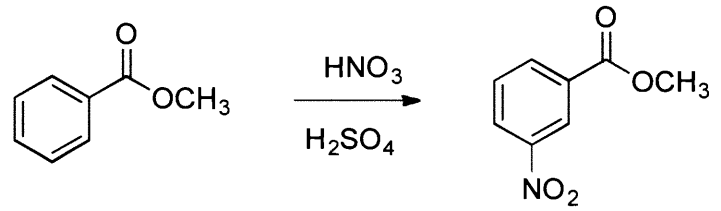
Week 7-9
Carboxylic Acids

Preparation of Methyl 3-Nitrobenzoate

Week 7

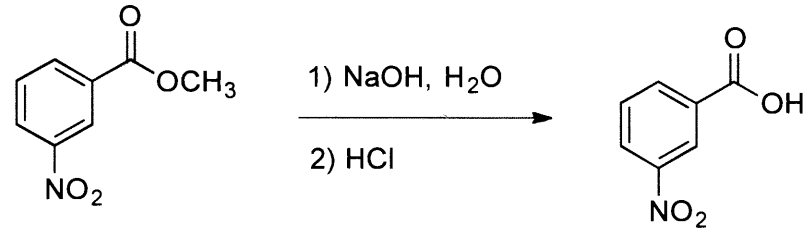


Methyl benzoate

Methyl 3-nitrobenzoate

Preparation of 3-Nitrobenzoic Acid

Week 8

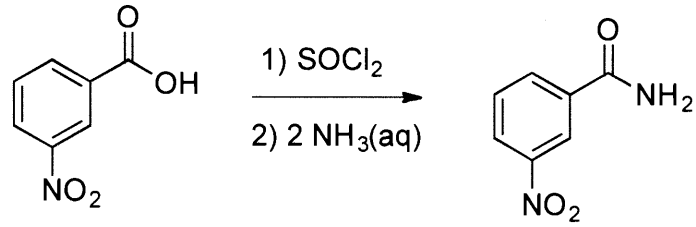


Methyl 3-nitrobenzoate

3-Nitrobenzoic acid

Preparation of 3-Nitrobenzamide

Week 9



3-Nitrobenzoic acid

3–Nitrobenzamide

Preparation of Methyl 3-Nitrobenzoate

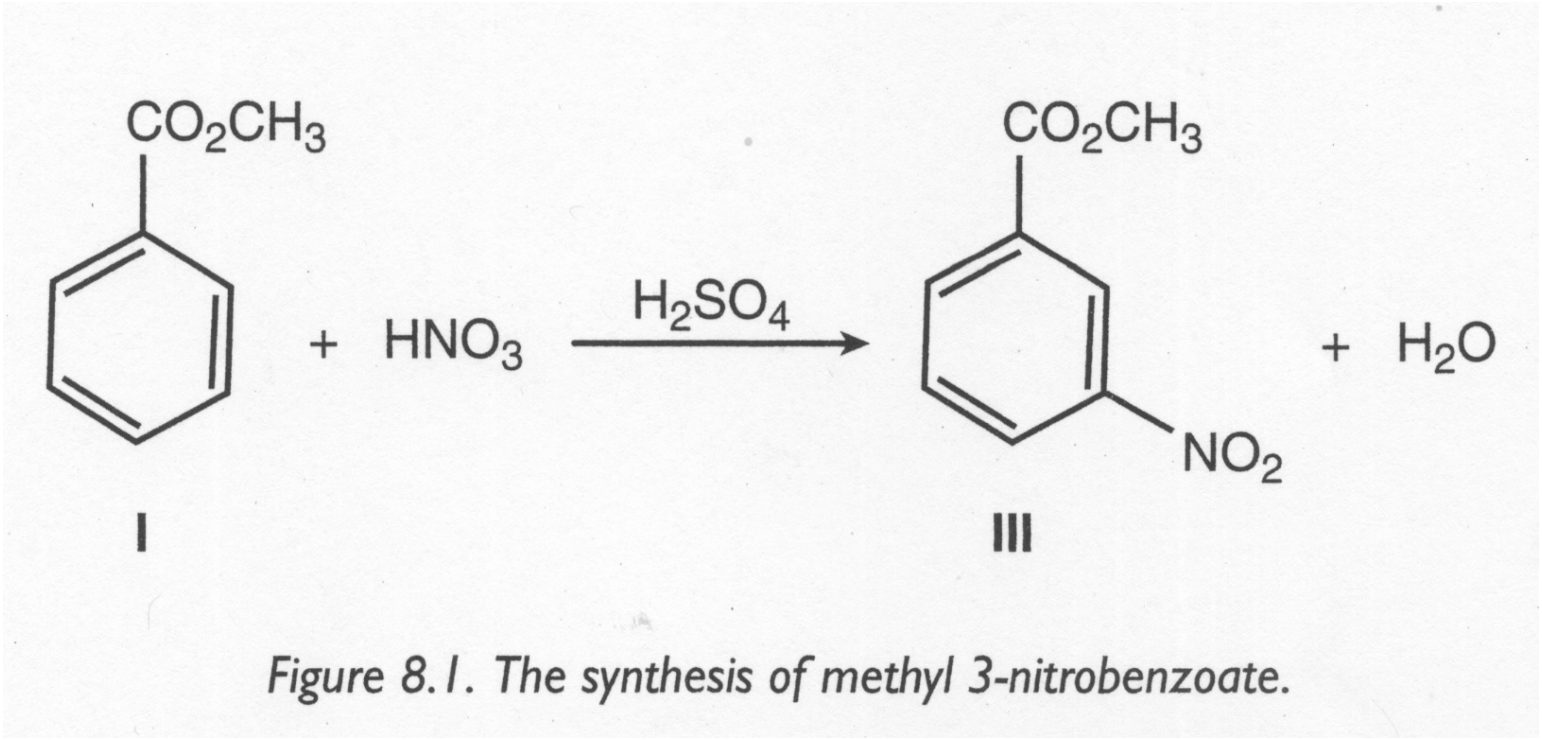


Figure 8.1. The synthesis of methyl 3-nitrobenzoate.

- **We are using concentrated acids. Be careful!!!**
- **All glassware must be dry! Excess of water will stop the reaction.**

Preparation of Methyl 3-Nitrobenzoate Mechanism

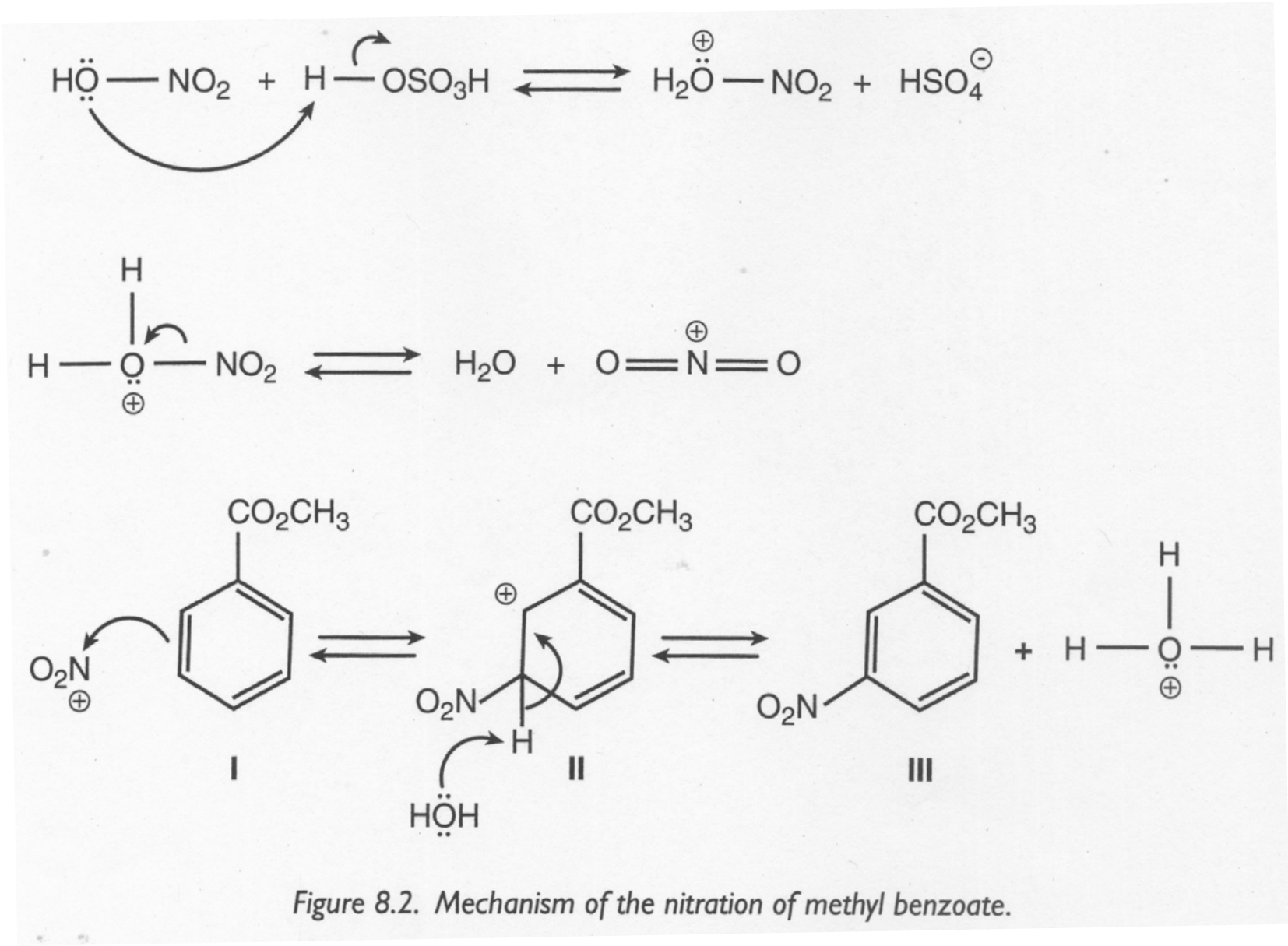
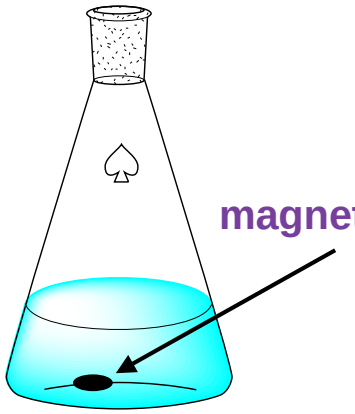


Figure 8.2. Mechanism of the nitration of methyl benzoate.

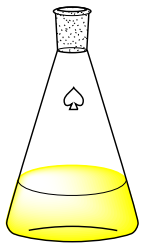
Preparation of Methyl 3-Nitrobenzoate



magnetic stirrer

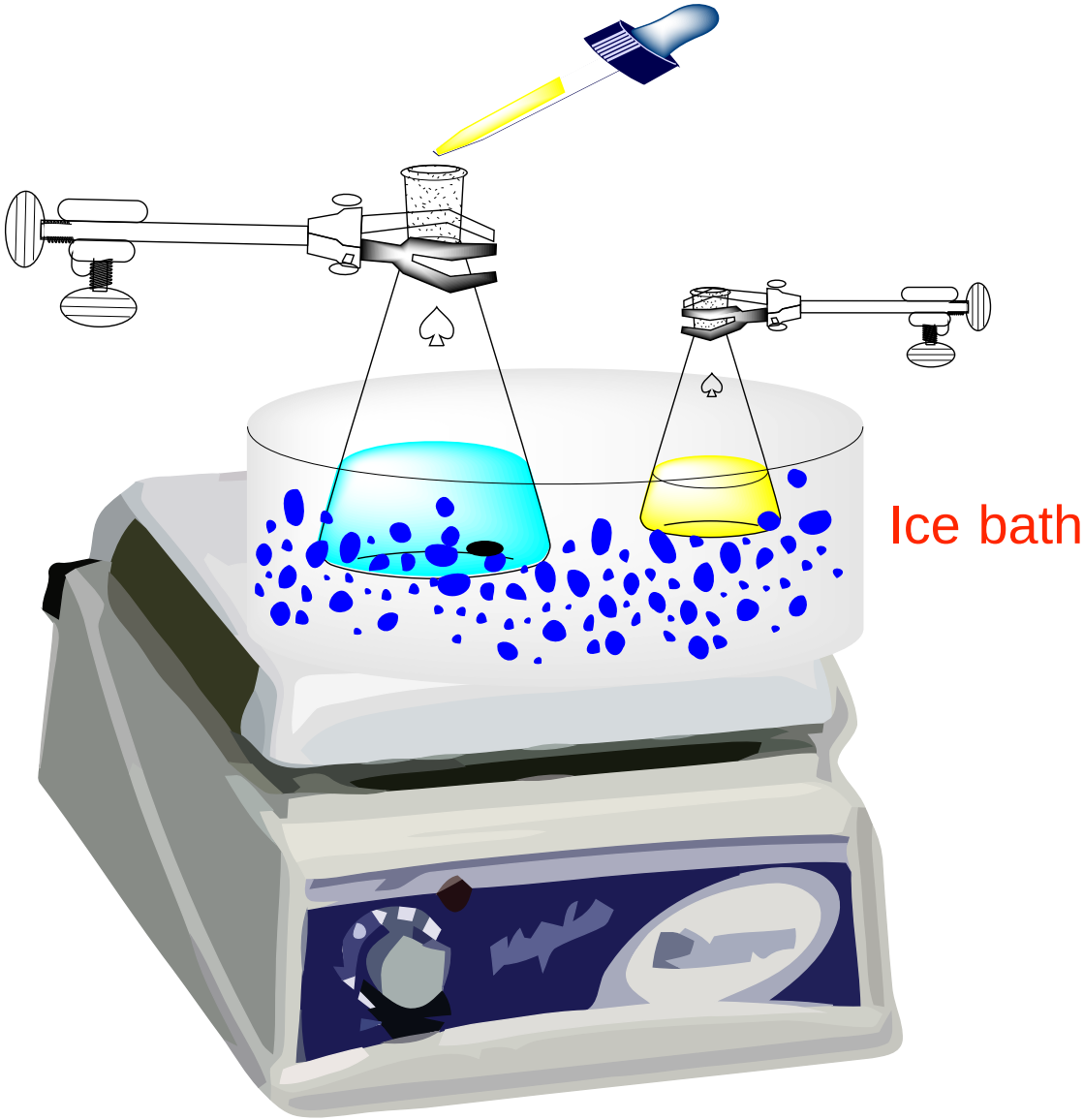
50 mL
Erlenmeyer Flask

7.5 mL H_2SO_4 18M
2.1 mL Methyl Benzoate



25 mL
Erlenmeyer Flask

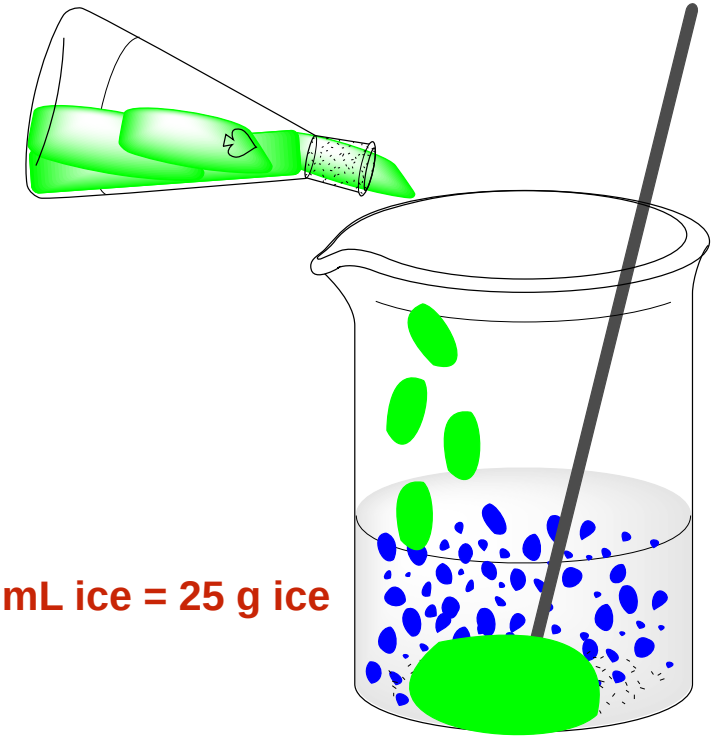
2.5 mL H_2SO_4 18M
1.75 mL HNO_3 16M



Ice bath

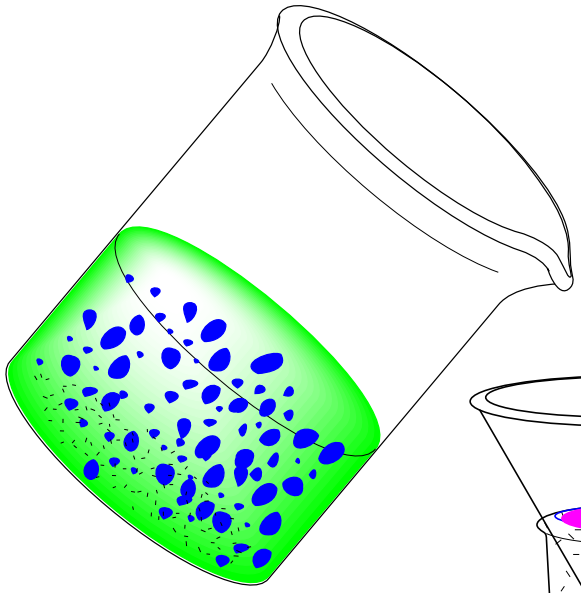
- **Dropwise addition of the HNO_3 solution.**
- **When the addition of the HNO_3 solution is over remove from ice bath and allow to warm to RT.**

Isolation of Methyl 3-Nitrobenzoate

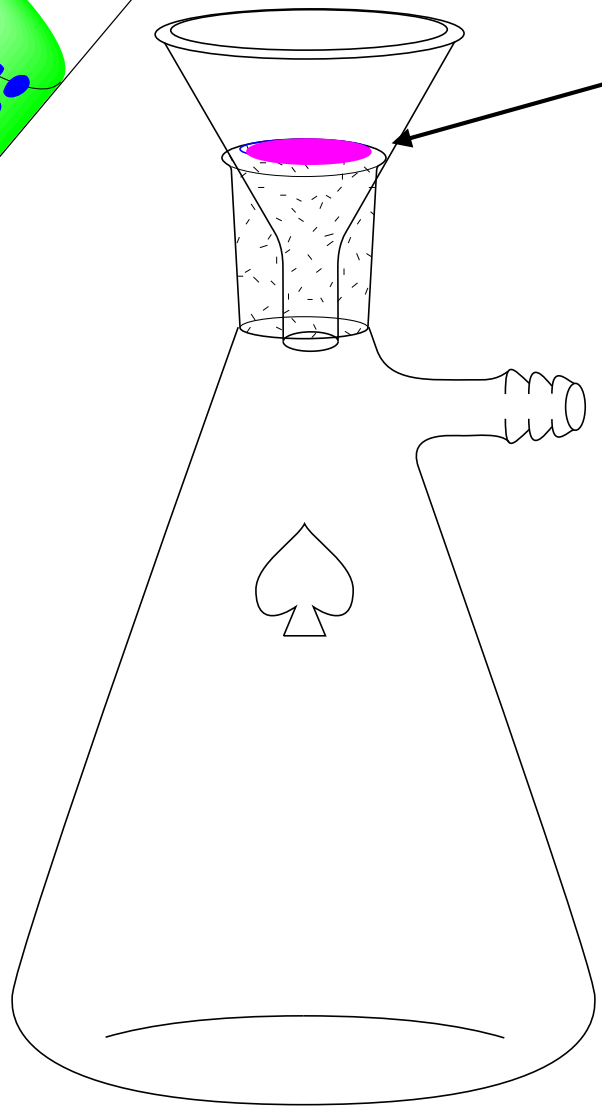


50 mL ice = 25 g ice

250 mL Beaker



Filtration

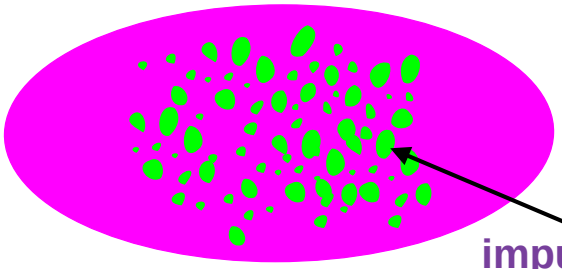


filter paper

Aspirator

- Add reaction mixture into 25 g of ice

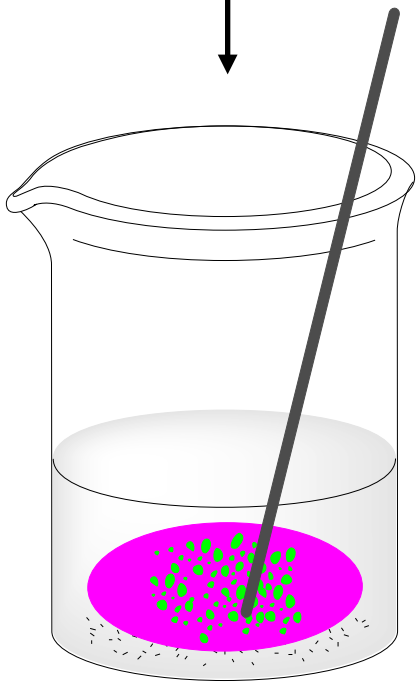
Trituration of Methyl 3-Nitrobenzoate



filter paper

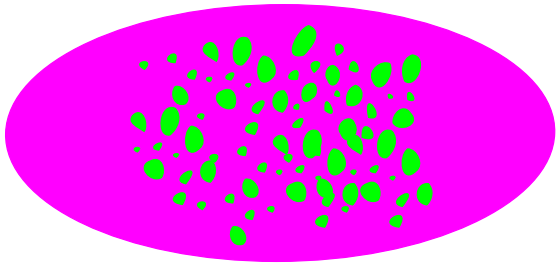
impure methyl
3-nitrobenzoate

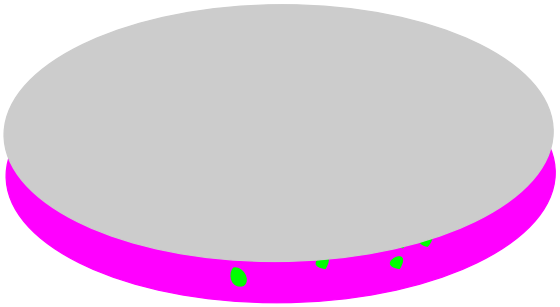
Place in beaker
Redissolve benzoate
with water into beaker



Suction

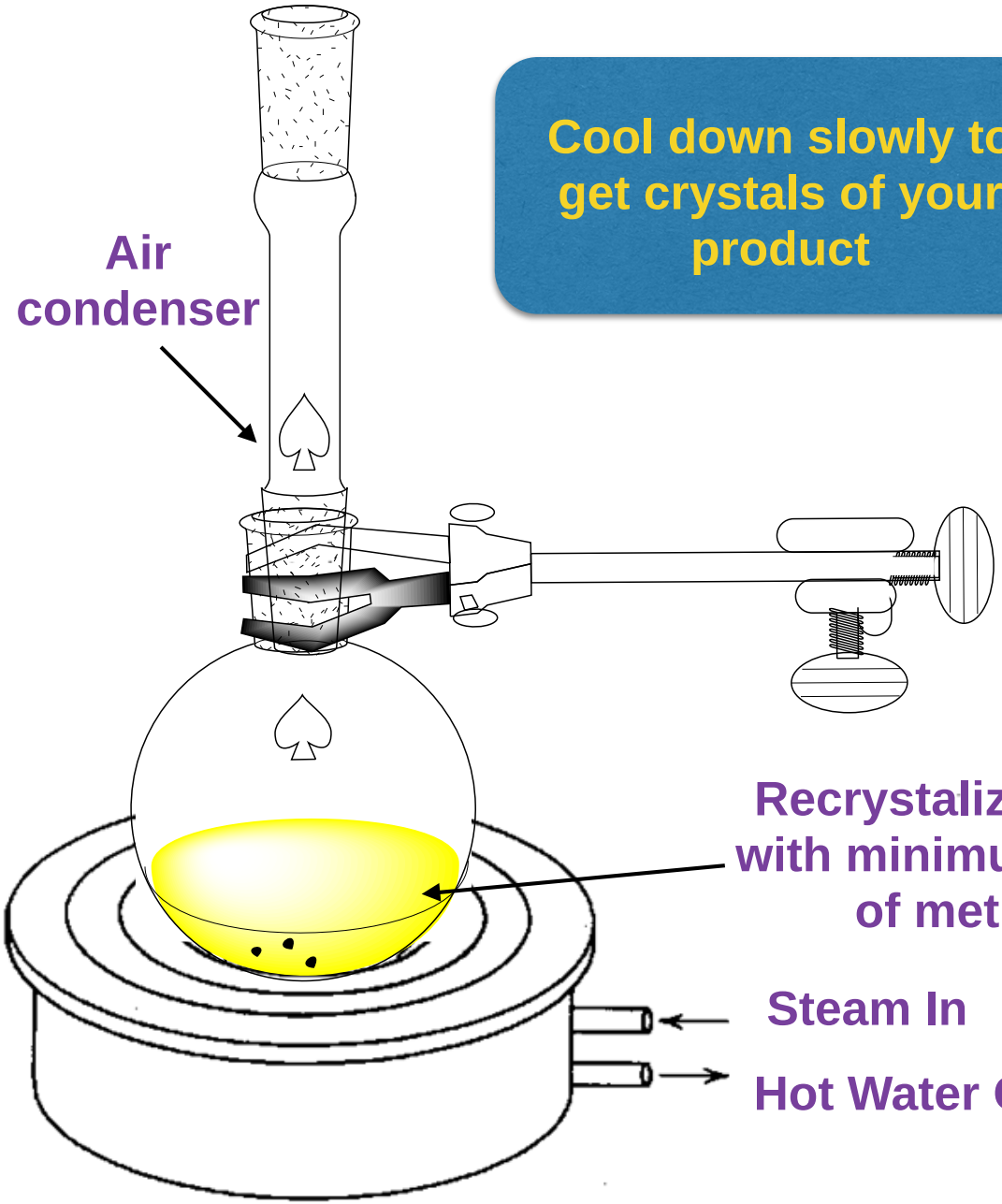
Filtration





Dry filter cake with
filter paper

Recrystalization of Methyl 3-Nitrobenzoate



- Pre-weigh flask or beaker
- Transfer your product, weigh the flask again
- Based on your isolated product calculate % yield

Calculation of % Yield

Calculate the percent yield for the formation of ammonia in the following reaction.

$$\underset{2.2\ g}{N_2} + \underset{1\ g}{3\ H_2} \longrightarrow \underset{1.2\ g}{2\ NH_3}$$

$$\text{Percent Yield} = \frac{\text{Actual Yield}}{\text{Theoretical Yield}} \text{X } 100$$

$$2.2\ g\ N_2 \text{ X } \frac{1\ mol\ N_2}{28\ g\ N_2} \text{ X } \frac{2\ mol\ NH_3}{1\ mol\ N_2} \text{ X } \frac{17\ g\ NH_3}{1\ mol\ NH_3} = 2.7\ g$$

$$1\ g\ H_2 \text{ X } \frac{1\ mol\ H_2}{2\ g\ H_2} \text{ X } \frac{2\ mol\ NH_3}{3\ mol\ H_2} \text{ X } \frac{17\ g\ NH_3}{1\ mol\ NH_3} = 5.7\ g$$

Limiting reagent is the one you run out. In this case N_2

$$\text{Percent Yield} = \frac{1.2}{2.7} \text{X } 100 = 44\ \%$$