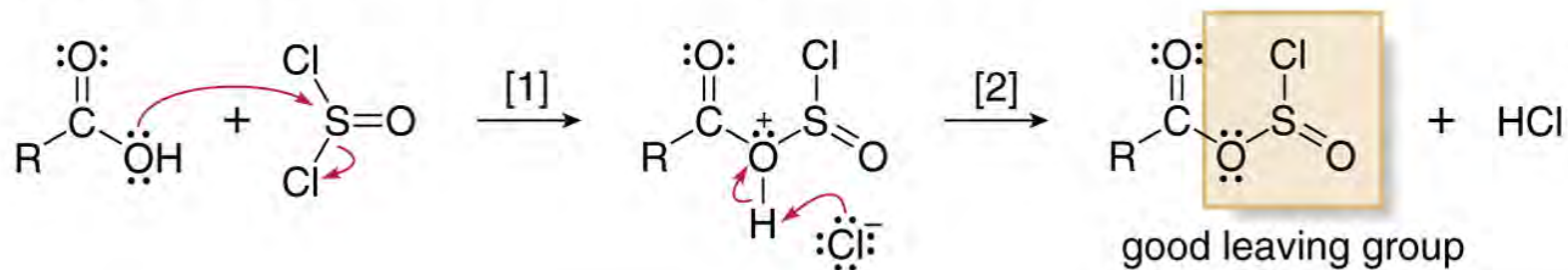


Mechanism of Acyl Chloride Formation

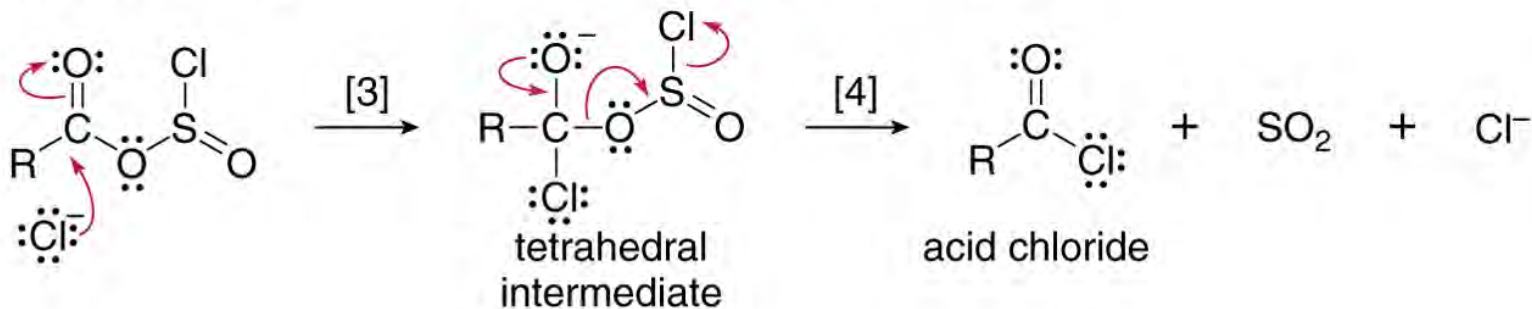


Mechanism 22.5 Conversion of Carboxylic Acids to Acid Chlorides

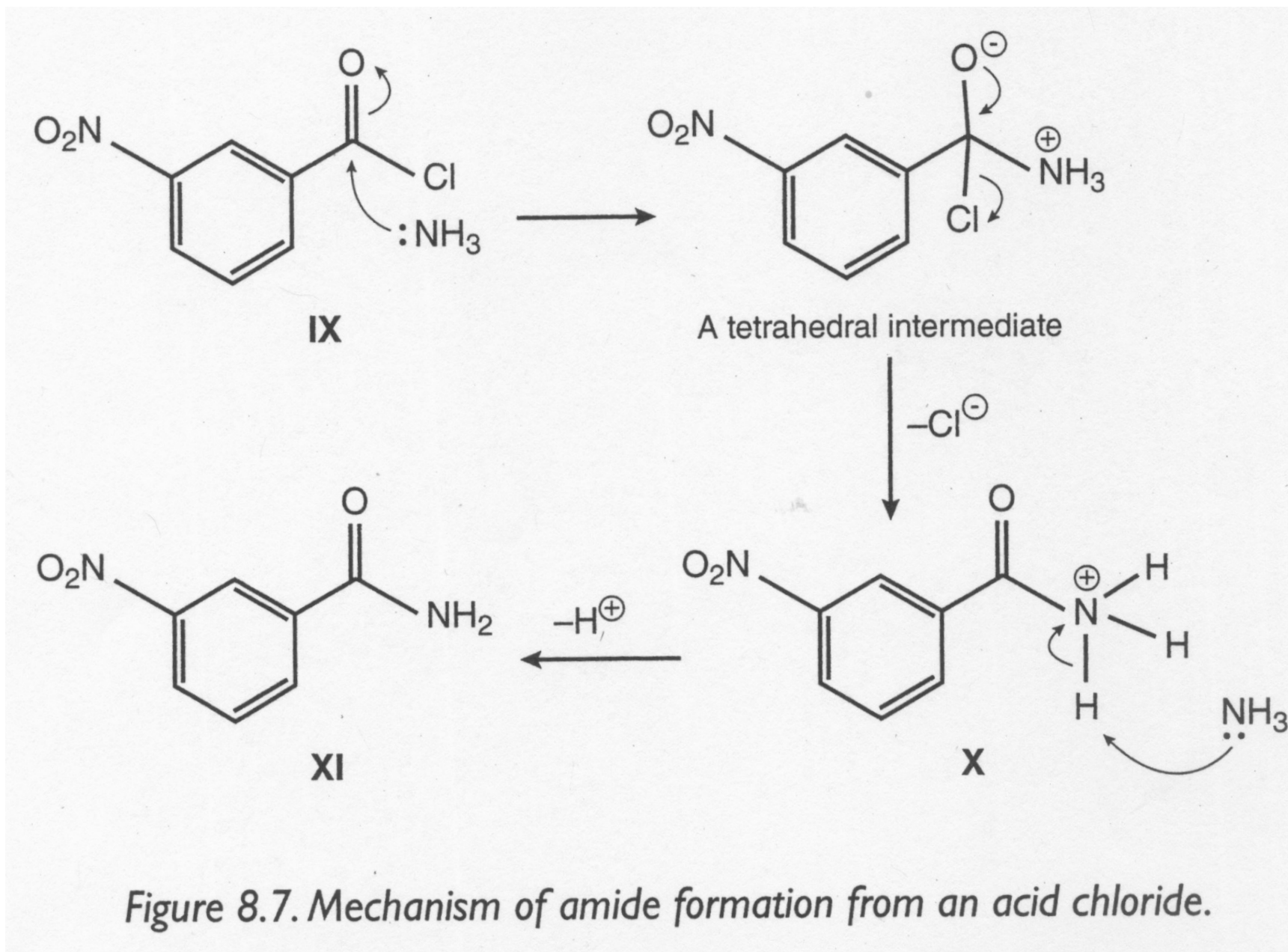
Steps [1] and [2] Conversion of the OH group into a good leaving group



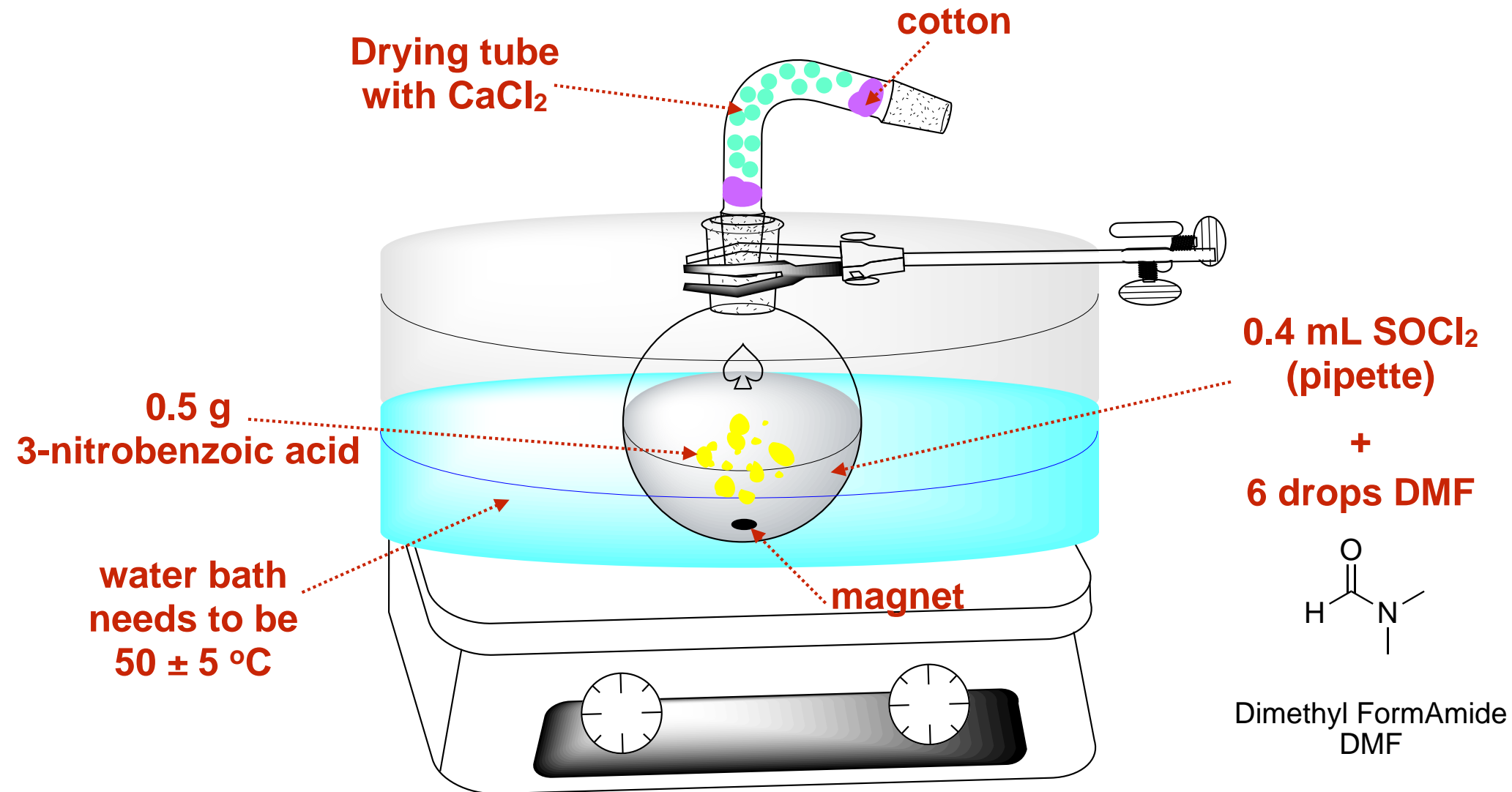
Steps [3] and [4] Substitution of the leaving group by Cl



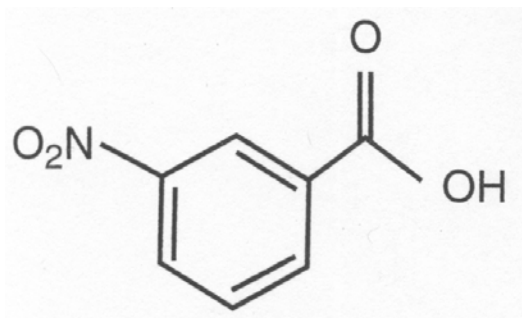
Mechanism of Amide Formation



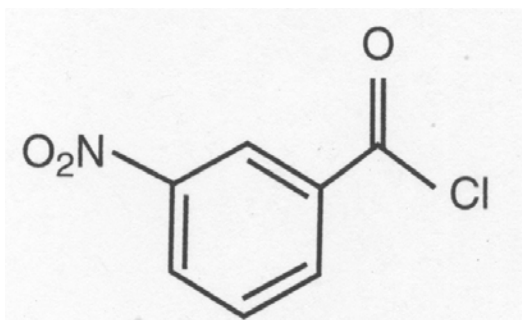
1st Step: Formation of Acyl Chloride



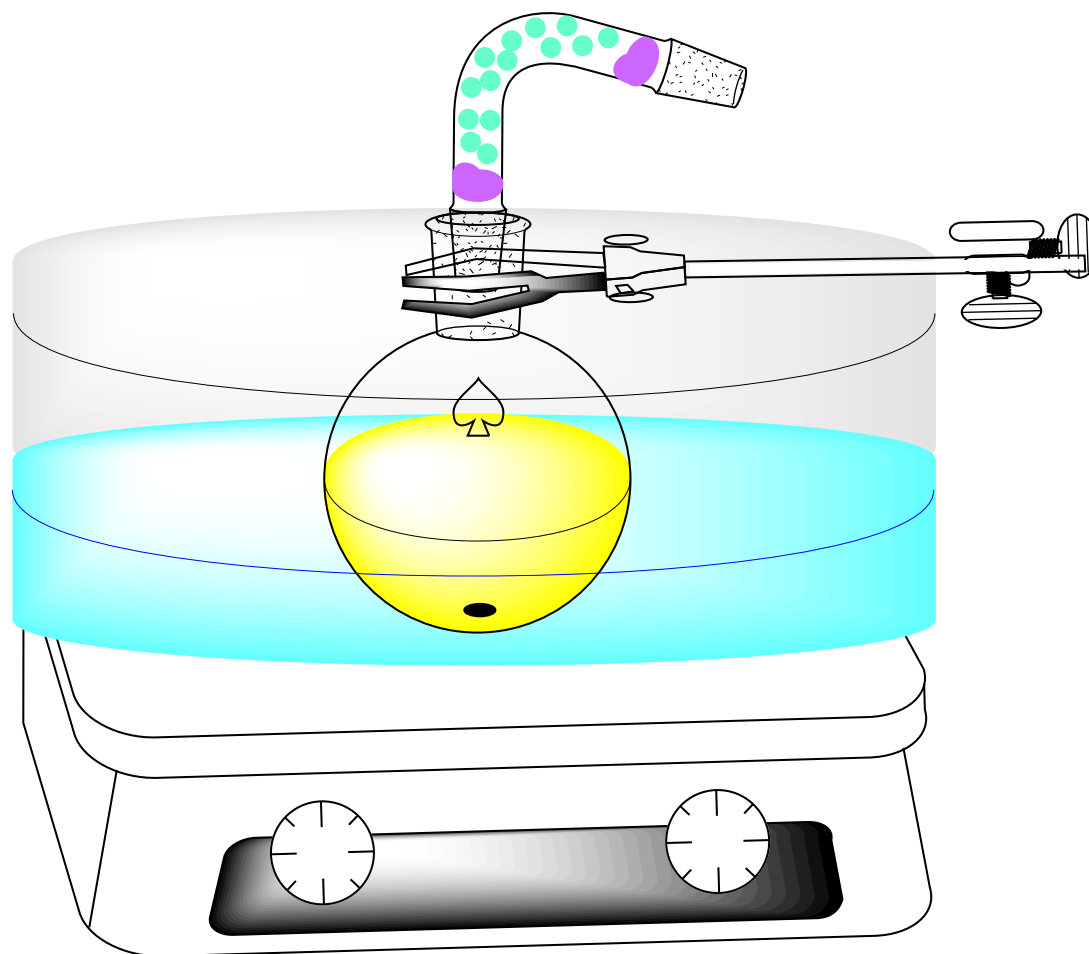
1st Step: Formation of Acyl Chloride



mp = 135 °C

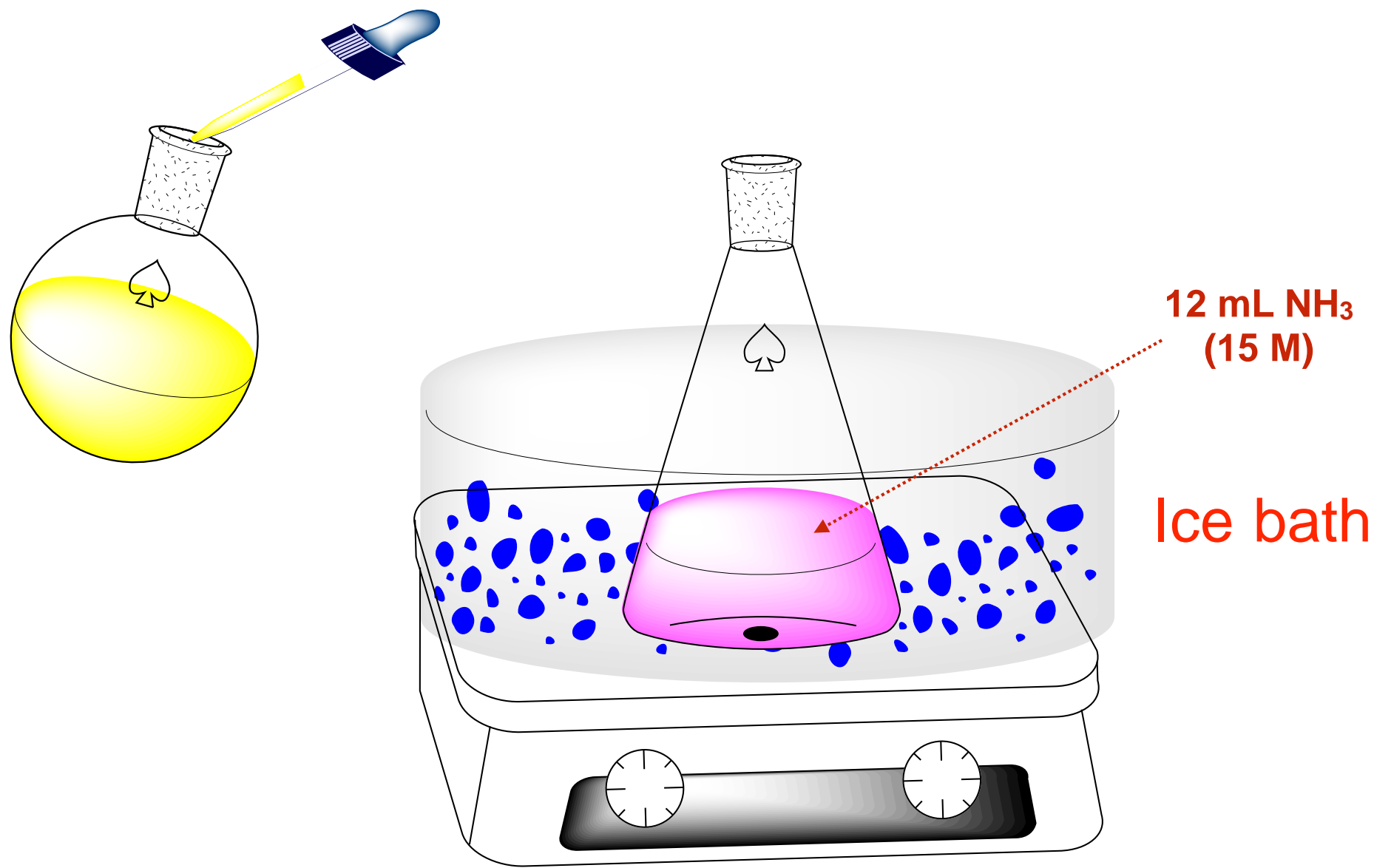


mp = 75 °C

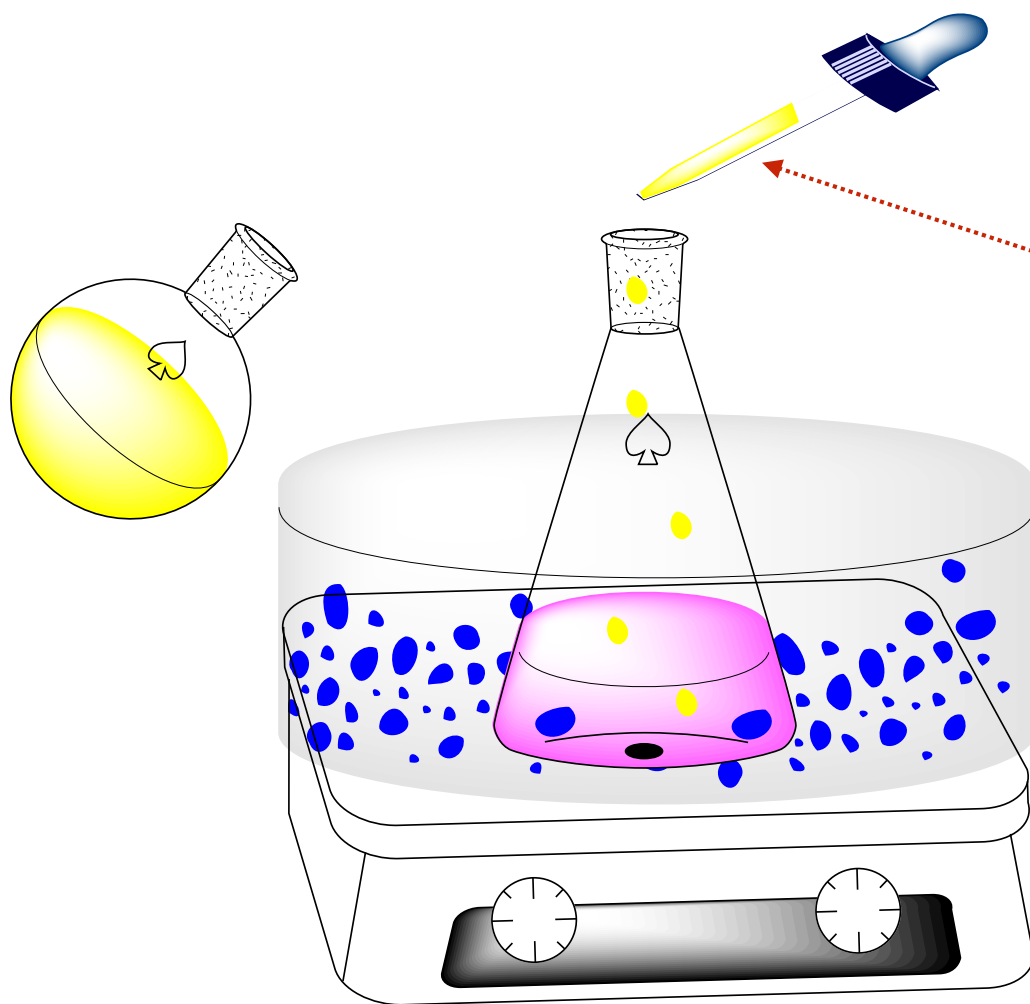


- Heat for 10 min, until everything has dissolved
- If after 10 min there is still solid add extra 0.2 mL SOCl_2
- Continue heating/stirring until no solid remains

2nd Step: Formation of Amide



2nd Step: Formation of Amide



**Add the reaction mixture
drop wise, slowly!
(1 drop / 2 sec)**

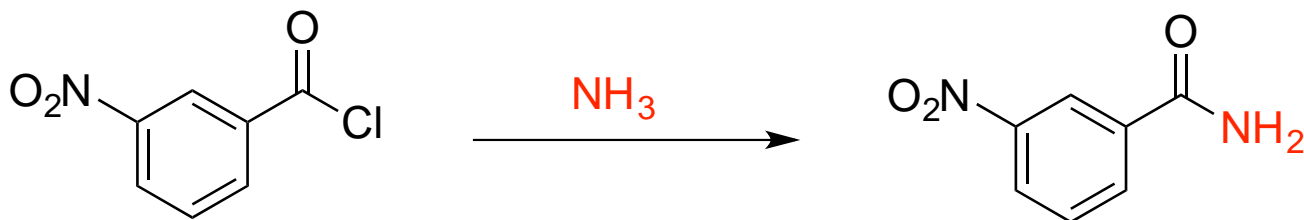
- **Ammonia NEEDS to be cold!
Keeping 1-2 pieces of ice in
the ammonia solution during
the addition will help to keep
the temperature close to 0 °C**

2nd Step: Formation of Amide

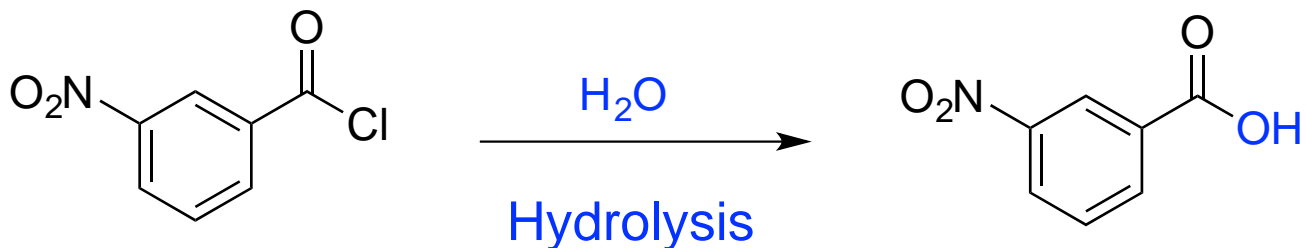
What if you do a quick addition ?



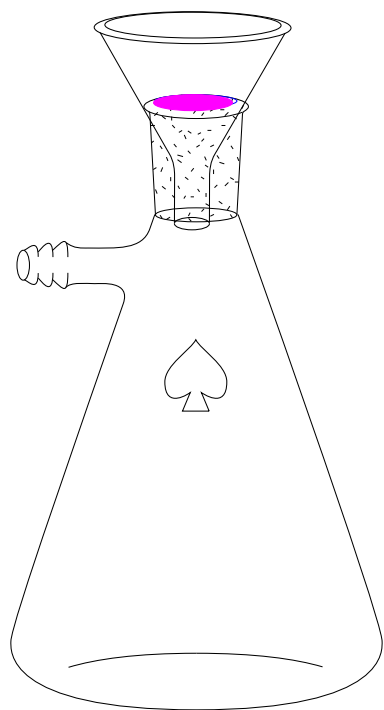
- At low temperature, NH₃ reacts faster than H₂O:



- If the temperature is higher, the H₂O, which is in excess will react too!!!



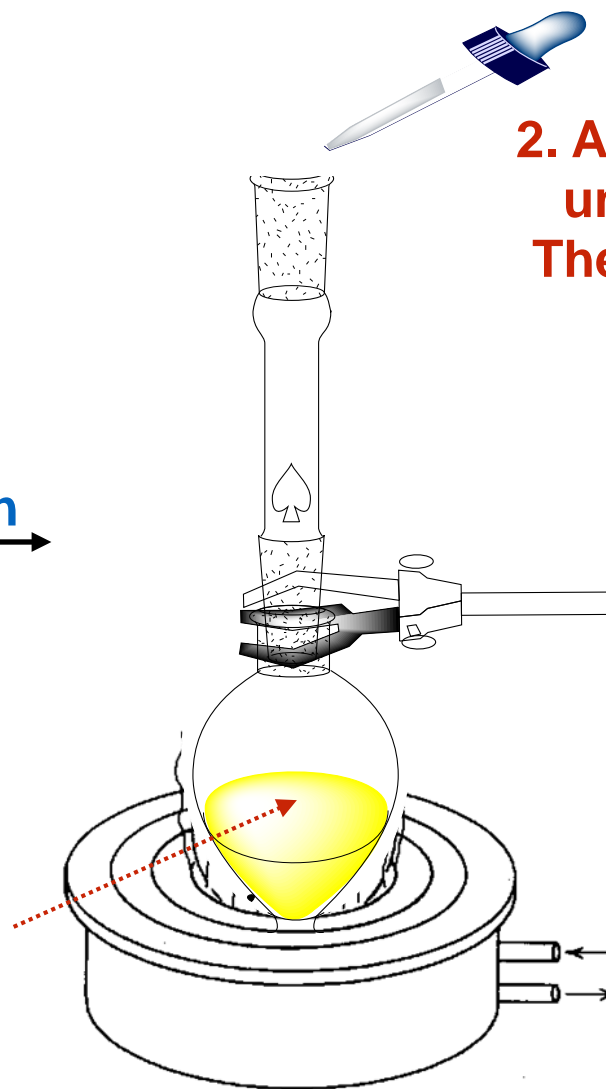
3rd Step: Amide recrystallization



Collect amide with suction filtration

Wash several times with water to remove NH_4Cl

Recrystallization using Ethanol / water



1. Dissolve product in minimum amount of EtOH

2. Add water drop wise until it gets cloudy. Then add one drop of EtOH

Cool down
Collect crystals

Report all yields (weeks 7-9) and melting points!

Submit all products in properly labeled vials for full credit.

The glassware for next week's experiment must be completely dry. Clean the following equipment and place them in a labeled 250 mL beaker and dry them thoroughly in an oven for 30 minutes. Make sure to take the hot glassware out of the oven and place them **in** your drawer before exiting from the lab; you may risk losing them forever. The estimated replacement cost is approximately \$100.

8 mL vial, 5 mL vial, Claisen adapter, air condenser, drying tube, glass stirring rod, and triangular stir bar.