

How to set up a reflux apparatus

Introduction

Reflux is a chemistry lab technique that heats a solution, produces vapor, and then condenses and returns the outgoing vapor into its original mixtures. Reflux is widely used in undergraduate organic chemistry labs.

Before doing this activity, students should have experience using boiling chips, heating mantles and magnetic stir plates in fume hoods. This instruction set might not be appropriate for students that do not have previous experience in organic chemistry labs. Insufficient experience will likely to cause damage to the equipment.

During certain reactions such as Fischer Esterification, the reaction mixtures need to be heated at a high temperature for a long time. Mixtures composed of solutions such as carboxylic acid and ethanol have low boiling points. They will easily evaporate from the solutions and thus hinder the reactions. Using a reflux apparatus, the reaction can be carried out around the boiling points so that no reagents will be lost due to evaporation. The cooling water that circulates in the reflux condenser will condense the vapors as they rise up and keep the reactions going, relatively unintended.

This instruction set is intended to help students who are going to perform Fischer Esterification reaction using reflux technique in organic chemistry lab. Normally, the whole process including the setup and the reaction will take about 2 hours. However, the general principles and the instructions on setting up the reflux apparatus can be applied to any reactions that needed to be heated for a long time. In the latter case, you should ask your lab instructors for more details.

Safety Warning

- Know chemical hazards that will appear in the lab beforehand.
- Be careful with concentrated sulfuric acid. It is highly corrosive.
- Make sure that all the tubings are securely attached to the condenser.
- Use hose clips in case the water leaks.
- Do not plug the heating mantle directing into the wall outlet.
- Do not touch the heating mantle once the reaction has started.
- Do not move the whole apparatus after it has been set up.



Equipment and compounds needed for the Setup

- 18 g of 3-methyl-2-butenic acid
- 100 ml of absolute methanol
- 5ml of concentrated sulfuric acid
- Hood with a standard variastat outlet and a regular outlet.
- Heating mantle
- Magnetic stir plate
- Sand
- 250 ml round-bottomed flask
- Boiling chip
- Reflux condenser
- Two rubber tubings
- Iron stand
- Clamp

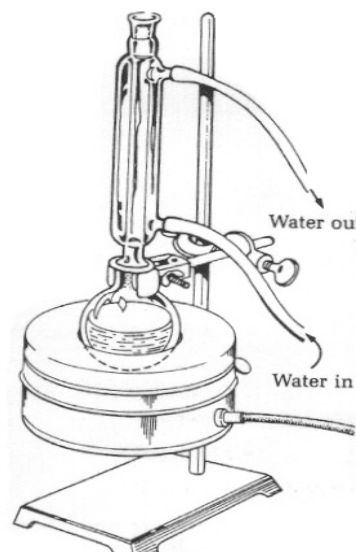


Figure 1 Reflux Apparatus

Procedures

Basic Setup

1) *Setting up the heating mantle*

- Choose the right size of the heating mantle and put it on a magnetic stir plate.

→ *The heating mantle should fit the flask size. The temperature will be difficult to control if the heating mantle lacks contact with the flask. The heating mantle will overheat or burn out if left unintended for too long.*

- Add the sand into the heating mantle. Make sure the sand occupies 2/3 of the heating mantle.



Figure 2 Heating mantle



Do not plug anything in to the outlets during the preparation stage in case the heating mantle becomes overheated.

2) Setting up the glassware

- Make sure that the round bottom flask and the reflux condenser are cleaned and dried thoroughly.
- Add 18 g of 3-methyl-2-butenic acid, 100 ml of absolute methanol and 5ml of concentrated sulfuric acid into the round bottom flask.
- Put a boiling chip into the flask.
- Clamp the flask over the heating mantle. Make sure the bottom of the flask is well covered by the sand to achieve sufficient heat transfer.
- Clean the reflux condenser and insert it to the round bottom flask. Make sure the glass joints are lightly greased with silicon.
- Clamp the reflux condenser to the iron stand in the upright position.



Figure 3
Round bottom flask

3) Setting up the tubes

- Connect one end of one rubber tubing with the water tap and the other end with the bottom interface of the reflux condenser.
- Connect one end of the other rubber tubing with the top interface of the reflux condenser. Make sure that the other end leads to a water sink.
- Use tape to attach the tubing to the edge of the sink in case the water leaks.



- **Make sure tubes are securely attached to the condenser.**
- **Use hose clips in case the water leaks.**

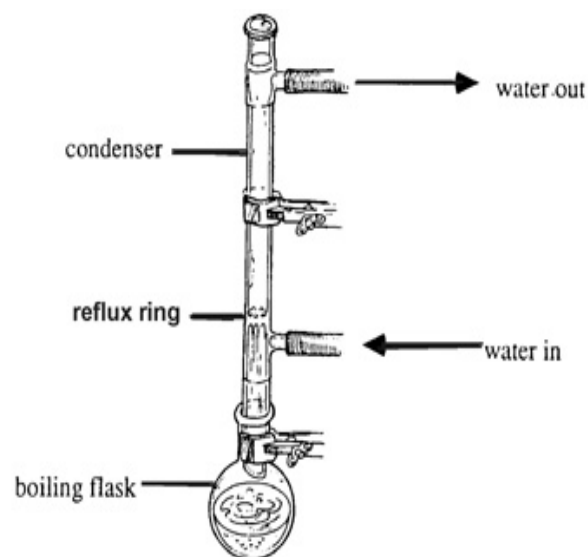


Figure 4 The direction of water flow

Initiate the reaction

1) Plugging in power cords

- Check Varistat, a variable voltage controller, to make sure that it is off.
- Set the voltage dial to around 50-70 Volts.
- Plug the heating mantle into the Varistat on the left side of the hood.
- Plug the magnetic stir plate into the regular outlet in the hood.

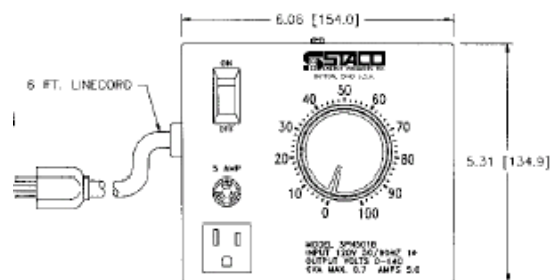


Figure 5 Varistat



Do not plug the heating mantle directly into a wall outlet. High voltage will burn the heating mantle out rapidly and it has the potential to start a fire.

2) Operating the apparatus

- Switch the right knob on the magnetic stir plate and make the boiling chips stir the mixture evenly.



Figure 6 magnetic stirring plate

- Turn the water on slowly. The water must flow at an even rate or otherwise the tubing will pop off due to the high water pressure.



Figure 7 water tap

- Set an alarm to indicate the end of the reaction.



Figure 8 Alarm

→ Now the set up for the reflux apparatus is complete!

Overall setup



The overall setup is shown in Figure 9. The picture was taken during a lab period in CHEM 213M, Spring 2016, in 221 Whitmore Lab, Penn State.

Figure 9 Overall setup for reflux apparatus

Tips

- Do not touch the heating mantle once the reaction has already started.
- Do not move the whole apparatus after it has been set up.
- Know the properties of the chemical compounds using in the lab beforehand.
- Make sure the sink is not stuck.
- Perform multiple TLC tests to monitor the progress of the experiment.

References

- 1) Figure 1 Reflux apparatus [image] <http://www.organicchem.org/oc2web/lab/exp/lp/lpreflux.html> (Accessed Aug 2, 2016)
- 2) Figure 2 Heating mantle [image] <http://pudap.com/heating-mantel/> (Accessed Aug 2, 2016)
- 3) Figure 3 Round Bottom Flask [image] <http://www.duran-group.com> (Accessed Aug 2, 2016)
- 4) Figure 4 The direction of the water [image] <http://chemistry.stackexchange.com/questions/13911/water-flow-in-condenser> (Accessed Aug 2, 2016)
- 5) Figure 5 Variostat [image] <http://variac.com/> (Accessed Aug 2, 2016)
- 6) Figure 6 Magnetic stir plate [image] <http://basicsscience.com> (Accessed Aug 2, 2016)
- 7) Figure 7 Water tap [image] <http://www.ecvv.com/product/2609587.html> (Accessed Aug 2, 2016)
- 8) Figure 8 Alarm [image] <http://realitysnub.wordpress.com> (Accessed Aug 2, 2016)
- 9) Danger sign [Image] <http://cliparts.co/danger-sign> (Accessed Aug 2, 2016)