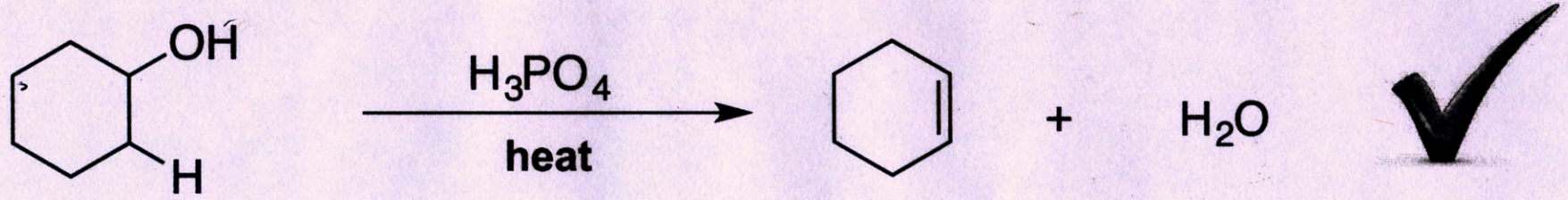
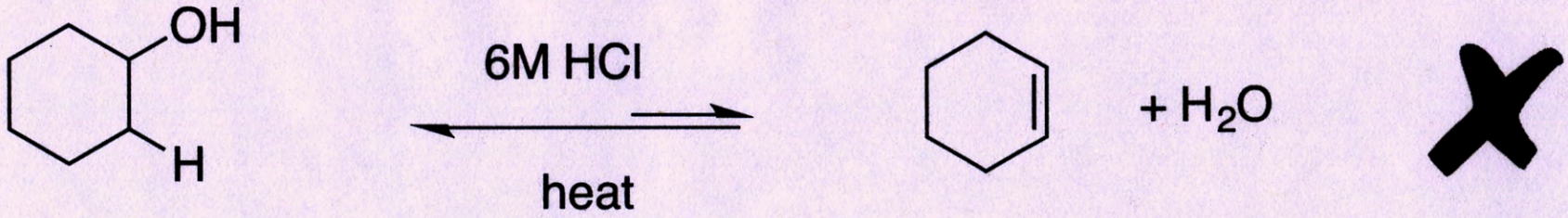
Week 6
Distillation II: Preparation of Cyclohexene
Dehydration of Cyclohexanol





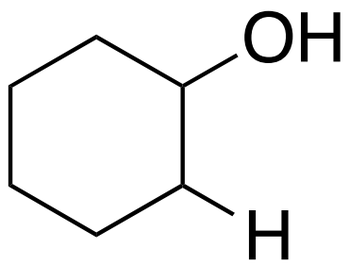
E1 Elimination

Overall….

- Set up dehydration reaction
- Distill product (cyclohexene) out, collect in a "cooled" flask.
- Using a Separatory Funnel extract cyclohexene into ether layer.
- Distill ether out to purify your cyclohexene!

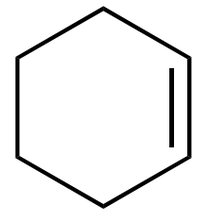
Dehydration of Cyclohexanol

bp = 169 °C



H_3PO_4 **heat**

2.5 mL
(automatic dispenser)



\+ H_2O

bp = 83 °C

5 mL
(automatic dispenser)

You only get 1 shot of cyclohexanol!
If you mess up and go for 2nd you will lose lots of points!

1st Distillation: Distill Cyclohexene Out

- Keep heating BUT keep temperature below 100 °C
- Continue at rate 1 drop / sec until rate drops and reaction flask turns yellow emitting white fumes.

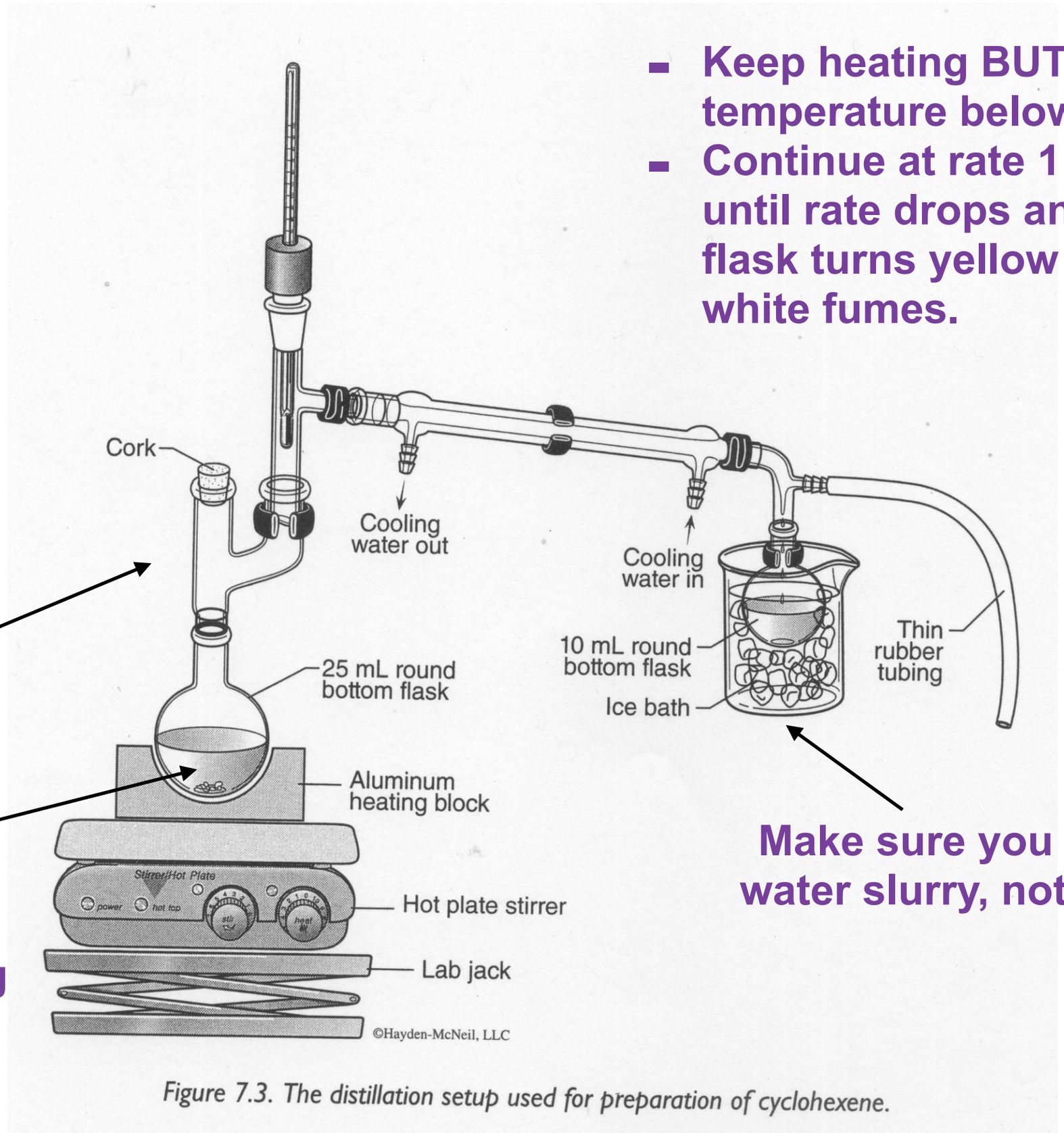


Figure 7.3. The distillation setup used for preparation of cyclohexene.

Simple Distillation + Claisen adapter!

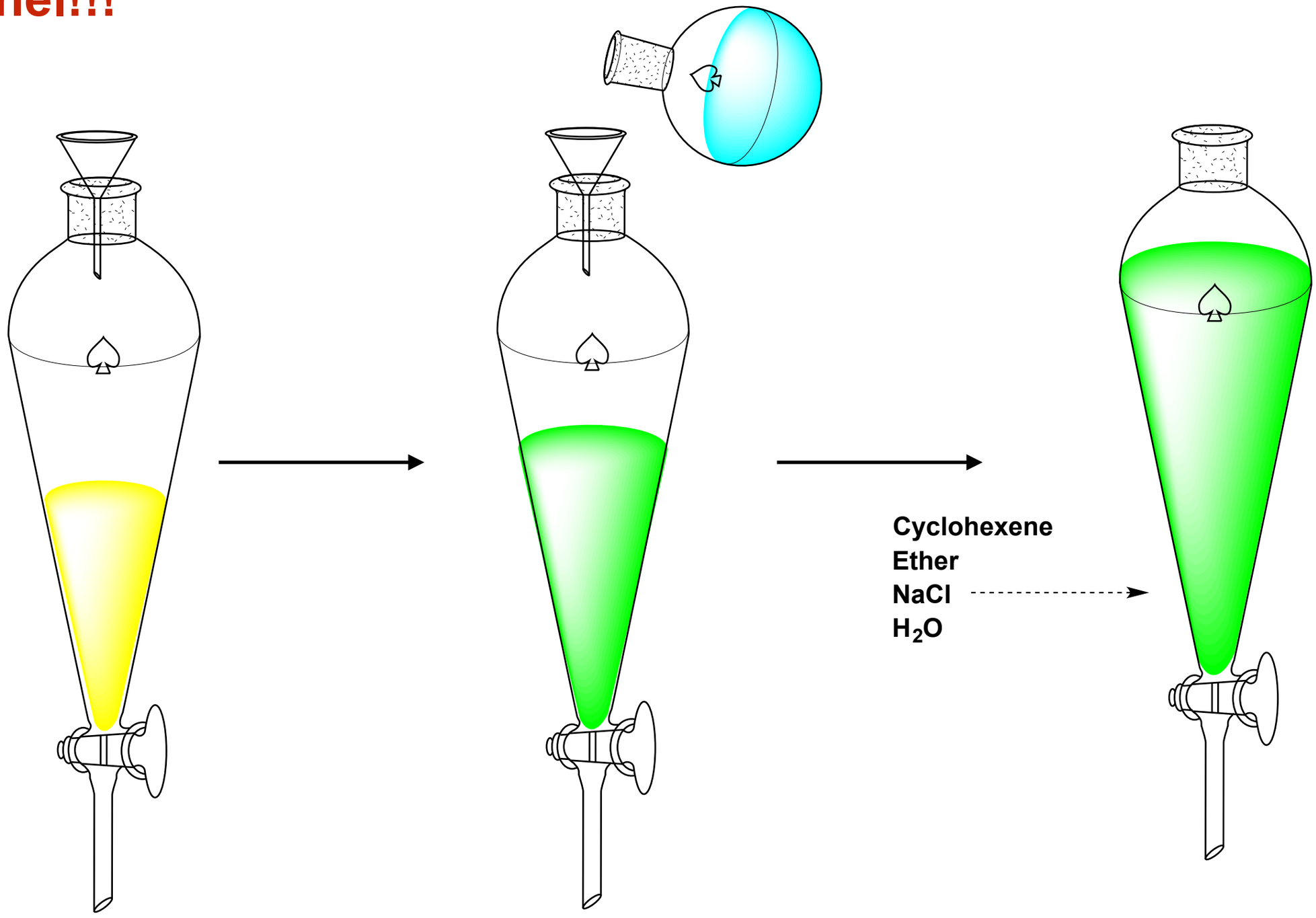
Boiling chips important for smooth boiling (increase surface area)

Make sure you use ice/ water slurry, not just ice!

Extraction Using Separatory Funnel

You need your MSU ID to check out the sep funnel!!!

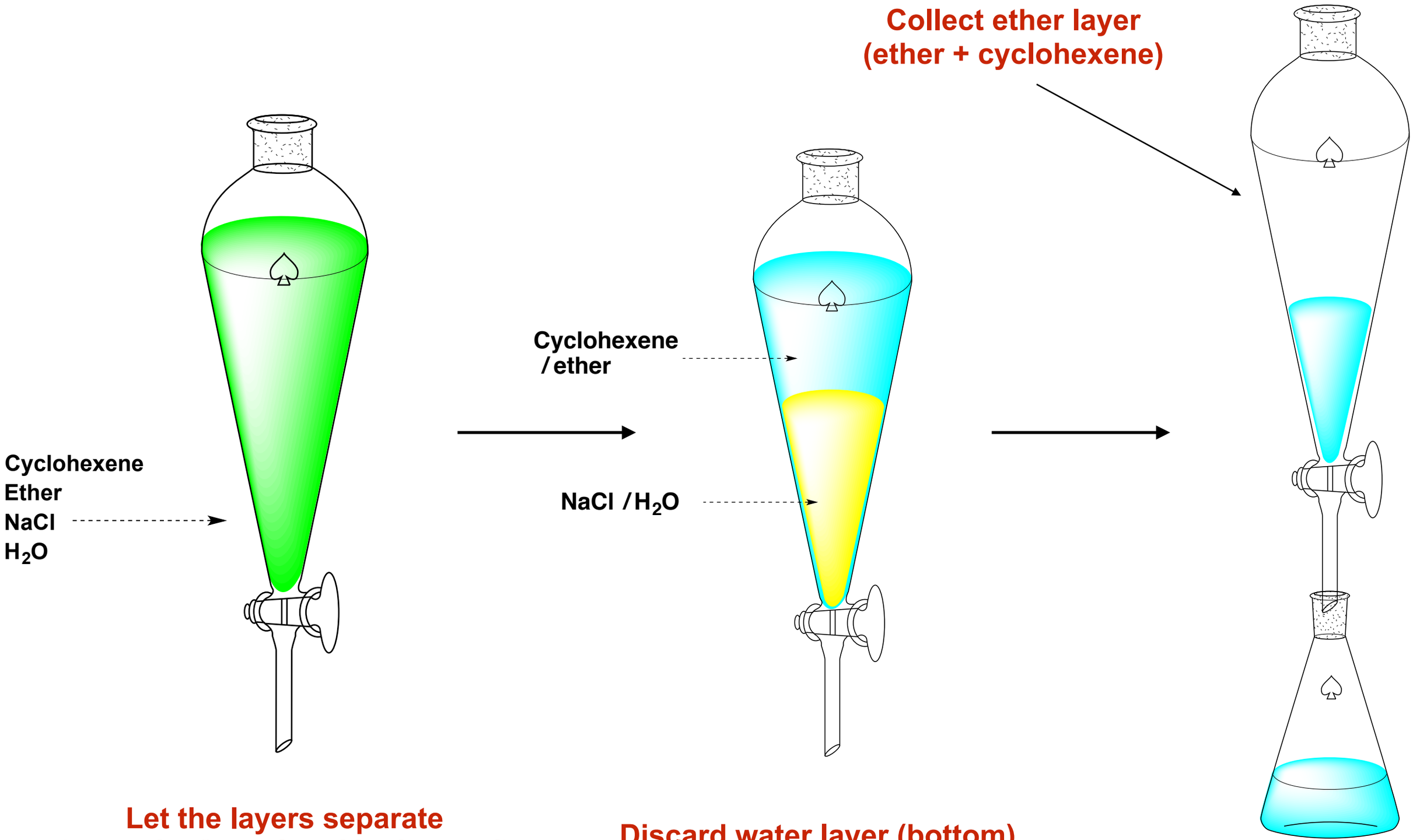
Before using the funnel wash it with acetone and dry it to make sure it is clean!



Before adding your reaction mixture check funnel for leaks using only NaCl (sat)!!!

Stopper your funnel and shake!

Extraction Using Separatory Funnel



Let the layers separate
(open the funnel and let it stand)

Discard water layer (bottom)

Add anhydrous
Na_2SO_4 to dry!

2nd Distillation: Distill Ether Out

Ether: bp = 36 °C

Cyclohexene: bp = 83 °C

Simple Distillation + Claisen adapter!

Boiling chips important for smooth boiling (increase surface area)

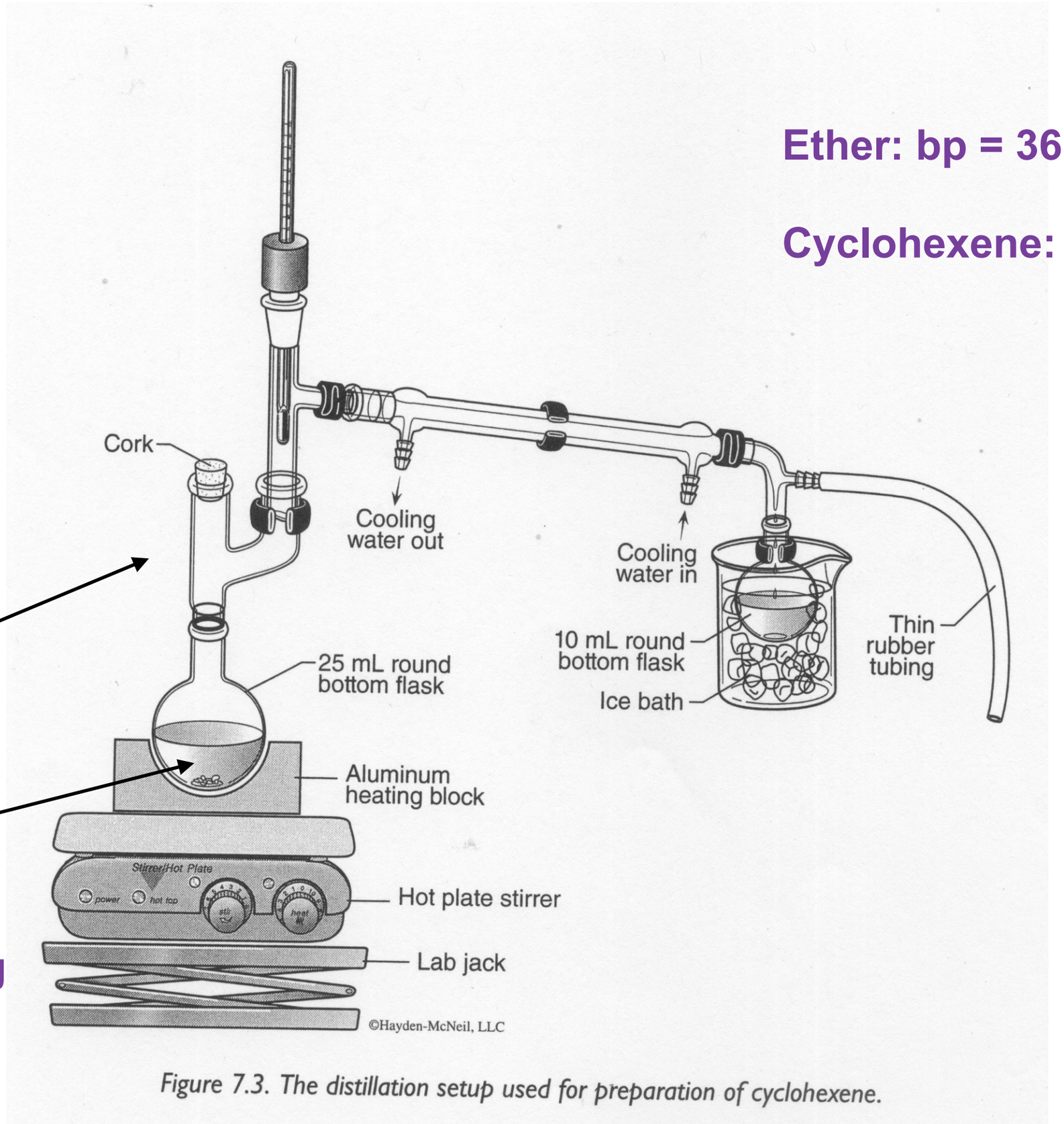


Figure 7.3. The distillation setup used for preparation of cyclohexene.

Distillation 2 Grading

Purity: 60 Pts.
Yield: 50 Pts.
Safety Questions: 10 Pts.
Questions: 10 Pts.
Total : 130 Pts.

You MUST TURN IN your final product
to your TA
Or else, you will earn the minimum grade
for the experiment (60 Pts)